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OPTIMISATION OF COD REMOVAL FROM THE EFFLUENT DISCHARGED BY THE WET CORN PROCESSING INDUSTRY IN WESTERN CAPE

by

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DECLARATION

I, Thuletu Mbita, declare that the content of this thesis represents my own unaided work, and the thesis has not previously been submitted for academic examination towards any qualification. Furthermore, it represents my own opinions and not necessarily those of the Cape Peninsula University of Technology.

28 February 2021

Signed **Date**

ABSTRACT

This study was performed at a wet corn milling(WCM) plant in the Western Cape (WC). It explores the application of Six-Sigma DMAIC to develop a procedure for reducing the carbon oxygen demand (COD) concentrations in the WCM effluent, using the scientific approach – adsorption coconut granular activated (CGAC) method – relevant quality tools and quality techniques.

The research method followed a structured Six Sigma DMAIC framework for primarily investigating the root causes of non-conforming COD concentrations in the effluent generated by the WCM plant. Thereafter, it seeking a suitable procedure to improve the current management of the COD concentrations by the WCM plant to consistently adhere to the legislated COD concentrations requirement.

The data used in this research was collected by means of quantitative laboratory experiments. Where, trial one experiments used 36 acidic samples, and trial two used 36 alkaline samples. The data was interpreted and analysed using statistical tests. The validity of the data was assured by means of applying reference standards and repeated measurements under different environmental conditions.

The findings indicated that there was a gap in the current control measuring system used to ensure that the effluent was free of product cross-contamination. This research also found that the current procedures used for addressing the management of non-conforming of COD concentrations were not effective. Furthermore, the current COD detection system was found to be working, but not effectively enough; therefore, urgent continuous improvement is required to better its performance output.

This study recommends that an additional process step is required for treating the non-conforming COD concentrations to comply with the legislated COD standard requirement. Moreover, improvement is required in the skills development of the process owners to better monitoring of the interlinked processes. The current COD detection system requires improvement to enhance its performance.

Keywords: COD concentrations, effluent quality management, quality improvement, Six Sigma DMAIC, Process flow, RCA, hypothesis testing, and FMEA.

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DEDICATION

This thesis is dedicated to my late Aunt Nombi Rhangana, my late Grandma Makhuboni Viginia Ntsume, my mother Patricia Ntsume, my mom Pauline Jacobs, and my son Intando Mbita

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LIST OF ABBREVIATIONS

Abbreviation	Full name
COD	Chemical oxygen demand
CGAC	Coconut shell granular activated carbon
DMAIC	Define, measure, analyse, improve and control
FMEA	Failure mode effect analysis
g	grams
GAC	Granular activated carbon
IRS	Internal Reference Standard
Min	Minutes
pH	Potential hydrogens
QC	Quality control
SA	South Africa
SANAS	South African National Accreditation System
SHE	Safety, Health and Environment
SOP	Standard operating procedure
TOC	Total organic carbon
WC	Western Cape
WCM	Wet corn milling
ISO 9001:2015	International Organization for Standardization for quality management systems
ISO 14001:2015	International Organization for Standardization for environmental management systems
ISO 17025	General requirements for the competence of testing and calibration

GLOSSARY OF TERMS

Term	Definition
Activated carbon	A powdered, granular or pelletized carbon product obtained by heating or chemically treated charcoal to increase its adsorptive influence (Rogowsky, 2006: 1).
Adsorption	A process of adhesion of atoms, ions or molecules onto the surface of an activated carbon (Roque-Malherbe, 2007: 39).
Carbon	A naturally occurring non-metallic element that is present in all organic compounds (Jhaveri & Roosa, 2009: 110).
Chemical oxygen demand	Test used to measure the amount of dissolved oxygen required to oxidize and stabilize organic and inorganic content of the sample solution (McKinney, 2004: 225).
Functional groups	The group of atoms within a molecule that are responsible for its chemical behavior (Hanson, 2001: 1).
Gemba walk	A walk conducted in a process to evaluate its state (Mann, 2005: 95).
Hydrocarbons	Compounds that consists of hydrogen and carbon (Arora, Sachdeva, & Sardana, 2018: 128).
Insoluble proteins	Proteins that cannot be dissolved (Hermann & Razin, 2002: 511).
Macro channels	Sub-process of steps in a process (Welch, 2008: 120).
Mechanism	A systematic sequence of elementary reactions that occur during the chemical change in a chemical reaction (Tomlin & Turanyi, 2014: 38).
Potential of hydrogen	The parameter that is used to express the acidity or alkalinity of a solution on a scale that ranges from 1 to 14 (Cameron & Craig, 2009: 17).
Proteins	Groups of organic macromolecules that contain hydrogen, carbon, oxygen and nitrogen (MacLaren & Morton, 2012: 50).
Solute	The analyte sample that gets filtered (Raj, 2002: 117).
Wet corn processing	A process of milling corn, involving preliminary soaking in another liquid and separating the corn into its various components (Ndlovu, 2013: 18).

CHAPTER 1: SCOPE OF THE RESEARCH

1.1 Introduction and motivation

This introductory chapter provides insight into the research environment of this research study, which seeks to create the most efficient filtration-bed granular activated carbon (GAC) procedure for optimal removal of COD concentration in the WCM-generated effluent in the WC. The primary concern of this research is the inconsistent adherence of the WCM to meet the government COD requirement for the disposal of the discharged effluent, and this chapter provides the background to the research issue, the intent of this research, and research goals of this report.

Brouckaert, Buckley and Gianadda (2002) explain the wet corn milling processing as a process of separating raw corn kernel into its components and transforming corn starch into different grades of glucose syrups. According to Garcia Einschlag (2011: 8), the amount of water used during wet corn milling processing is 0.64 m^3/ton product; with the amount of COD loads 2.65 m3/ton product. Ndlovu (2013 :72) commented that only one of the WCM plants in South Africa (SA) discharges 1782 kilolitres of effluent per day with regard to this context. In addition, WCM is typically among the top two processes that produce the largest amounts of effluent with high COD concentrations across the worldwide (Islam, Sangeetha, & Thangadurai, 2020: 2006). Garcia Einschlag (2011: 8) states that wet corn milling consists mostly of organic matter, which is the source of high COD in it.

Brouckaert, *et al*. (2002) explain that the effluent generated by wet corn milling processing commonly contains high concentrations of dissolved solids; which translates into an elevated level of COD in the effluent. Ndlovu (2013: 69) reasoned that the high protein and starch content which is emitted as a direct result of the wet corn milling process is the primary reason why COD concentrations in the effluent is so high. Significantly, Das, Misra, Rao, and Swamy, (2005: 41) observe that elevated COD concentrations in water leads to the depletion of biodiversity in aquatic organisms. High COD content signifies the lack of oxygen water, hence the lessening of biodiversity (Van Schoor, 2005).

Rising levels of COD concentrations in the effluent generated by the WCM industry is causing concern about effluent quality control in this industry field (Akpor & Muchie, 2011: 2379). The poor quality (high COD) of the discharged effluent leads to increasing eutrophication and bacteriological pollution of rivers and dams, which is also significant (Hassan & Schreiner, 2011: 77). Ndlovu (2013: 1) stated that due to a rise in water demand in SA, the situation was exacerbated; which is a partial consequence of the growth and expansion of the WCM industry. For this reason, the amount of the effluent discharged into the municipal sewage system has also increased (Islam, *et al*., 2020: 2006). Therefore, SA made it mandatory, through the promulgation of the South African Water Act No. 54 of 1956 that the quality of the effluent should be treated to the required standard before its release into the municipal systems (Mema, 2010: 60). The legislation for the treatment of effluent specifies that the COD concentrations of the effluent discharged may not exceed 5000 ppm which is equivalent to 5000 mg/L; according to the South African Water Act, No. 36, 1998 (South Africa, 2013: 7). Bwapwa and Jaiyeola (2016: 1) moot that the availability of usable water is a major concern in SA and forecast that water demand will exceed supply by 17% by 2030.

This foregrounds the important role of water in the industry and also the critical need for effective management of water. To prevent depletion of aquatic organisms, the South African government has legislated that municipalities must treat effluent water before it is discharged into rivers or oceans (Hassan & Schreiner, 2011: 77). Hence, this study focuses on decreasing COD concentrations in WCM effluent to improve the quality of the effluent discharged into the municipal water system in the WC.

1.2 Background

This quantitative study was conducted in a laboratory at a WCM plant in the WC, SA. The WCM manufactures different types of products from raw corn, and in 2013 it was reported that they generated an average of discharges 1782 kilolitres of effluent each day (Ndlovu, 2013: 72). Garcia Einschlag (2011: 8) state that effluent generated by the WCM is categorised as 'high COD concentrations', due to its high protein and starch content. The focus of this study is on the laboratory treatment of the effluent generated by WCM with GAC to manage the level of COD by reducing the concentrations of proteins and starch in the effluent to below admissible legislated levels.

Ndlovu (2013: 1) reports that the concentrations of the COD in the effluent generated by the WCM industry in SA differs from day to day, depending on the characteristics of the raw material and the effectiveness of the WCM processes. Moreover, in an interview, Mr Gwadla, systems manager at the WCM (2017) admitted that there are instances when effluent COD levels discharged by the WCM exceeded the regulated COD limit of 5000 ppm. He added that the average COD concentrations of the effluent ranged from 2000 ppm to as high as 30 000ppm. Mr Jackson, System plant manager (2017) pointed out that an increase in effluent COD concentrations was noted after the WCM plant had attempted to reuse its processed water to reduce fresh water supply consumption.

Notably, the Department of Water Affairs and Forestry (2000) states that failure to comply with the legislated effluent COD specifications results in financial penalties, or potential loss of the effluent discharge license and this can ultimately result in company or industry shutdown. Furthermore, Hot Water Treatment (2013) asserts that poor quality of the discharged effluent results in additional fresh water being required to dilute the effluent, besides additional treatment chemicals, which elevates the labour cost required to treat the effluent at the City of Cape Town Water Treatment Stations. Thus during production runs at the WCM plant, a production operator collects samples which are then sent to the WCM internal laboratory for specific tests to be done (Gwadla, 2017). Six different effluent samples are collected from various sampling sites, namely condensate, glucose effluent, wet milling effluent, and sump effluent on the production line at 2 hour intervals twelve times per day (Jackson, 2017). In an interview, Gwadla, (2017) stated that the laboratory's testing equipment is calibrated annually by a South African National Accreditation System (SANAS) accredited vendor to validate the precision of instrumentations used in the laboratory. The tests performed on the samples are the total organic carbons (TOC) test, potential hydrogens (pH), conductivity and temperature measurements (Gwadla, 2017). The samples are then combined in an auto total effluent sample tank to form a 24hour composite sample for that day, and the same tests are redone on the composite sample. The results of the laboratory tests are communicated to the production operators for further process monitoring and adjustment, for pH, and for conductivity adjustments (Gwadla, 2017). Currently, there is no pre-treatment system for effluent except for pH control prior to discharge to municipal pipes, even when the COD concentrations exceeds the municipality's specifications for industrial effluent discharge. Thus at present all the effluent, including the non-conforming effluent, is discharged into the municipal drains.

Concurrently, a City of Cape Town (Water and Sanitation Department: Scientific Services Branch) representative collects one effluent sample from the composite sampling effluent tank on the WCM site once every six weeks (Gwadla, 2017). Multiple analyses, including COD, are performed on the sample in City of Cape Town laboratories to determine if the effluent conforms to the prescribed limits. If the effluent does not meet minimum specifications, the City of Cape Town will issue a contravention to the WCM organisation for the discharged effluent and a follow-up sample is taken without pre-notification to the WCM plant (South Africa, 2013: 1-14).

Contraventions that are issued by the City of Cape Town, result in an increase in charge of effluent treatment and is calculated using the following factors: COD concentrations, pH, conductivity and the amount of potable water used for the particular month (Gwadla, 2017). The municipality stipulates that the COD value used in the industrial effluent charge process is calculated using the average of the last four COD measurements recorded by the City of Cape Town Water and Sanitation Department: Scientific Services Branch (South Africa, 2013:7).

Even though the WCM is ISO 14001 certified, at present this WCM organization does not have facilities to pre-treat its effluent COD prior to discharge, when it exceeds the municipality regulatory limit (Gwadla, 2017). Therefore, this research sets out to assess different filtration procedures for optimal removal of COD from the WCM effluent.

1.3 RESEARCH PROBLEM STATEMENT

The effluent discharged from a WCM does not consistently adhere to the required regulatory specification limit for the COD concentrations in the discharged effluent by the WCM plant based in northern suburb industrial area in the WC.

1.4 Aim of the study

This study aims to develop and document a processing step, which will improve the quality of the effluent (reduce COD concentrations) discharged by a WCM plant located in the WC province of SA to consistently adhere to the legislated discharge standard. In addition, the process introduced must be easily controlled at the operational level and will consider environmental concerns.

1.5 Primary research question

Can a quality management procedure be developed for a WCM plant to optimise the removal of COD in the effluent discharged to meet regulatory specifications consistently?

1.6 Investigative questions

1.6.1 Which stages in a WCM process contribute to increasing concentrations of COD in the discharged effluent?

1.6.2 What are the causes of the high COD content present in the discharged effluent of the various process stages?

1.6.3 Can a process variable(s) be identified that when adjusted yields the optimal removal of COD from WCM effluent?

1.6.4 Can a modified procedure be employed to reduce COD concentrations in effluent generated by the WCM process to comply with the regulatory specification consistently?

1.7 Research objectives

1.7.1 Investigate which stages in wet corn processing have a significant influence on the subsequent high COD in the discharged effluent.

1.7.2 Establish the causes of the increasing levels of COD in the effluent generated by a WCM.

1.7.3 Identify variables that can be adjusted to yield the optimal removal of COD from WCM effluent.

1.7.4 Establish an effective procedure for removal of the COD in the effluent generated by a wet corn milling process.

1.8 Conceptual framework

This study examines the extent to which different variables are able to influence the removal of COD concentrations from the effluent discharged by a particular WCM plant. The adjustable variables which are referred to in the third research question (as mentioned in section 1.6.3) and objective (as mentioned in section 1.7.3) above are the dependent variable in this study, includes the COD concentrations in the effluent sample. The independent variables are the quantity of the GAC applied for the filtration, pH and filtration time. A Six Sigma approach is proposed by Jelena, Krivokapic, Sokovic, and Vujovic (2009: 4) that is capable of framing the concepts included in this framework.

1.8.1 Properties of GAC

Research conducted by Aluyor and Badmus (2008: 3887**)** demonstrated that GAC filtration is an effective method for COD removal from effluent. They claim that GAC is the most chemically stable when compared to other methods and it is readily available. Consistent with this, Gaikwad and Mane (2013: 642) also reported that GAC is the best filtration method because of its multifaceted nature. Moreno-Castilla (2004: 5) noted that the functional groups in GAC that contain oxygen, namely carboxylic, phenolic, lactonic and carbonyl groups, are the most important since these result in most efficient adsorption activity of the GAC surface area.

Granulated activated carbon exist in a form of coconut-shell activated carbon (CGAC) (Dawn, Kumari and Nirmala, 2015: 238). Galloway (2019) reasoned that the CGAC's internal structure consists of millions of pores that form interconnected capillary passages for the molecules, which provides the optimal removal of COD. This research is focussed on COD removal using coconut shell GAC as an adsorbent.

Jong, Posttinger, and Sanford (2008: 97) stated that CGAC provides the most efficient removal of COD concentrations from the effluent. This is in agreement with the view of Singh and Verma (2019: 288), who adds that a key element of activated carbon's adsorption is the extensive internal surfaces of the pores, which allows the filtered sample to pass through varying pore sizes and optimizes the adsorption activity. The aim of this research is to establish a modified adsorption method to achieve optimal removal of COD from the effluent generated by a WCM plant in the WC.

1.8.2 Effluent quality management

The South African Department of Water Affairs and Forestry (2000) expresses the view that effluent management involves monitoring the quality of effluent that is discharged from organizations. Therefore, an integral part of effective effluent management is conducting experimental measurements of the effluent to ensure the quality is adequate. The results of measurements are analysed to evaluate whether the discharged effluent conforms to the prescribed regulatory requirements. The quality of the effluent is affected by quality variables which are also the components of water quality (as mentioned in section 1.2 of this chapter). The amount of these components that are present in the effluent has an impact on the quality of the effluent. It is believed that introducing effective and efficient operations will lead to a quality of the effluent discharged that conforms to requirements. These activities include process control tests, data analysis, and management commitment, to be able to implement interventions that ensure the quality of the effluent is improved and maintained in accordance with effluent quality regulations (Icon Water, 2015). Botes, Oelofse, Taljaard, Vilijoen (2004) stated that introducing efficient management of the quality of the effluent involves quality improvement initiatives which include the use of quantitative studies, which has advantages such as those highlighted in the section that follows.

From the preceding discussion it is deduced that CGAC filtration procedures may be regarded as an effective mechanism to remove COD concentrations from WCM effluent and thereby manage the quality of effluent.

1.8.3 Factors affecting GAC adsorption

According to Nekoo and Shohreh (2013: 87) and Davids (2006: 109), adsorption of the hydrocarbons by GAC can be improved by artificially manipulating variables during the adsorption procedure to optimize the removal activity of COD concentrations from the solute. Ghodale and Kankal (2014: 38) reported that there are many variables that can influence the optimisation activity of GAC; however, in this study, only three variables were studied, namely CGAC surface area, CGAC contact time with the solute, and the effect of hydronium ions (pH). These variables were manipulated in this study with the aim of establishing the most suitable procedure for the optimal removal of COD concentrations in the WCM effluent.

Ushakumary (2013: 5), is in agreement with Nekoo and Shohreh (2013: 87) that different quantities of GAC beds affect the amount of COD concentrations removal in the GAC adsorption process. Wu (2004) also agrees with Ushakumary (2013: 8) that the availability of GAC exposure/capacity optimizes the removal of COD concentrations during the GAC filtration process. Another relevant variable that the three authors note for impact on the optimisation of GAC filtration is the contact time. Ghodale and Kankal (2014: 38) concur that the increase in GAC contact time with solute during the filtration process results in optimal removal of the concentrations of the contaminants. Furthermore, Davids (2006: 109) highlights another variable in reporting that the optimal removal of COD concentrations in a solution can be achieved when the pH of the filtered solution contains more hydroxyl ions and fewer hydronium ions. HAYCARB Activated Carbon Solutions (2020) mention the following advantages of using a GAC adsorption method:

- Removal of both organic and inorganic substances from the effluent,
- reduction of residual substances that contain chemicals from the effluent,
- large scale removal of residual COD concentrations over sufficient low of a solute,
- reduced land area requirement for GAC implementation,
- reduced sensitivity to daily flow variations,
- simplicity of implementation and operational flexibility control at a plant level, and
- it is dust-free and enables easy filtration of the treated effluent.

Thus, different CGAC filtration procedures investigated during the conduct of this research study by manipulating the different variables mentioned above with the aim of identifying the most significant procedure that results in an optimal removal of COD concentrations in the effluent discharged by the WCM plant.

1.8.4 Effluent quality improvement

Industrial effluent is considered to be one of the most significant sources of water pollution. The United Nations (2017) argues that effluent discharged into coastal areas, rivers and lakes results in serious problems and causes negative effects for the ecosystem and consequently human life. Moreover, Makgae (2011) claims that in the past, industry was solely geared towards economic aspects and totally neglected ecological issues. In short, industrial activities release huge quantities of wastes into the environment.

The municipal bylaws regulating effluent in South Africa are considered to be strict according to Hot Water Treatment (2013), as the industries are currently facing unprecedented discharge fees in the view of this author. In addition, South Africa introduced a range of additional legislative measures aimed at improving the quality of the environment (South Africa, 2013: 7). It is worth noting that effective regulation of hazardous waste requires sufficient compliance and enforcement capacity on the part of Department of Environmental Affairs (Makgae, 2011). The author also elaborated that the waste management and improvement in South Africa are currently governed by means of a number of pieces of legislation, which include:

- The South African Constitution Act 108 of 1996,
- Hazardous Substance Act 5 of 1973,
- Environmental Conservation Act 73 of 1989,
- National Water Act 36 of 1998,
- National Environmental Management Act No. 107 of 1998,
- Minerals and Petroleum Resources Development Act 28 of 2002,
- Air Quality Act 39 of 2004, and
- National Environmental Management: Waste Act 59 of 2008.

This study sets out to develop a suitable industrial procedure to reduce the COD concentrations in the effluent discharged by the WCM plant in order to consistently meet regulatory requirements.

1.9 Methodology and research design

According to Williams (2007: 66), research methodology involves systematic methods adopted by a researcher to answer the research questions of the particular research study. Kumar (2008: 6) also acknowledges that research methodology is concerned with identifying a systematic approach to find solutions to the research problem of a study. Furthermore, Basson and Uys (2005: 8) mention that when doing research, there are two methodological models, namely quantitative and qualitative methodology.

Quantitative research is based on the measurements of quantity or amount, whereas qualitative research is concerned with phenomena involving types or qualities (Kothari, 2004: 3-4). With specific reference to laboratory experiments, Dijkstra, Forbes, and France (2005: 551) mention that quantitative research involves an empirical research study whereby the experimental data is used to describe a correlation or a relationship between a dependent and independent variable. Heppner, Owen, Thompson, Wampold, and Wang, (2016: 117) add that laboratory experiments examine 'causality' by systematically varying or altering an independent variable or a set of independent variables. This study is focused on laboratory experiments to establish a more efficient and effective procedure for the reduction of COD concentrations in the WCM effluent. The laboratory experiments in this study involve stipulating the independent variables of the effluent to improve the quality of effluent discharged by the WCM.

Experimental designs are sometimes known as the scientific method because of their popularity in scientific research (Muijs, 2011: 11). According to Taylor (2005: 95), the scientific method involves manipulation of experimental variables under rigorously controlled conditions. Duckworth and Hoffmeier (2016: 38) propose that Six Sigma DMAIC methodology can be used in scientific methods to find solutions to the problem of interest. This methodology uses the scientific method and quality tools to provide solutions to a problem (Elshennawy, Gupta, Mcshane-Vaughn, & Walker, 2009: 319). Furthermore, Juneja, Sharma, and Verma (2014: 1065), claim that Six Sigma may be used as a research methodology during the process of academic research. Therefore, this study deduces that Six Sigma is appropriate for the process improvement undertaken.

Tayntor (2003: 23) state that the five phases of the DMAIC may be referred to as a process improvement tool. DMAIC is an acronym for five interconnected phases of a process improvement study namely 'Define', 'Measure', 'Analyse', 'Improve' and 'Control'. The Six Sigma DMAIC approach employs data for improving, optimizing, and stabilizing processes of interest (Gejdos, 2015). The DMAIC tool is thus adopted as a framework in this study in an endeavour to improve the quality of effluent discharged by WCM by reducing the COD concentrations.

1.10 Data collection and analysis

The analytical results of experiments performed in the laboratory at the WCM plant in the laboratory at a WCM plant in the WC are the primary data of this research study. Systematic and stratified random sampling are the sampling methods used in this research to ensure that the data interpretation of the findings achieved from the effluent samples is representative of the WCM effluent.

The samples collected were tested for COD concentrations before and after CGAC treatment, using a benchtop photometer. Both tests have been conducted in duplicate. Three different types of analytical tests (one for each variable) were analysed. In order to decide whether the procedure resulted in optimum removal of COD concentrations from the effluent. The above-mentioned three independent research variables were artificially manipulated and their influence on COD concentrations were evaluated with the aim of establishing whether the procedure resulted in optimal removal of COD concentrations from the effluent. The result of the observations for each treatment were documented, including the COD concentrations result.

The representative effluent samples were collected from the effluent reservoir and analysed for COD concentrations before treatment. The sampling methods, data collection methods and data analysis methods applied in this research study are described and discussed in greater detail in Chapter 4 and Chapter 5 of this thesis. A quantitative approach was followed because of the analytical nature of this study, namely laboratory experiments performed during this research.

1.11 Data validity

According to Haradhan (2017: 59-60) both internal validity and external validity are crucial when performing experiments to provide the reader with assurance that the conclusions of the research study are correct. Babbie and Rubin (2010: 83) define validity as the extent to which an empirical measure adequately reflects the real meaning of the concept under consideration. Franzen (2002: 34) state that "…validity in an empirical sense may be defined as a statistical relationship between the results of a particular procedure and characteristics of interest". Furthermore, Taylor (2005: 2) adds that validity in empirical studies is proven by performing a validation procedure which involves the close scrutiny of logical arguments, and gathering empirical evidence to assure that the method followed yields accurate and valid results.

Internal validity the measure of the consistency of the measurements or the degree to which the instruments measure the same way each time that it is used (Breakwell & Rose, 2006: 73), under the same specified conditions with the same subject. The reliability of the data collected by this study is assured by:

- Keeping the laboratory temperature constant throughout the conduct of the research,
- keeping the analytical balance used at controlled room temperature, to prevent moisture interference and eliminate random errors,
- storing the GAC under controlled conditions that are specified by the supplier, and
- weighing the amount of the GAC used in the experiments accurately on a pre-calibrated analytical balance.

In this study validity measures were taken to provide the assurance that the quantitative primary data was truly representative of the population and secondly, to ensure that the data analysis and interpretation of the data was valid, accurate, trustworthy and repeatable.

1.12 Ethics

Gray (2011: 63) refers to ethics as the "rules of conduct" in research. Ethics is a word that is derived from the Greek word "ethos", meaning one`s character and ethics are linked to morality (Bless, Higson-Smith, & Kagee, 2007: 140). Ethics is the branch of philosophy that addresses questions about morality; research ethics is concerned with moral behaviour in research contexts (Wiles, 2013: 4). The following ethical procedure was observed in this research study:

- The researcher obtained approval from the organization on which the research was to be conducted,
- the researcher signed a confidentiality letter on the security of information shared or found, that might cause harm to the organization of interest,
- the researcher adhered to the obligation of ethical practice during the research,
- in this research, the findings were reported honestly, and
- no data was fabricated to support a conclusion.

1.13 Research assumptions

Dantzker and Hunter (2012: 51) define research assumptions as a statement of concepts that are believed to be true with little or no evidence supporting it. Burns and Grove (2011: 48) argued that assumptions are things that a researcher takes for granted and accepts as valid without concrete proof. The research assumptions of this study are as follows:

- Sampling was performed following with the approved SOPs,
- the same grade experimental apparatus was used throughout this study,
- all analytical instruments to be used were calibrated and verified before use,
- the results of the measurements obtained were recorded accurately and kept safely, and
- the results were accurate, and interpreted following a standard operating procedure.

1.14 Research constraints

According to Koh and Owen (2000: 38), research constraints refer to uncontrollable events that might interfere with the results of the study, restrictions related to restrictive weaknesses that present potential boundaries to the validity of the result, or a limit to which the study was significantly confined. The data was collected from a WCM plant that is located in the WC. The results and findings of this study cannot be generalised to different organizations that do not have an identical process and environmental factors.

1.14.1 Limitations

Kuiper (2009: 255) defines limitations as the inadequacies of the study that cannot be controlled by a researcher. Koh and Owen (2000: 91) add that limitations are uncontrollable events that may interfere with the results of a study. Limitations may include any changes that take place in samples due to the time duration from when the data needs to be collected and measured, as well as weaknesses in the measurement instruments. Limitations are the variables or boundaries of the research established by factors or people other than the research.
The following limitations were part of this research:

- The availability of literature which directly speaks to the selected research topic,
- absence of past studies in the selected study environment. and
- this study was only performed on one WCM plant located in the WC region in SA.

1.14.2 Delimitations

Kuiper (2009: 255) states that delimitations are the characteristics that limit the scope of a study and it defines the boundaries of a study which are controlled by a researcher. Sharma (2014: 96), writes that delimitations indicate what is going to be included in the study. The delimitations of this study will consist of the following:

- Participation in this study included only one WCM in the laboratory,
- the research was limited to a WCM plant that is located in the WC region, and
- this research focused on COD removal using adsorption method, but it focused on using the laboratory GAG filtration technique.

1.15 Chapter outline

- **Chapter 1- Scope of the research:** This chapter outlines the aim of this study namely, to reduce COD concentrations from the effluent discharged in WCM plant located in the WC Province. This chapter briefly explains where the study was conducted; the research environment, including the significance of the study; the research statement, questions and objectives; the rationale and conceptual framework; research design and methodology; data collection and analysis; ethics; research assumptions and constraints; and the research plan.

- **Chapter 2- A holistic perspective of the research environment:** This chapter elaborates on the WCM plant processes and their outputs, systems implemented in the WCM plant, quality management of the effluent and the government regulations applicable to this industry. The main focus is on the COD concentrations of the effluent generated by the WCM process stages, and their impact on the composite of the effluent COD concentrations prior to discharge into the municipality drains. This chapter also navigates the current practices or measures of controlling the quality effluent (COD concentrations) adopted by the WCM plant.

- **Chapter 3- COD removal literature review:** A brief high level review of the importance of maintaining low COD content from the discharged effluent and its effect on the environment is provided in this chapter. Accompanied by an in-depth literature discussion on the concept of wet corn processing effluent (COD) quality improvement in laboratory-scale. The relevant scientific ways of reducing CODare explored, to establish a suitable method for this study. The methodology selected to explore the effectiveness of the selected COD removal method is presented, namely Six Sigma DMAIC.

- **Chapter 4- Research Design and Methodology:** This chapter starts by presenting theories and worldviews of research. Following this, details of the research design and research methodology that are relevant in this study are provided. The discussion of research design includes a description of all the applicable data collection and interpretation methods. The discussion on research methodology showcases the systematic approach of collecting, analysing, interpreting and making conclusions about the results obtained from the collected data. The ethics and data validity of this study are provided.

- **Chapter 5- Data interpretation and analysis of the results:** This chapter will present the data interpretation and analysis as described in Chapter 4, following the Six Sigma DMAIC methodology. The first data analysis conducted on the raw effluent COD using systematic random sampling and applicable tools to describe the data is described. Then further data, collected using stratified random sampling and used to test the effectiveness of the selected COD removal scientific method, is presented. The validity of the study is discussed, together with the risks of the method as tested using Failure Modes Effects Analysis (FMEA).

- **Chapter 6- Conclusion and Recommendations:** In this chapter, the research problem, research questions, and research objectives are revisited with the purpose of evaluating if they have been achieved. Chapter 6 concludes by providing suggestions for further studies that can be conducted in this field for more improvement, research recommendations and conclusion.

1.16 Chapter 1 summary

This chapter presented a high level context and background of the research environment with the justifications for performing this study, which took place at a WCM plant in the WC. The focus of this study rested on the COD level of the effluent discharged in the WCM plant. The aim of this study was to reduce the effluent COD level, in order for it consistently meet the regulatory effluent discharge requirement. To achieve the research objective, the quality improvement methodical approach known as Six Sigma DMAIC was adopted. Six Sigma's DMAIC provides a framework and directs the sequence of laboratory experiments and data analysis that are performed in a process of finding a way to optimise the removal of the COD from the WCM discharged effluent.

In the next chapter a holistic background to this research project is presented in order to provide clearer insight into the environment where this research took place, and the research problem.

CHAPTER 2: HOLISTIC OVERVIEW OF THE RESEARCH ENVIRONMENT

2.1 Introduction to Chapter 2

This chapter serves as the natural progression from Chapter 1 as it provides the reader with an in-depth holistic environmental overview relevant to COD concentrations in the effluent generated by the WCM plant in the WC. It commences with an explanation of how the water is formed and sustained and presents the profile of water contained on the planet. Thereafter, it discusses the availability of freshwater and factors that influence freshwater scarcity. An effluent profile is then presented and the consequences of non-conformity of the COD concentrations in the discharged effluent are highlighted. This chapter concludes with a discussion of the impact of the reusing the in-process water in the WCM processing stages on the quality of COD concentrations.

2.2 importance of water management in South Africa

Water Wise Rand Water (2018) commented that it is an undisputed fact that all the living things on this earth are dependent on water for survival. The USGS Water Science School (2016) states that the water on this earth is old and constant; therefore, water cannot be increased nor decreased and this emphasises the importance of recycling water. Frerot (2011: 20) reports that the earth's surface is covered with approximately 72% water, of which 97% is contained in in the sea and 2.5% is underground water and water that is frozen around the north and south poles and mountain glaciers. The author concludes that only 0.3% of the fresh water is available for human use. Logan and Power (2010: 25) provide the image of hydrologic cycles in on **Figure 2.1** below.

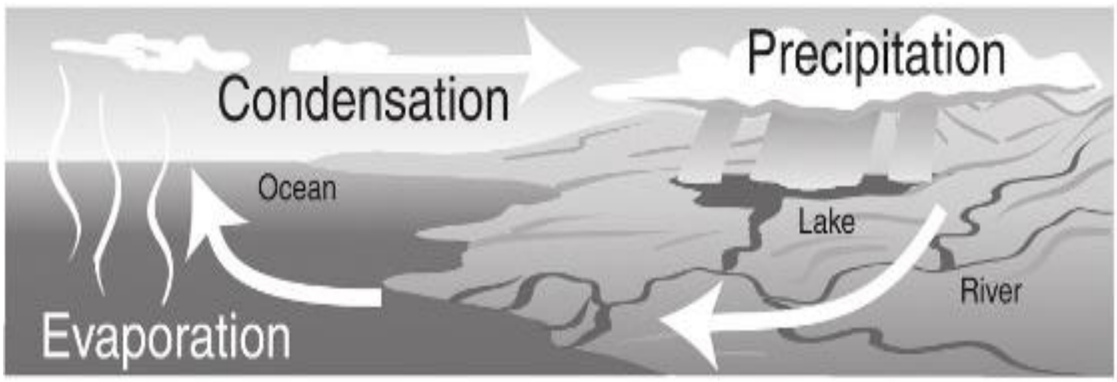

Figure 2.1: Hydrologic cycle (Logan & Power, 2010: 25)

Water News (2018) argues that water shortage is a major concern in SA, and that the WC worst drought in 2016 and 2017. Accordingly, the City of Cape Town declared increasing water restrictions from 2016 to 2018, which included an increase in water consumption fees. It is noteworthy that the World Cup

Legacy Report (2011) states that SA receives an annual rainfall of 492 mm, whereas the rest of the earth receives 985 mm, thus the percentage of rainfall in SA is a mere 50% compared to the global average rainfall. This agrees with Water News (2018), which also reported that since January 2016 the rainfall per meter square received in SA ranges from 300 to 500mm per annum, which is much less than the worldwide average.

South Africa has thus completely revised its legislative and policy framework, to govern effluent quality discharged into the oceans and dams due to increasing COD concentrations, effluent volumes and decrease in the availability of freshwater (Water Wise Rand Water, 2018). Hence, SA made it mandatory, through the promulgation of the South African Water Act No. 54 of 1956 that the quality of the effluent should be treated to the required standard before it is discharged into municipal treatment dams (Mema, 2010: 60-61).

2.3 Management of effluent in South Africa

In the past, industrial effluent regarded as an unimportant and troublesome by-product of a manufacturing process (Water Wise Rand Water, 2018). However, since 1996, South African municipalities implemented a regulatory requirement for the legislation of the discharge of effluent. This legislation specified the minimum requirements of discharged effluent intending to protect municipal sewage pipes from damage caused by the effect of the pH of effluent or flammable or corrosive effluent (Water Wise Rand Water, 2018). A COD concentrations regulatory requirement was implemented to avoid overburdening the local treatment works due to public and environmental pressure (Hot Water Treatment, 2013). Recently Water Wise Rand Water (2018) reported that the municipal limits have become even stricter on South African industries by tightening these requirements. This was due to increased volumes of the discharged effluent and COD`s in the effluent received in the municipality effluent treatment facilities (Gumbo, Malaka, Nare & Odiyo, 2010: 1). The allowable specifications in the discharge effluent are illustrated in **Table 2.1** below.

Table 2.1: Effluent legal specification regulations (Department: Government Communication and Information System Republic of South Africa Department of Water Affairs, 2014)

Variables	Not less than	Not to exceed
Temperature at point entry	0 ^{0}C	40 ^{0}C
Electrical Conductivity at 25 ^{0}C	-	500 mS/m
pH value at 25 ^{0}C	5.5	12.0
COD	-	5000 mg/L

Table 2.1 above indicates the legal quantity (stipulated measures) that are permitted by the South African Department of Environmental Affairs in the discharged effluent. These regulations are used to calculate the effluent charge should the plant fail to meet the requirements. Industrial effluent charge is explained in the section that follows.

2.4 Industrial effluent charges

A formula is used by City of Cape Town to calculate the effluent charge and then invoice an organization for the volume of freshwater used and the amount of effluent generated, which takes the results of all the variables highlighted in **Table 2.1** into consideration (Water Wise Rand Water, 2018). If both the COD concentrations and the freshwater consumption are very high, the organization is levied a significantly high bill (Gwadla, 2018). Therefore, the WCM embarked on a project of reducing the cost of the water consumption by reusing the process water; however, this consequently resulted in even higher COD concentrations readings (Ndlovu, 2013: 1). When COD concentrations are extreme in the effluent discharged by the industrial plants, it requires more chemicals, more energy and excessive use of other materials in the effluent treatment plant to treat it to the acceptable levels (Gwadla, 2018). Therefore, to overcome the cost of the effluent treatment, the Department of the Environmental Affairs has generated a formula that they use to bill the discharged effluent from different industries which is inclusive of the non-conforming COD concentrations charge (Department: Government Communication and Information System Republic of South Africa Department of Water Affairs, 2014).

The industrial effluent charge cost is a penalty associated with not meeting the effluent specification limits imposed by the municipality of a city in South Africa (Department: Government Communication and Information System Republic of South Africa Department of Water Affairs, 2014). There is an additional cost associated with of the non-conforming COD concentrations in the discharged effluent, and this cost is added on the cost of effluent management (Jackson, 2017). Historical records at the WCM indicate that all the other variables that were measured in the WCM discharged effluent conforms to the environmental regulations, except for COD requirements (Gwadla, 2017). This emphasizes the importance of the development of an appropriate method to reduce COD concentrations since this will not only reduce the risk to aquatic species but also further reduce the WCM penalties issued to WCM plant due to noncompliant effluent. (Jackson, 2017).

According to Van Schoor (2005) the legislation issued pertaining to the discharge of industrial effluent stipulates that the COD level should not exceed 5000 ppm, to prevent industries from discharging high COD concentrations levels in the effluent to the effluent treatment plants. Hassan and Schreiner, (2011: 77) report that the COD limit was made tighter due to increasingly excessive volumes of poor quality effluent discharged into effluent treatment plants because of increased industrial activities. As a result, municipalities were experiencing financial problems treating the effluent in accordance with the requirements before it could be released into the rivers or oceans (Van Schoor, 2005). As the results, in addition to the tight chemical specifications in the discharged effluent, the effluent charge fee was introduced, calculated using the excessive values of the specified chemical contents into the tested effluent per plant (Hot Water Treatment, 2013).

The next section provides a background to the WCM processing to clarify the internal environment and the effluent process profile.

2.5 Background to the WCM processing plant

The WCM plant at which this research study took place is a significant contributor to industry on the African continent, as it processes about 30% of 600 000 tons of corn per annum into gluten 20, gluten 60, corn germ, starch and starch-based products (Jackson, 2017).
The wet corn processing plant located in the WC consists of a total of eight departments to ensure that its business unit is effective and sustainable (Jackson, 2017). The eight departments are:

1. Quality Control (QC),
2. Health, Safety, and Environmental (SHE),
3. Production (Wet Mill and Glucose Refinery),
4. Engineering,
5. Logistics (outbound and inbound),
6. Human Resources,
7. Process Engineering, and
8. Finance.

Jackson (2017) states that all these departments work together to ensure that the organization makes a profit, creates employment and takes good care of its stakeholders. The most important function of the QC Department, working in conjunction with the SHE Department is responsible for the implementation of systems to assure all the stakeholder`s requirements are met (Gwadla, 2017). These stakeholder`s requirements include compliance with the regulatory specifications for the COD in the discharged effluent (Gwadla, 2017). A brief discussion of the management systems implemented at the WCM is presented below.

2.6 International organisational standards systems

The WCM plant has been certified to ISO 14001:2015, which is a standard that provides guidance on an environmental management system to ensure that the activities carried out by this organization unit do not cause harm to the environment (Dentch, 2016: 1). ISO 9001:2015, provides guidance on quality management systems to ensure that the quality of the product meets regulatory and customer requirements (Purushothama, 2015: 1).

It is a requirement for any certified organization or plant to comply with ISO standards to maintain certification (Jackson, 2017), thus it is also important to strive for continual improvement of the ISO implemented systems, as outlined by the standard, to ensure that it is effectively implemented (Gwadla, 2017). According to the guidelines of the National Environmental Management Act No. 107 of 1998, the WCM plant is acquired to comply to the legal requirements established by the Environmental Affairs for COD content in the discharged effluent (South Africa, 2013).

The implementation of the aforementioned systems at the WCM plant demonstrates the organisation's commitment to striving for success in its business (Gwadla, 2017). The WCM organization has also pledged to comply with the applicable government legislation, such as national, provincial and local environmental, quality, and health and safety legislation and regulations (Jackson, 2017). Aligned with this commitment, this research study sets out to assist the WCM plant to improve their current system. A description of the wet corn processing system in operation at the WCM plant is discussed in the section that follows.

2.7 Introduction to WCM

Ndlovu (2013 :17-19) describes wet corn processing as a process of softening dry corn with water, then grinding it before it is separated into its various corn components. This description of the process is aligned with the view of Gunasekaran, Yaghmour and Yasri (2015) who add that a wet corn process is employed to fractionate wet corn into its various corn parts, namely corn germ, gluten 20, gluten 60 and corn starch. Wet corn starch is further modified to four different grades of glucose syrup (Taylor, 2004: 155).

Lakdawala and Lakdawala (2013: 90-91) comment that during this process the WCM industry utilises and generates huge quantities of potable water and effluent respectively. The authors add that effluent generated by the WCM is rich in proteins, both soluble and insoluble proteins, and hydrocarbons. Ross (1989: 231) argues that the characteristics of the effluent generated by the WCM vary depending on the compounds saturated in the effluent, which include proteins and carbohydrates that give rise to the levels of COD in the effluent.

Ndlovu (2013: 24) notes that effluent generated by the corn milling industry is classified as highly contaminated because it contains significantly high levels of protein and starch (also known as carbohydrates). Specifically, the effluent generated by corn processing contains traces of organics, namely soluble and insoluble proteins, fats and hydrocarbons generated during the wet milling processes (Babuna, Orhon, Ovez & Ozgun, 2002: 539). Ross (1989: 231), states that "organizations that generate effluent are held responsible to comply with the regulations of the requirements of the quality of discharged effluent, to prevent harm to the environment." The quality requirements that Ross (1989: 231) refers to are associated with the following variables in effluent: pH, conductivity, total solids, COD, temperature, and fats.

2.8 WCM stages

The wet corn process in the corn milling begins with the dry corn kernels being soaked in steep tanks filled with dilute aqueous sulphur dioxide solution (Chaney, Eckhoff, Haken, Hicks, Niu, Singh, Tumbleson, & Yang, 2005: 421). According to Taylor (2004: 154-157), the wet corn milling system consists of many interconnected process flows as it produces many different types of products. The main process stages include:

- Corn inbound quality inspections and storage,
- Corn steeping,
- Wet corn milling,
- Corn components separation,
- Starch modification or saccharification, and
- Glucose refinery.

Each process step is explained in the following sections.

2.8.1 Dry corn inbound quality inspections and storage

In the first stage, the dry corn received via rail is sampled and evaluated to confirm that it conforms to predetermined quality specifications before use (Gwadla, 2017). After all the critical tests establish compliance with specifications, the accepted corn is cleaned to remove all unwanted particles and then stored in silos, ready for the second stage. In an interview with Jackson (2017), he commented that the moisture content of dry corn helps to determine the time required to steep the corn sufficiently.

2.8.2 Corn steeping

The second stage (which is also the first active step in the wet milling of corn) is called steeping (Serna-Saldivar, 2019: 528-529). This process stage involves softening of the corn kernel by soaking in a water-based sulphur dioxide solution to facilitate the separation of the various components of the corn kernel (Chaney, *et al*., 2005: 421). The steeping stage marks the start of the wet milling process and commences when the corn is placed in warm water and sulphur dioxide under controlled conditions to aid the softening of the corn kernels in accordance with a specified procedure (Gunasekaran, *et al*., 2015).

2.8.3 Steeped corn grinding and corn component separation

After the corn have been steeped accordingly, the steeped corn are separated by grinding them (Chaney, *et al*., 2005: 421). When the grinding process is completed, the ground mush is spun down in a centrifuge to separate the mush to the components of the corn kernels (Taylor, 2004: 154-157). The corn kernel components are corn germ, gluten 20, gluten 60 and corn starch (Jackson, 2017). The separation stages are explained below.

- **First separation stage**: The corn germ is separated from the endosperm fragments in hydro-cyclones, using different densities and then dried (Gunasekaran, *et al*., 2015). All the process water generated by this process stage is deposited into the main effluent sump (Jackson, 2017**).**

- **Second separation stage**: The remaining kernel fragments undergo a further milling process to enhance the separation of gluten 20 from the slurry, which is essentially the external cover of the corn kernel (Rahman, 2007: 120). The gluten 20 is dried in accordance with specified conditions and whatever process water that is extracted during this stage is also deposited into the main effluent sump (Jackson, 2017).

- **Last separation stage**: The final residual slurry consists of gluten 60 and wet starch. It is then further separated in the primary separator apparatus on the principle of the difference in their respective densities, as gluten 60 is less dense than starch (Chaney, *et al*., 2005: 421). Thus the lighter particles of the gluten 60 dissociate from the heavier particles of corn starch (Demirbas & Gupta, 2010: 80); then gluten 60 is dried and stored. The corn starch component that is left is dried, and another portion of wet starch is further processed into different glucose syrup products (Jackson, 2017). All the effluent generated by these three different separation stages is collected and measured.

2.8.4 Wet starch modification

Stage five is a liquefaction process stage also referred to as a pre-saccharification process stage (Jackson, 2017**)**. During this stage the wet starch is prepared for the saccharification process stage by correcting its dextrose equivalence, pH and density (Chaney, *al.*, 2005: 421). After the liquefaction process stage has been completed, the saccharification occurs, which involves conversion of the liquefied starch into a desired glucose syrup grade, using selected enzymes under controlled variables (Demirbas & Gupta, 2010: 80). All the process water generated by these two process stages is first transferred into the effluent pH correction tank to correct its pH before it is discharged into the main effluent sump (Jackson, 2017**)**.

2.8.5 Glucose refinery

After the saccharification process has been completed, the product undergoes glucose syrup refinement which is the final process stage (Demirbas & Gupta, 2010: 80). Here, the glucose syrup by-product is first filtered to remove all the fats and proteins from it (Jackson, 2017). The filtered, fat-free and protein free product then undergoes an ion-exchange process to purify the product by removing all the ions (Ndlovu, 2013: 21-23) The then ion-free product is concentrated by means of evaporating all the excess water before it is stored in the final product storage tanks (Gwadla, 2017).

The effluent generated by the ion-exchange is transferred to the effluent pH correction tank before it is discharged into the main sump, and all the other effluent generated by fat and protein removal, evaporation and glucose spillage is collected in the main effluent sump (Jackson, 2017).

The diagrams presented below summarises the different stages of the effluent generated in the WCM processing. **Figure 2.2** depicts process flow from dry corn delivery, corn processing, corn by-products refinery and final product storage, as described in the foregoing section.

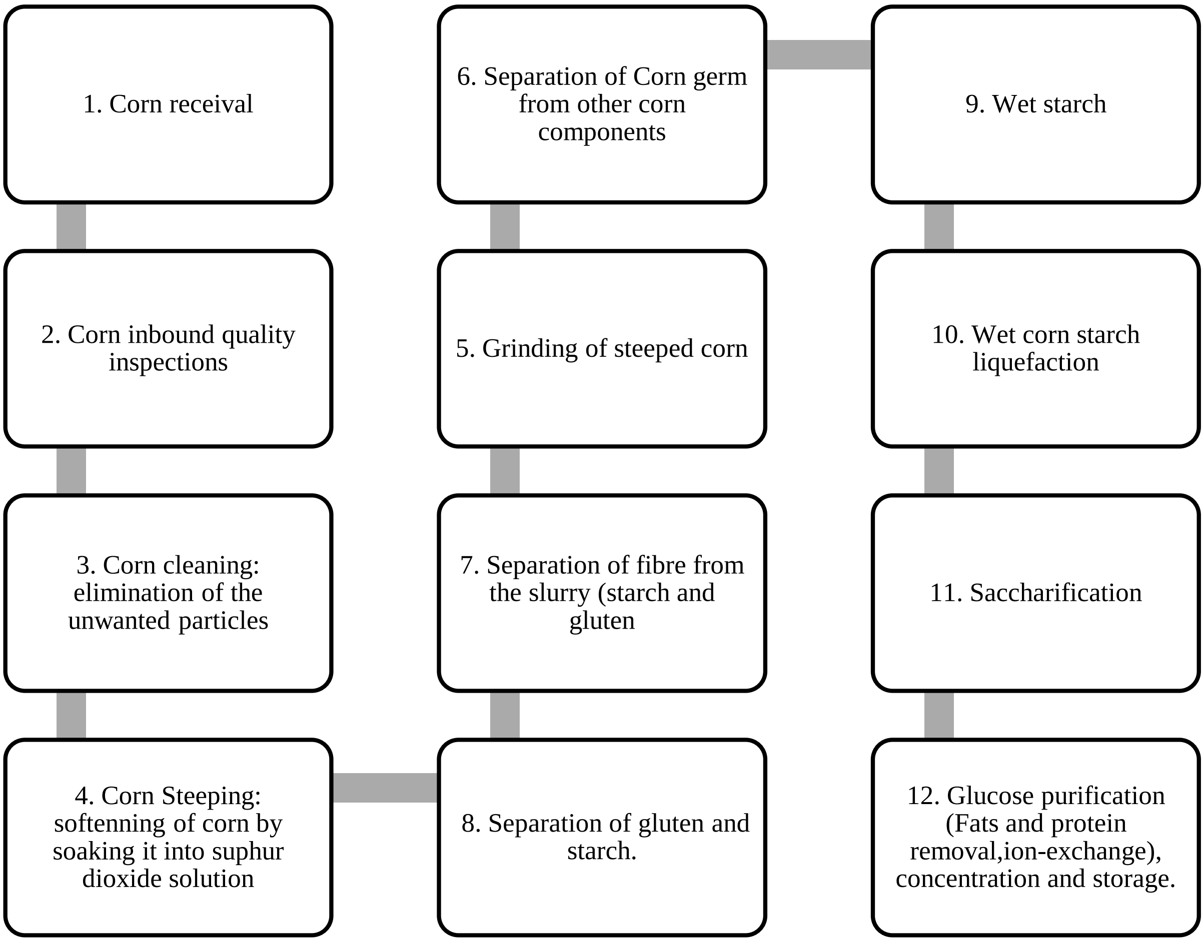

Figure 2.2: Main process stages of corn processing (Ndlovu, 2013: 17-23)

The quality of the effluent generated by each stage and its implications in to the environment are discussed in the following section.

2.9 Effluent generated by WCM processing stages and their environmental impact

Consistent with the discussion presented in the preceding section, Gunasekaran, *at el*. (2015) confirm that the individual process steps where the effluent is generated in the wet corn milling process are as follows:

- Steeping,
- Corn germ processing,
- Gluten 60 processing,
- Gluten 20 processing,
- Dry starch processing,
- Wet starch conversion,

- Glucose syrup filtration,
- Glucose syrup purification – ion exchanger,
- Glucose syrup concentrations – evaporation, and
- Spillages and cleaning water.

At the WCM plant, effluent samples are collected on a daily basis every two hours from each one of these effluent streams, thus each effluent stream serves as a sampling point. The samples are analysed in a laboratory to determine their quality in terms of COD. The effluent quality analysis includes the measurements of TOC, pH and conductivity. All the effluent generated in the wet milling and refinery processes is then collected in the effluent tank. The collected effluent is then dosed with caustic solution or hydrochloric acid to correct the pH to the required specification; the corrected effluent is then discharged into the main effluent sump, prior to discharge to the municipality (Jackson, 2017).

2.10 The impact of poor quality management of effluent

Water is a vital resource for living organisms and for other activities such as manufacturing, farming and some human activities such as cooking (Rampal & Sharma, 2018: 548). Despite the importance of water, Atem and Otieno (2016: 61) acknowledge that water is a very poorly managed resource, especially in developing countries. High concentrations of chemical compounds in discharged effluent have had a harmful effect on the environment, particularly in the 21st century (Rampal & Sharma, 2018: 548). Essentially, this is water pollution.

The Department of Environmental Water Affairs established and published the standard requirements pertaining to the content of chemical compound in discharged effluent (Department: Government Communication and Information System Republic of South Africa Department of Water Affairs, 2014), since the discharge of poor quality industrial effluent alters the natural balance in the environment (Rampal & Sharma, 2018: 548). An imbalance in the environment results in an array of detrimental effects in South Africa including massive fish mortalities (Selvarajan, Sibanda & Tekere, 2015**)**, thus an approach to managing effluent quality from an environmental pollution control perspective evolved as a result.

The discharge of nonconforming COD concentrations can compromise the quality of the treated effluent at municipal effluent plants, as the effluent will require more resources and makes it more expensive to be treated to the required specifications before it is released into the rivers or oceans (Edokpayi, Msagati, Odiyo & Popoola, 2015: 7301)**.** The release of high COD treated effluent into the rivers causes lack of oxygen inside the water which is extremely toxic to aquatic species (Igbinosa & Okoh, 2009).

Therefore, it is critical for the industries that discharge effluent to comply with the COD requirement of the discharged effluent which is a maximum of 5000 ppm (Van Schoor, 2005).

Significantly, South Africa is a country with limited water supply and the water demand places substantial pressure on water service providers in the country (South Africa 2014). Hence, the monitoring of good quality of the effluent is required to sustain the water cycle without compromising the ecosystem of the aquatic species and causing harm to the environment (Frerot, 2011: 20).

2.11 WCM quality assurance and control overview

Gwadla (2017), speaking in his capacity as production manager at the WCM, mentioned that to overcome the negative implications of poor quality management of the effluent, a quality effluent management system is in place. Hence, the quality control analytical chemistry laboratory in the WCM plant is part of the system to ensure that the effluent generated in the WCM is measured against the standard requirements (Gwadla, 2017). The interviewee further explained that effluent samples are then sent to the QC chemistry laboratory for analysis; the effluent is analysed for the following variables: total TOC or COD, pH, and conductivity.

Liptak (2003: 1229) discovered that COD concentrations can be directly measured using an online or in-line automated detector and can measure from 0 to 5000 ppm COD ranges. Rhosonics (2017) add that the on-line COD detector is a quick automatic method that provides a number of results in a period of time compared to off-line or manual detection. Despites of what the last two authors had highlighted, the WCM plant employs the manual method to measure the COD.

In a separate interview with Jackson (2017), speaking in his capacity as quality manager of the WCM, it was highlighted that the plant has a dosing system for correcting the pH before discharge; there was a theory that the volumes of the effluent generated by the processes with low COD concentrations could be diluted to reduce the COD of the total effluent to the required levels. However, the data gathered from the laboratory using TOC analysis contradicted or disproved the theory; it was indicated that the volumes were not sufficient to have an impact on reducing the COD levels in the total effluent (Gwadla, 2017). TOC is a quick accurate COD analysis test; provided that the coefficient factor is taken into consideration to correct the reading (Bai, Carpenter, Hwang, Ikhmayies, Li, Monteiro, Peng, and Zhang, 2013: 217).

Gwadla (2017) explains that to assure validity of results, external calibration which includes reliability tests of the instruments is performed annually by an external SANAS accredited service provider.

Moreover, the daily and monthly instrumentation maintenance and internal verification using an approved reference standard are performed internally. These are the instruments employed in this dissertation, namely pH meter, analytical balance, conductivity meter, and a benchtop photometer analyser unit. These instruments were used during the conduct of this research to establish an optimal GAC procedure for reducing the concentrations levels of the effluent discharged from the WCM plant to consistently comply with the regulations.

2.12 Chapter 2 summary

This chapter commenced with the presentation of an overview of the research environment focused on the water crises that are currently being experienced in SA. Thereafter, factors pertaining to the quality of the effluent discharged into municipal effluent treatment facilities and the implications of poor management of effluent quality were discussed. Finally, the chapter provided background information regarding the WCM plant, processes and systems in the context of this research study.

Chapter 3 will present a reader with current literature focused on the methods and procedures of improving the removal of the COD concentrations in the effluent.

CHAPTER 3: LITERATURE REVIEW

3.1 Introduction to Chapter 3

This chapter offers the reader a detailed theoretical insight into the main subject of this study: reduction of the COD concentrations in WCM effluent, through the evaluation of literature. Fink (2014: 3) describes a research literature review as a reproducible method for identifying and evaluating completed work produced by other researchers. Machi and McEvoy (2016: 7) state that a literature review presents a logically argued case, founded on a comprehensive understanding of the current state of knowledge about a topic of study. Keeping in mind what the authors mention above pertaining to the literature review, the conceptual process followed in the literature reviewed in this chapter is illustrated below:

- Background and motivation of this study,
- possible existing solution to the research problem, and
- application of Six Sigma DMIAC and quality tools.

The discussions that make up this literature review are presented in a manner that is focused on achieving the main objective of this study; which is to establish a procedure to optimally reduce the COD concentrations in the effluent discharged by the WCM plant.

3.2 Background and motivation to the research problem

Ndlovu (2013: 17) describes wet corn processing as a process of softening dry corn in a solution before it is separated into its various corn components. Gunasekaran, *et al.* (2015) mention that a wet corn process is employed to fractionate wet corn into its various corn parts namely, corn germ, gluten 20, gluten 60 and corn starch. Lakdawala and Lakdawala (2013: 90-91), comment that during this process the WCM industry utilises and discharges huge quantities of potable water and effluent respectively. Ross (1989: 231) notes that the characteristics of the effluent generated by the corn processing is rich in proteins and carbohydrates which increases the concentrations of COD in the effluent. Garcia Einschlag (2011: 8) states that effluent generated by the wet corn milling industry is classified as highly contaminated because it contains significantly high levels of protein and starch. Ahsan and Ismail (2019: 2) pointed out that the strategies to ensure proper treatment of the COD concentrations in the effluent must be developed to ensure conformance to legislated standard requirements.

3.3 A possible existing solution to the research problem

3.3.1 Introduction to adsorption

Chaturvedi, Deshmukh, Ingole, Joshi and Kulkarni (2014: 1211) state that the COD concentrations in effluent is a parameter used to categorise the state of the effluent (as being of good quality or bad quality). According to the Department of Water Affairs and Forestry (2000), management of the COD concentrations in effluent involves measuring, analysing and controlling the COD concentrations within the required specification. Ahsan and Ismail (2019: 2), assert that control of COD concentrations in effluent can be achieved using physical and chemical or biological methods. Ademiluyi, Amadi, and Amakama (2009: 39) recommend that the physical and chemical adsorption method is the most efficient and sustainable for COD removal. Bhandari, Ranade, and Sorokhaibam (2016) argue that the adsorption by physical and chemical method is a well-established technique for removal of the COD concentrations in the effluent.

Bonilla-Petriciolet, Mendoza-Castillo and Reynel-Avila (2017: 2) mention four advantages of chemical and physical adsorption methods, namely its low capital cost, ease of operation, minimum sludge generation and reusability. Activated carbon exists in three different forms, namely granular, powdered, and pellet (Dawn, *et al.*, 2015: 238). Only the use of the granular form is explored in this study.

Ahsan and Ismail (2019: 2) write that granular activated carbon is recognized by the United States Environmental Protection Agency as one of the best methods of environmental control due to its large specific pore surface area; this makes it a powerful adsorbent with the ability to adsorb a wide range of contaminants. Mazille (2019) observes that activated carbon is a material prepared in such a way that it exhibits a high degree of porosity and an extended surface area to optimize the rate of adsorption. Yusuf (2018: 16) comments that adsorption offers a cost-effective solution with reusable options using different regeneration methods. Ansari and Mohammad-Khan (2009: 859), Donau Carbon (2011: 2) and Rashed (2013) concur that the following are advantages of using GAC for effluent COD removal:

- Removal of both organic and inorganic substances from the effluent,
- reduction of residual substances that contain chemicals,
- enormous removal of residual COD concentrations,
- reduced land area requirement for GAC implementation,
- reduced sensitivity to daily flow variations,
- simplicity of implementation and operational flexibility control at a plant level, and
- it is dust free and enables easy filtration of the treated effluent.

Evaluation of the above advantages assisted the researcher with the selection of the form of the adsorbate to be used to develop a sustainable method for the optimal reduction of COD concentrations. It is believed that this will enable the WCM plant to consistently manage COD concentrations in the effluent discharged to the municipality to required standard in future.

Among the different types of materials that can be used for adsorption, Ahsan and Ismail (2019: 3) report that coconut granular activated carbon (CGAC) is a very good adsorbent material because it contains cellulose, hemicellulose and lignin. Dawn, *et al*. (2015: 238) explain that GGAC material is derived from coconut shell. Karalei and Suryavansh (2014: 14) applied low cost CGAC for reducing the COD and biochemical oxygen demand concentrations in dairy effluent. According to Evuti, Jibril, Noraini and Poh (2013: 16), activated carbon is considered more economical since it is made from coconut shell agricultural waste. Taking previous research studies that have been reviewed into account, CGAC was adopted in this research as an appropriate method of exploring the most efficient procedure for optimal COD removal from the WCM effluent. The main benefits of this method include its effectiveness and environmental friendliness.

3.3.2 Optimisation of COD concentrations removal using the GAC adsorption

In the context of this study, optimisation refers to the monitoring of the selected varaibles in the adsorption process with the purpose of achieving the research objective. According to Moreno-Castilla (2004: 5), the composition of the functional groups of the GAC determine the adsorption strength of the GAC surface area to adsorb filtered contaminants. Bhise, Deshpande, Patil, Patil, and Raskar (2013: 67) found that the GAC functional groups enhance the rate of adsorption process, which involves attraction of the adsorbent by forces on the GAC surface area. Wu (2004: 5) explains that the availability of the GAC surface area also plays an important role when optimizing the adsorption process; as it increases the chances for more adsorbate to be directly in contact with the adsorbent. Yangui (2013) commented on another factor that has a direct influence on activating the rate of adsorption, the GAC pore size. Wu (2004:7) agrees with Yangui (2013) that the GAC pore sizes provides transportation pathways through which the adsorbate solution travels.

The reviewed literature indicated that the CGAC adsorption method has more advantages when applied. The influence of the variables is studied with the aim to determine the variable profiles that will result in the most reduction of the COD concentrations. Therefore, providing an ideal answer to the third research objective which states, "Identify variables that can be adjusted to yield the optimal removal of COD from WCM effluent".

3.3.3 Impact of the variables on adsorption activity

According to Galloway (2019) the activity of CGAC adsorption depends on many factors such as contact time, adsorbent mass and pH content of the adsorbate. Ushakumary (2013: 7-9), Nekoo and Shohreh (2013:87), and Wu (2004: 7) report that the capacity of adsorption of the contaminants depends on different variables of the adsorption process; which include:

- The contact time of the effluent with GAC,
- the flow rate of untreated effluent through GAC adsorbent,
- the potential of hydrogen (pH),
- temperature,
- effect of inlet untreated effluent concentrations, and
- availability of the GAC surface area.

Three variables, namely the contact time, pH content, and GAC surface area or GAC weight are explored in this study, when determining the most effective process for the optimal removal of COD concentrations in the WCM effluent. The literature reviewed in this Chapter will helped the researcher to theoretically guide the research to develop an experiment; which is described in Chapter 4, to answer to the third investigative question: “Can a process variable(s) be identified, that when adjusted, yields the optimal removal of COD from WCM effluent?”. The literature of the impact of the three variables to be explored in this study is discussed in the next three points.

3.3.4 Filtration contact time

El-Gawad and EL-Aziz (2018: 228) report that an increase in the contact time in an adsorption process results in more removal of COD concentrations. Ushakumary (2013: 7) notes that an increase in contact time results in more adhesion of solute molecules on the GAC surface; which enhances the removal of the COD concentrations. Liang, Liu, Lu, Pan, Xu, Zhang and Zhu (2011: 2-3) commented that an increase in contact time results in increased adsorption result in more reduction o-f COD concentrations in the solution. A study conducted by Goswami and Kulkarni (2013: 181), also concluded that increase in the contact time improves the removal of COD concentrations from the filtered solution. The influence of contact time on reducing WCM COD`s in the effluent is discussed in Chapter 5.

3.3.5 pH content of the sample or solute

According to Ushakumary (2013: 7), the pH content of the adsorbate or solute also plays a vital role in accelerating the adsorption process. In a study conducted by Goswami and Kulkarni (2013: 181), they reported that as the pH decreases from 14 (alkaline) to 1 (acidic), the rate of the COD removal in the solution increases. El-Gawad and EL-Aziz (2018: 228) also achieved similar results when they applied the GAC adsorption method for COD removal in the effluent. This study sets out to determine if the pH range have a significant impact in the optimal removal of CODs in the WCM effluent.

3.3.6 GAC surface area

Nekoo and Shohreh (2013: 87) claim that there is a direct relationship between the optimal removal of the COD concentrations and an increase in GAC surface area. Liang, *et al*. (2011: 2) report that the increase in GAC surface area could result in optimal COD concentrations removal, if the ions in the solution are available to bond with electrons available on the surface of the adsorbent. Bonilla-Petriciolet, *et al.* (2017: 10) agrees that an increase in adsorbent surface area results in an increase in the rate of adsorption. Guided by the views of Nekoo and Shohreh (2013: 87), Liang, *et al*. (2011: 2) and Bonilla-Petriciolet, *et al.* (2017: 10) mentioned above, this study sets out to explore the effect of the CGAC surface area on the optimal removal of COD concentrations in the WCM effluent.

3.4 Summary of the GAC adsorption method

The discussion of the variables that influence adsorption activity presented above illustrates that the GAC adsorption method is capable of achieving optimal removal of the COD concentrations; provided that variables such as contact time, pH and GAC are controlled accordingly. The control of the selected variables involves adjusting them to a certain degree to achieve the optimal COD concentrations removal.

In the next section, the applicable methodology and tools that can be used together with the CGAC adsorption method in a process of reducing the COD in the WCM effluent.

3.5 Application of Six Sigma DMAIC

Murray (2016) reports that the Six Sigma DMAIC problem-solving method has been proved to result in significant improvements using the appropriate quality tools. In this study, Six Sigma DMAIC will serve as a road map for data collection and analysis with the purpose of determining a solution for the research problem of this study.

3.5.1 Introduction the application of Six Sigma DMAIC

Boruah and Nath (2015: 589) used the Six Sigma DMAIC to improve the sustainability of environmental management, to improve the quality in the discharged effluent to conforms with the legislated standard specification. Furthermore, it is noteworthy that Mihai, Pana, Presura, Robescu and Silivestru (2016: 30) also used the DMAIC framework to develop a continuous improvement strategy when they adopted a new approach to reduce non-compliance in effluent treatment plants. Belamkar and Singare (2016: 2042) report that the DMAIC is suitable for improving the process after identifying the root causes of poor performance. Graves (2012) notes that the DMAIC is very useful when developing and implementing new initiatives. According to Cahyadi, Hernadi, Kurniawan, Prasetyani, and Rimantho (2017: 849), Six Sigma DMAIC methodology can be successfully used to improve the quality of COD concentrations in effluent discharged by industrial activities. An explanation of the five systematic phases of the DMAIC methodology given by Graves (2012) is explained below:

- **D – Define:** Define where the process might fail to meet customer or statutory requirements,
- **M – Measure:** Measure and determine if the sub-processes in a process meet customer or statutory requirements,
- **A – Analyse:** Evaluate the root causes of not meeting customer requirements or statutory requirements,
- **I– Improve:** Introduce changes in a current process to meet customer requirements or statutory requirements, and
- **C – Control:** Confirm the new standard operating procedures are documented accordingly.

In Chapter 5 of this research study, the extent or severity of the research problem is outlined in the Define and Measure Phases. In the Analyse Phase, the root causes of the researched problem identified from the two Phase, called Define and Measure are established. The outcomes of the Analyse Phase are used as inputs in the Improve Phase when establishing the proper solutions. In the Control Phase, control measures of the solutions are established to sustain improvement.

Patel (2014: 275) acknowledges Six Sigma DMAIC as a well-structured continuous improvement methodology that seeks to identify and eliminate defects or failures. Cahyadi, *et al*. (2017: 849) comments that quality improvement in Six Sigma DMAIC methodology is driven by statistical analysis of the process data. In Chapter 5 of this research study, selected statistical tools and techniques are employed in different DMAIC phases to analyse and interpret the collected data (quantitative). The application of the selected quality tools and techniques used in Chapter 5 are described in the next Chapter, Chapter 4.

3.5.2 Data analysis using quality tools

The Management Associate Information (2013: 1025) adopted the Six Sigma DMAIC method to improve a process performance in their study. Khandula and Singh (2015: 71) argue that each phase in the DMAIC process consists of a set of tools or techniques used for analysing data. Antony, Banuelas and Kumar (2006: 13) comment that Six Sigma DMAIC employs both statistical and non-statistical data to improve non-conformances in processes. Christmann (2012: 143) adds that the statistical tests provide a mechanism for making quantitative conclusions about the data that is being studied. The quality tools and techniques that are adopted in the context of Six Sigma DMAIC applicable in this study are presented in **Figure 3.1** below.

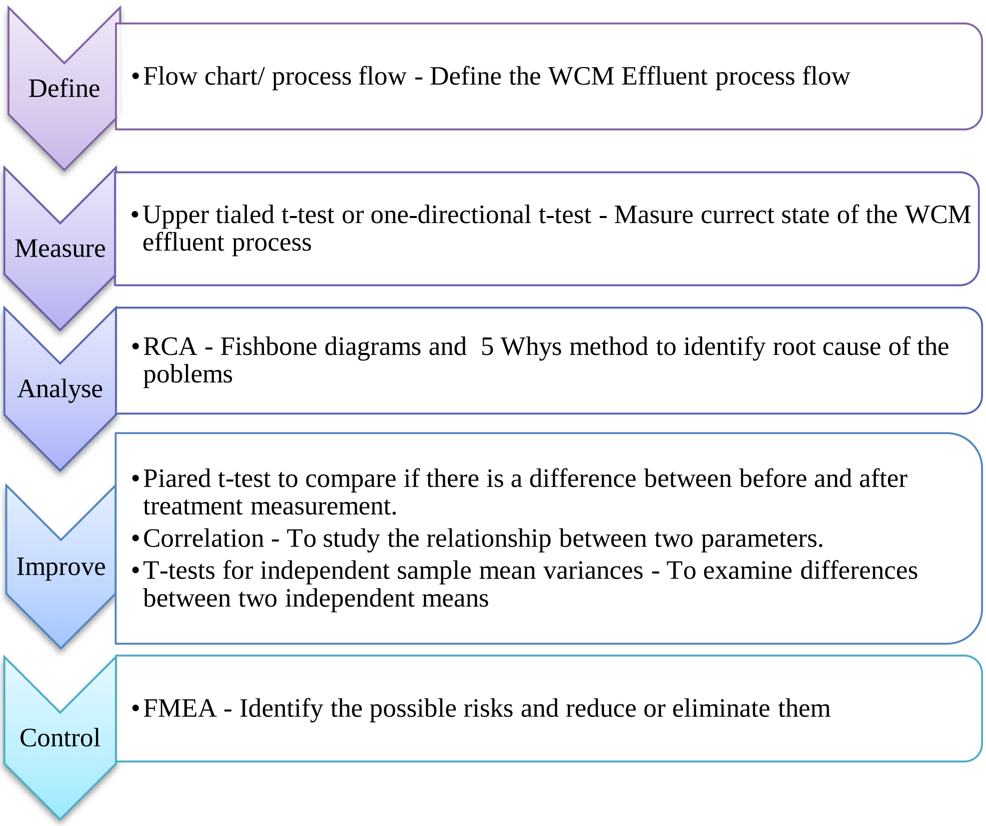

Figure 3.1: Application of quality tools and techniques in DMAIC methodology

The discussions of each tool or technique used in each Phase of the Six Sigma DMAIC are provided on the next points.

3.5.3 Process flow within the Define Phase

Antony, *et al*. (2006: 58) reports that a process flow or process map can also be used in the Define Phase to understand the key inputs and outputs of a process being studied. Davis and Yen (2000: 27) note that a process flow diagrams can be employed to identify main primary data collection areas. Vanzant-Stern (2012: 117) expounded that the process flow diagrams can be used in Define Phase as an information tool to help to identify the measurements required in the Measure Phase. Management Associate Information (2013: 1025) add that a process flow helps to measure the current performance against the target performance. From the abovementioned, it is deduced that a process flow may be employed to define all the WCM effluent process steps; as a primary step in answering the first investigative question of this research study in section 1.6.1, in Chapter 1.

3.5.4 T-test within the Measure Phase

Larson (2014: 132) comments that the Measure Phase focuses on measuring the baseline of a process to describe its current performance and to determine the key area(s) of improvement. According to Cramer and Howitt (2004: 166-167), a one-tailed t-test for one sample is considered to be a tool which may be used to determine whether the mean of a sample differs significantly from the true value or target value. Nestor and Schutt (2015: 276) explain that the one-tailed t-test is employed when a researcher wants to specify the exact direction of the difference.

According to De Muth (2014: 180), in one-tailed t-test, a rejection region is located at one end of the sample distribution. Christmann (2012: 143) notes that the decision to reject or accept the null hypothesis is made on the basis of the outcomes after comparing the critical value with the calculated statistical value. Hahs-Vaughn and Lomax (2012: 143) comment that the degrees of freedom and confidence limits (probability) must be clearly defined in order to find a critical value; which determines the decision to reject or accept the null or alternate hypothesis. Soderstrom (2008: 65), acknowledges that a one-sided or lower tailed t-test implies that the region of rejection lies only below the null hypothesized value. The author further comments that claims for the one-tailed t-test include the following hypotheses:

- Null hypothesis - H_0: Population parameter ≤ hypothesis value
- Alternate hypothesis - H_1: Population parameter > hypothesis value

Singh (2007:159) adds that the critical limits are the factors that define the acceptance or rejection of the stated null hypothesis. The critical values ($t_{critical}$) are found from the t-table, at the certain probability of the confidence of limits at a given degrees of freedom (Sirkin, 2006: 250). The degrees of freedom are determined using the number of participants minus the number of groups (Nestor & Schutt, 2015: 276). Guided by the preceding discussion, in the Measure Phase presented in Chapter 5 of this study, the t-test is used to examine the effectiveness of the COD concentrations in the pre-identified effluent steps or channels. The t-test result analysis will help this research to fully answer the first research question of this study, by demonstrating the WCM effluent macro channels that are not performing in accordance with the legislated standard requirement.

The results of the data analysis in this Phase will provide the research with direction pertaining to the areas that need to be investigated further. This process is supported by Chien, Dou and Huang (2019: 581), as they define the purpose of the Measure Phase as to gather the data and analyse it, to describe the nature of the process and to define the extent of the research problem. Therefore, in this research study, the identified non-conforming WCM effluent macro channels in the Measure Phase will serve as the inputs in the Analyse Phase.

3.5.5 Analyse Phase quality tools

Management Associate Information (2013: 1025) reports that in the Analyse Phase, the root causes are investigated from the key process inputs that have an impact on the process outputs. Maass and McNair (2010: 8) assert that the Analyse Phase validates the sources of variation or the potential failures that have resulted in ineffective performance of the process. Akpolat (2004: 44) notes that the Analyse Phase helps to identify the critical factors for improvement. In this Phase, the root causes of nonconforming COD concentrations were identified to establish alternate solutions.

3.5.5.1 Fishbone diagrams within the Analyse Phase

Gupta (2005: 227) acknowledged that within the Analyse Phase, a Fishbone or Ishikawa diagrams can be used as quality tool for exploring the potential causes of the problem. Vanzant-Stern, (2012: 117) commented that in the Analyse Phase, the Fishbone diagrams may be employed to investigate the root causes of the bottlenecks identified in the Measure Phase. Marcel (2011: 169) agreed that the Fishbone provides the researcher with the identification of areas that could harm the process. A Fishbone or Ishikawa diagrams as seen in **Figure 3.2** below, is an excellent tool for facilitating brainstorming when the potential root causes of a failure are unknown, using six Ms, namely Man, Measurements, Material, Milieu, Methods, and Machine (Barsalou, 2015: 70).

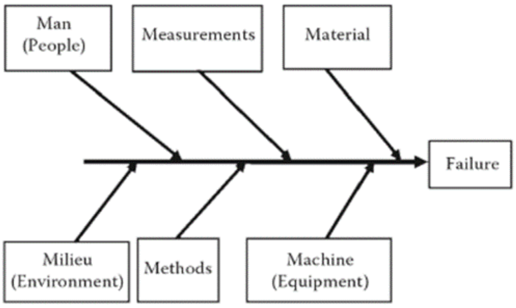

Figure 3.2: Fishbone and Ishikawa diagrams and six Ms (Barsalou, 2015: 70)

Rosenblatt and Shelly (2010: 70) state that a Fishbone diagrams is used to investigate the root causes of a failure by drawing the bone main bone linked to the problem, and the sub-bones that represent the possible causes. Carrell and Peterson (2010: 22-23) note that a Fishbone diagrams helps to visually explore all the potential causes that could result in a researched problem. Barsalou (2015:69) expounded that the 6Ms are used when creating a Fishbone diagrams to establish the root causes. Gupta (2005: 228-229) mentions the two benefits of using a Fishbone diagrams, namely gaining an understanding of the process issues and their relationship to the causes of the problems. Carrell and Peterson (2010: 22) add that in the Analyse Phase, the 5 Whys method is used in the Fishbone to further investigate the potential causes to identify the root causes. The 5 Whys method, discussed in the next section, is used to unpack the potential root causes of non-conforming COD concentrations in the WCM effluent identified using a Fishbone diagrams.

3.5.5.2 5 Whys method within the Analyse Phase

Rafinejad (2007: 338) states that the 5 Whys method helps to establish the factual root causes of problems. Laplante (2015: 978) explains that the root cause analysis (RCA) is a technique in which the 5 Whys is used for narrowing down every single effect identified in the Fishbone diagrams. Carkenord (2009: 310) elucidates that the 5 Whys method helps to establish the root causes of a problem by repeatedly asking the question “Why?” at least five times. Carrell and Peterson (2010: 22) emphasize that the 5 Whys is a not an absolute rule, but it helps to peel off layers of symptoms to get to the root cause of the problem. They add that the extent of the 5 Whys depends on the depths of the root causes of the researched problem. The 5 Whys method is used with the Fishbone diagrams in Chapter 5 to identify the root causes of high COD concentrations in the WCM effluent. The RCA will help to provide answers to the second investigative question of this study: “What are the causes of the high COD contents present in the discharged effluent of the various process stages?”

The outcomes in this Phase were used to identify the most suitable and sustainable solution that were explored in the Improve Phase.

3.5.6 Improve Phase tools

Cahyadi, *et al*. (2017: 850) state that the Improve Phase sets out to eliminate the root cause(s) of non-compliance identified during the Analyse Phase. According to Boby, Kabir and Lutfi, (2013: 1057), the problem can be resolved in the Improve Phase by implementing the solutions utilizing some of the following approaches to improve the process acknowledged by Juneja, *et al*. (2014: 1067) as follows:

- Brainstorming and action tests,
- benchmarking,
- extracting the vital few contributing factors through screening,
- understanding the correlation of the vital few contributing factors,
- process optimisation and validation experiment,
- hypothesis testing, and
- new process flow.

Cahyadi, *et al*. (2017: 850) observe that laboratory experiments can be used in the Improve Phase to explore ways to find solution(s) to causes of undesirable variation. Garza-Reyes, Jirasukprasert, Rocha-Lona, and Soriano-Meier (2012: 473) add that the Improve Phase involves the execution of experiments, accompanied by statistical data interpretation and analysis to validate the reduction of problems. This suggests that it is appropriate to conduct experiments and simultaneously perform applicable statistical techniques to explore the results of the experiments to establish improvement, as presented in Chapter 5 of this study.

The purpose of the Improve Phase is to design and test the experiments, and analyse the results obtained to interpret the signs of the improvement solutions (Chien, *et al*., 2019: 581). Hypothesis testing is defined as a test that is used to evaluate the validity of the claims of the available data (Murphy, 2011: 20). According to De Muth (2014: 205), ANOVA hypothesis testing involves creating and validating of two separate hypotheses, namely the null hypothesis (H_0) and alternate hypothesis (H_1) using the data. Austin and Leong (2006: 99) mention that the most significant objective of hypothesis testing is to verify the correct hypothesis about the population data within a specified degree of freedom and certainty such as 95%.

The hypotheses tests were used in Improve Phase in Chapter 5 this study to examine the significance of the experimental adsorption procedures, designed from revised literatures of adsorption method; with the aim to answering this investigative question in section 1.7.3 in Chapter 1.

3.5.6.1 Paired two-tailed t-test within the Improve Phase

A two-sample paired t-test is employed to compare two levels of a discrete independent variable to statistically determine if the sample means are the same or different (De Muth, 2014: 183). A two-tailed two-sample paired t-test is a non-directional test conducted for examining whether there is a significant difference between the sample mean and the true value (Osborn, 2006: 168). Butler, Edwards, Jackson and Letswaart (2016: 163) used the paired t-test to determine significant changes after the improvement initiatives in a DMAIC project. Javier (2011: 144) also employed a paired t-test to explore the difference in electromyography to compare two conditions. Clarke and Woolson (2002: 154) reasoned that the paired t-test evaluates paired data with an assumption that the data of the pairs differences are normally distributed. Elliot and Woodward (2007: 71) write the null hypothesis testing of a paired t-test state that the difference between the means of two populations is zero (H_0: $\mu_d = 0$), and the opposite, called alternate hypothesis states that the difference is not zero (H_1: $\mu_d \neq 0$). The decision is made to reject the null hypothesis if the statistical value falls outside of the non-rejection region or beyond one or both tails (critical values), and then the opposite is accepted (Heckard & Utts, 2006: 448). The critical values are obtained from the t-table, using the degrees of freedom and confidence of limits or alpha divide by two (Heckard & Utts, 2006: 448). In Consumer Dummies (2014: 406) it is acknowledged that the degrees of freedom one minus the number of pairs are employed to obtain a critical value at an applicable confidence limits should be used. Dytham (2011: 95) used the paired t-test to compare the amount of chlorine that was present before and after treatment. Thus, in this study, it is assumed that the use of the two-sample paired t-test would be an appropriate tool to compare the COD concentrations means before and after the applications of the adsorption procedures.

3.5.6.2 Correlation studies within the Improve Phase

According to Ramu (2017: 51), correlation studies may be employed in the Improve Phase to estimate the significance of the improvement. Haber, LoBiondo-Wood, Scheider and Whitehead (2013: 168) comment that correlation studies enable an analysis of the relationship between pairs or groups of variables. Cunningham, Pittenger and Weathington (2012: 246) add that the correlation studies allow to make predictions about the dependent variable using an independent variable. Mertens (2005: 158) note that the correlation coefficient predicts if a relationship exists between an independent variable and a dependent variable. The coefficient of determination "r" is used to statistically quantify the estimate degree or strength (Norcross, 2011: 77). A correlation analysis enables a researcher to determine the degree or strength of a relationship and a type of relationship (positive or negative) between two variables (Burns and Grove, 2011: 35). After determining a relationship, the researcher must indicate the direction of the relationship, whether it is positive or negative and the degree strength of the relationship (Ariola, 2007: 48). Wilson (2019) comments that the closer the coefficients are to +1.0 and

-1.0, the greater the strength of the relationship between the variables, considering the direction indicated by the sign. Abraham, Franke and Koppen (2003: 55) add that when the prediction is perfect, a coefficient of determination of 1.0 is achieved, and when the prediction is opposite to the actual data, a coefficient of determination of -1 is achieved. Brewer and Picus (2014: 826) conclude that a correlation coefficient of 0.7 to 1 is considered a good correlation. The coefficient of determination is used in Chapter 5 to determine the strength and direction of a relationship between two or more variables tested in this study.

3.5.6.3 Two-tailed t-test for two independent samples within the Improve Phase

Martinez, Oppenlander, Shifflet and Shmerling (2020: 181) propose that a two-tailed independent t-test may be used to compare the means of two independent groups in the Improve Phase to determine if there is a statistically significant difference. The independent t-test is also referred to as a two-sample independent t-test and is an inferential statistical test used for determining a statistically significant difference between two means of two unrelated groups (Laerd Statistics, 2018). Damarla, Kundu and Kundu (2018: 22-23) comments that the test claim can be expressed as follow, null hypothesis is H_0: $\mu_1 = \mu_2$, while the alternate hypothesis is H_1: $\mu_1 \neq \mu_2$. The null hypothesis is only accepted if the calculated t-value (t-statistics) falls within the accepted region or range (Six Sigma Material, 2020). The critical value is obtained from the t-table, using the point of intersection of the degrees of freedom and the applicable confidence of limits (Clark, 2020). T-testing of two independent samples helped in this study to explore if there is a significant difference between the independent means of data obtained from the two experimental trials in Chapter 5. The details of the use of the test are described in Chapter 4.

Sobh (2008: 169) comments that the Improve Phase involves testing the possible solutions which are later reviewed and validated for their effectiveness and sustainability in the Control Phase. Literature pertaining to the application of the Control Phase is presented in the next section.

3.5.7 Failure modes effects application within the Control Phase

Khatri (2019) advocates that the FMEA tool be used in the Control Phase to assess and control the risks after improvement of a process or system. Beauregard, McDermott and Mikulak (2008: 1) write that FMEA is a systematic method used to identify and prevent incidents in a process before they occur. De Carlo, Gygi and Williams (2012: 288) opine that FMEA is used in the Control Phase to eliminate risks by evaluating the effectiveness of each control measure. Dhillon (2007: 60) comments that FMEA is commonly used in the industrial sector to analyse engineering systems to improve their reliability. Gupta (2005: 227) agrees that FMEA tool is used to anticipate potential problems and to prevent or reduce them from happening. Belokar and Rana (2017: 263) describe FMEA as a systematic and proactive method for evaluating a process to identify where it might fail and implement failure prevention to reduce or eliminate potential failures. Myers (2012: 306) writes that FMEA tool is employed to proactively identify potential failure risks by assigning a priority number or risk score to each detected risk. It employs three components to determine the priority of failures, namely severity, occurrence and detection (Jodejko-Pietruczuk, Mtynczak, Nowakowski & Werbinska-Wojciechowska, 2015: 153). Wasson)2016: 765) add that the FMEA use the Risk Priority Number (RPN) to predict the significance of the identified risks. Unnasch, Venkatesh and Waterland (2003: 3) reason that the RPN rating helps to quantify the significance of the failure risks, and also to prioritize them. Parsana and Thakore (2015: 413) write that the FMEA is employed to:

- Identify the impact (severity),
- how often the failure is likely to occur (occurrence),
- assess the likelihood of detecting the failure (detection), and
- establish the areas of focus improvement using RPN.

According to Dunscombe, Mundt, Pawlicki, and Scalliet (2011: 111), FMEA is used to identify the potential failure modes and their causes by listing all the process controls, which include quality inspections, training, work instructions, standard procedures, and checklists. The use of the FMEA tool will in this study will help the researcher when identifying potential failure risks associated with the process establish to improve the conformance of the COD with a legislated standard specification.

3.6 Chapter 3 summary

Relevant literature was presented to the reader, guided by the extent and complexity of this research study. Firstly, the literature related to the background and motivation of this study was presented, followed by literature pertaining to existing possible solutions to this research problem. Following this, literature on applicable methodology to be used in a study such as this when planning, implementing and optimizing the possible solutions was discussed.

Literature presented in this chapter included the importance of and motivation for this study, a review of the existing solutions of the researched problems, and evaluation of their effectiveness and sustainability. The next chapter will provide insight into the research methodology, research design and methods adapted this study.

CHAPTER 4: RESEARCH METHODOLOGY

4.1 Introduction to Chapter 4

Cash, Stankovic and Storga (2016: 220) state that proper research design should give a clear explanation of the plan to study a phenomenon and controls for all the possible biases that could distort the research findings. Maxwell (2005: 107) confirms that in a study, proper planning of the research design is very important; this entails the data collection methods, data analysis tools, and controls to eliminate both anticipated and unanticipated validity threats. This chapter offers the reader an outline of the methodological aspects relevant to this research. It also present detailed explanations of the data collection procedures and methods of data analysis to be employed in Chapter 5 of this research study. This will help the researcher to explore the effectiveness of CGAC application to the WCM effluent to achieve optimal COD removal.

Guided by the view of Kumar (2011: 10) who argues that the research methodology describes research methods, approaches and designs in detail and highlights those used throughout the study. This chapter commences with a review of research designs and methodologies; thereafter an appropriate method relevant to this study is selected. Details of the research design are discussed within the context of this study. The population of the study, sampling frame, sampling units, sampling methods, sample preparation, and sample treatments are also discussed. Statistical methods employed for interpreting the quantitative data are then presented in detail, with a brief review of their application and significance in the research.

This chapter of the thesis justifies the choices made by the researcher by describing the advantages and disadvantages of the research approach and design reviewed. Before details of the research design are presented, various research worldviews are briefly explained below to help to the reader navigate the selection criteria of the research paradigm of the study.

4.2 Research worldviews

Ling and Ling (2017: 2) declare that a view paradigm in research is a term that is used to refer to worldviews that underpin all aspects of a research undertaking from the intent or motivation for the research to the final design and outcome. Collis and Hussey (2014: 11) write that a paradigm is a theory or a group of ideas that describes or provides a framework of how something can be done. Creswell (2014: 6) explains four worldviews that are widely discussed in literature namely, positivism, constructivism, transformative and pragmatism. The major elements of each position of the worldviews are presented in **Figure 4.1** below.

Positivism	Constructivism	Transformation	Pragmatism
• Determination • Reductionism • Empirical observations and measurements • Theory verification	• Understanding • Multiple participant meanings. • Social and historical construction	• Political • Power and justice-oriented • Collaborative • Change-oriented	• Consequences of actions • Problem-centered • Pluralistic • Real-world practice oriented

Figure 4.1: Four worldviews (Creswell, 2014: 6)

Based on the descriptions of each research worldview presented in **Figure 4.1**, this thesis adopts a positivist paradigm. Davies, Howells and Sheldon (2011: 5) describe a positivist approach as one that involves the pursuit of models or laws that can be derived by conducting observations or measurements of the social world. Guided by this description, this study may be characterised as the use of positivistic empirical measurement to obtain quantitative data, as quantitative laboratory experiments are a central component of the study.

Kothari (2004: 4) elucidates on quantitative and qualitative approaches, stating that quantitative research is based on the measurements of quantity or amount of an item of interest, whereas qualitative research is concerned with phenomena involving types or qualities. Kostelis and Matthews (2011: 3) hold that a quantitative research design explores solutions to research questions by using quantifiable research variables, also known as 'parameters', that can be measured and can be assigned a numerical value. Thompson (2017) is of the opinion that quantitative research comprises certain strategic process elements which involve the collection of numerical data, a deductive views of the relationship between theory and research, a preference for a natural approach and an objectivist conception of social reality.

Furthermore, Grinnell and Unrau (2011: 396) propound that the inductive research process hypothesis is derived from existing theories. Depoy and Gitlin, (2011: 9) comment that a deductive research process follows when the empirical world is explored by collecting the data to test a hypothesis. It is worth noting that at the commencement of an inductive process approach, a researcher begins with few preconceptions as possible, allowing theory to emerge from the data. The nature of this research study lends itself to both inductive and deductive research processes respectively being followed to meet the objectives of this study. Chapter 3 of this study is inductive, since it reviews information on existing interventions concerning COD removal in effluent. Chapter 5 presents the findings of appropriate existing methods that have been tested to deductively develop a new theory for the optimisation removal of the COD in the WCM plant effluent. The research process flow developed by the research is illustrated in **Figure 4.2** below.

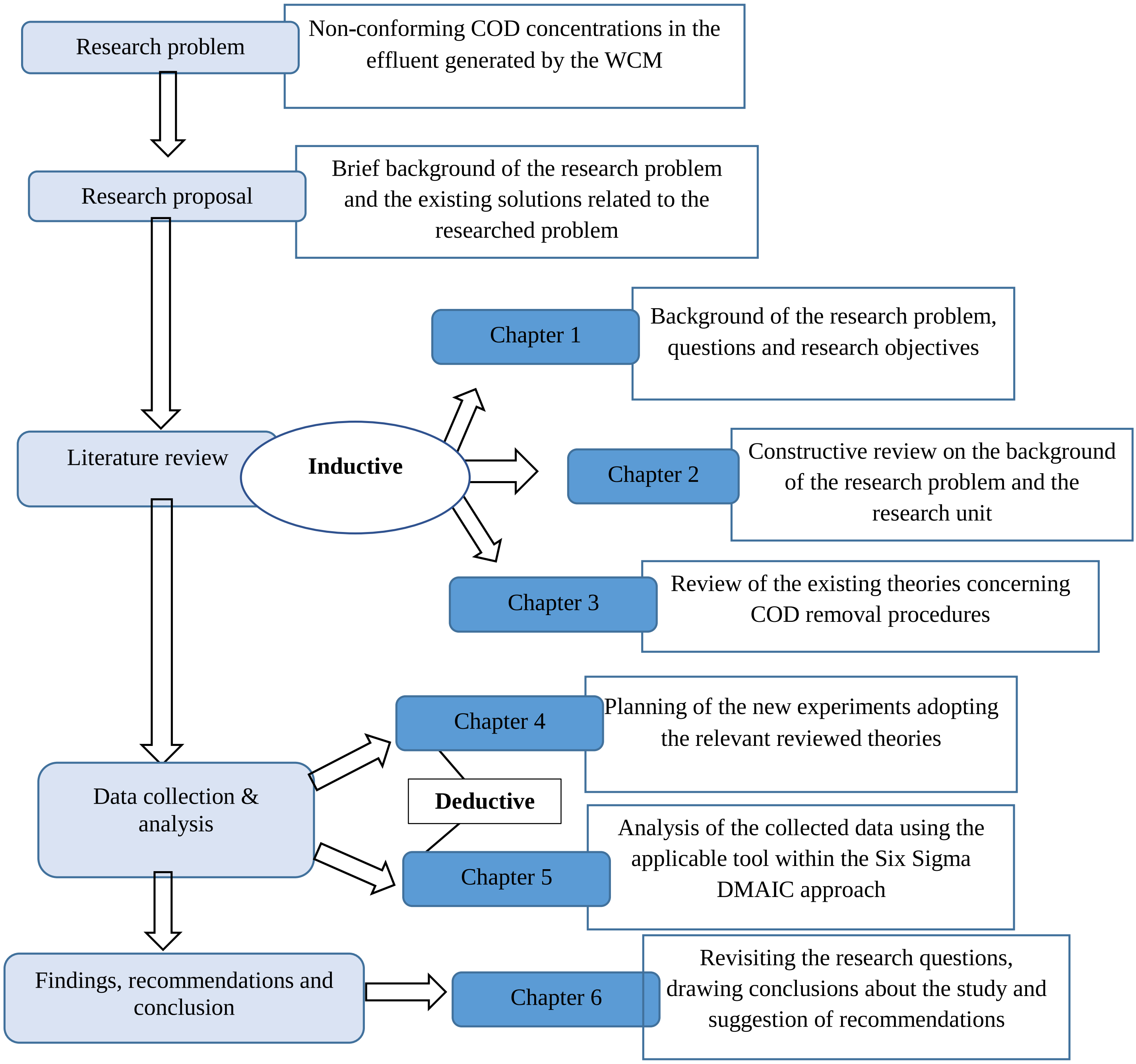

Figure 4.2: A process followed in this research

4.3 Six Sigma DMAIC research methodology

Bubevski (2018: 89) reports that the methodology of a research study are the specific procedures used to systematically solve research problems. Burton (2012: 35) agrees with Bubevski (2018: 89), that the research methodology of a study is a systematic method adopted by a researcher to perform a research study.

Chapter 3 discusses the uses of the relevant tools and techniques in Six Sigma DMAIC methodology to address problems and implement improvements. Khandula and Singh (2015: 71) acknowledge that each phase of Six Sigma DMAIC methodology entails the use of a set of quality tools and techniques for process improvement. In this chapter, a discussion of the application of the DMAIC to analyse collected data with the goal of providing answers to the four research objectives of this study is presented. The reader is reminded that the objectives are to:

- Investigate which stages in wet corn processing have a significant influence on the subsequent high COD of the discharged effluent,
- establish the causes of the increasing levels of COD in effluent generated by a WCM,
- identify the variable(s) that can be adjusted to yield the optimal removal of COD from WCM effluent, and
- establish an effective procedure for removal of the COD in the effluent generated by a wet corn milling process.

Significantly Duckworth and Hoffmeier (2016: 38) assert that DMAIC methodology is a body of systematic techniques used mostly in a scientific discipline, and is characterised by a series of steps for solving problems. Furthermore, Bless, Higson-Smith and Kagee (2007: 7-8) hold that properties of a scientific research method include being systematic and logical, replicable, transmittable and reductive. Thus based on this description, it may be deduced that DMAIC is a scientific research approach. Moreover, Duckworth and Hoffmeier's (2016: 38) assertion is consistent with Williams's (2007: 66) definitions of research methodology, which implies that DMAIC may be used as a method to conduct research.

Khandula and Singh (2015: 16) argue that Six Sigma DMAIC makes use of empirical data gathering, which implies the use of real world data gathering within the context of specific theories. Therefore, for the purpose of this research, the Six Sigma DMAIC approach was used to empirically collect data, and then analyse and improve a process using the data. Saleh (2014) offers the following graphic representation of the five Phases of Six Sigma DMAIC methodology in **Figure 4.3** which follows.

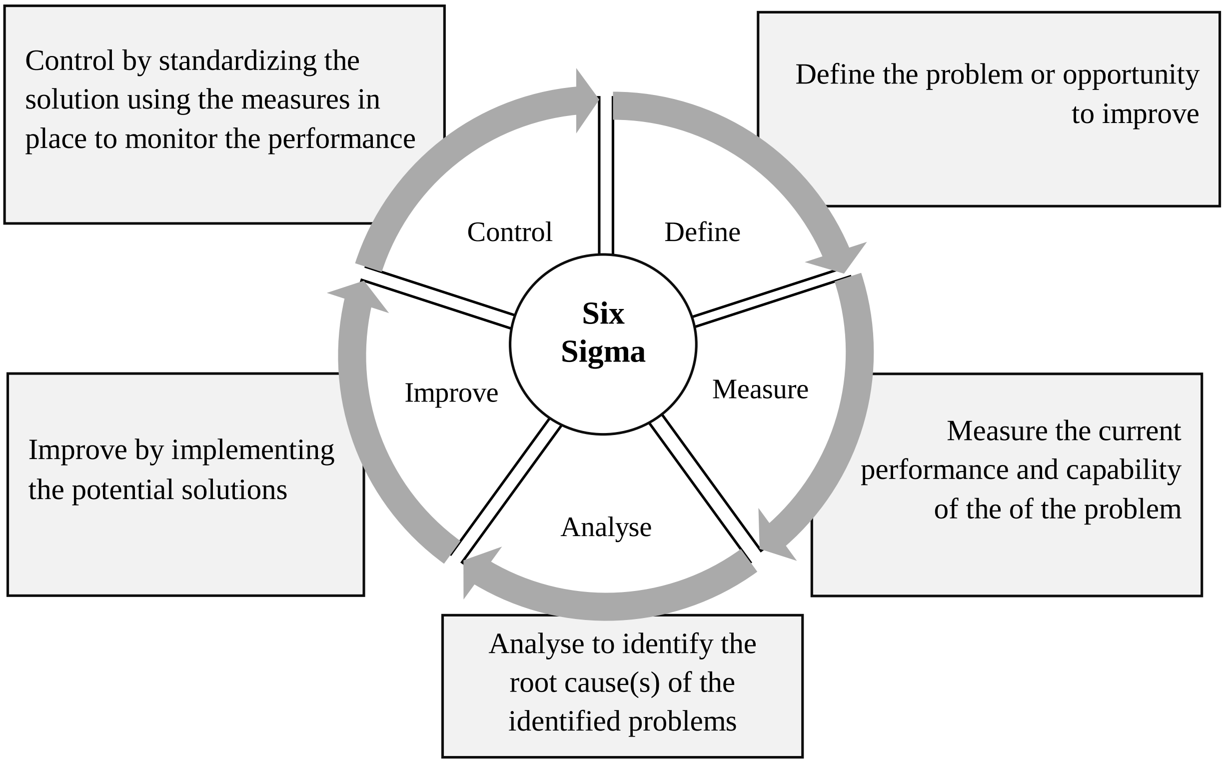

Figure 4.3: Six Sigma DMAIC methodology (Saleh, 2014)

Notably, the Six Sigma DMAIC methodology makes use of both scientific methods and quality tools to find solutions to research problem (Elshennawy, *et al.*, 2009: 319). The Six Sigma DMAIC methodology is therefore regarded as a suitable methodology for this thesis. The methodology is used in this thesis to investigate the primary research problem.

4.4 Data collection methods

Couper, Fowler, Groves, Lepkowski, Singer and Tourangeau (2009: 7) hold that sampling frames are the list of procedures employed to identify all the elements of a targeted population. Defining a population, Surbhi (2017), explains that a population is the total large group that consists of elements having at least one common feature, and a sample is a subgroup of the population that represents the entire group. The targeted population of this research study is the COD concentration in the total effluent generated by a WCM plant located in the WC.

Dudovskiy (2018) affirms that the careful selection of the data collection method is essential for good research and data collection involves primary sampling. Carter, Domholdt, and Lubinsky (2011: 92) declare that sampling is the process of selecting sub-group participants from a larger group of potential participants. Lohr (2009: 2) states that a good sampling method is therefore critical to provide research with conclusions that are valid in that accurate generalizations have been made from reliable data.

According to Lohr (2009: 2), the aforementioned underscores the importance of the proper selection of appropriate sampling methods to ensure study validity. Fleetwood (2019) mentions that sampling methods are divided into two categories, namely probability sampling and non-probability sampling. Daniel (2012: 4) expounds that probability sampling procedure uses the principle of randomization or chance. Tashakkori and Teddlie (2009: 171) state that sampling procedure involves the random selection of specific units or cases to ensure that the probability of inclusion for all the elements of the population is presented. Carter, *et al*. (2011: 97) comment that non-probability sampling is widely used in sampling that is non-randomized. Bradley (2013: 162) notes that there are different types of probability sampling, namely simple random, interval or systematic, stratified, and clustered or multi-stage; while non-probability sampling includes convenience, snowballing positive or judgemental, and quota sampling (Babbie, 2008: 203).

Probability sampling is adopted in this study to assure results that are representative of the entire population being studied. Daniel (2012: 75) reasons that probability sampling is recommended when dealing with the measurement of a heterogeneous population for the variable(s) of interest. There are two types of probability sampling that are used in this research study, namely systematic sampling and stratified sampling.

Guided by Fleetwood's (2019) definition that systematic sampling is a type of probability sampling method where the elements are chosen from a target population after a fixed sampling interval. A systematic approach was used the Measure Phase, to collect COD concentrations data from the WCM for a period of sixty days, in which twelve effluent samples were collected each day at even hours. These samples were collected from the eleven WCM effluent macro channels, to be analysed for COD content individually. The total of 660 COD concentrations means and a total of 7920 COD concentrations subgroups collected at even hours for sixty days are outlined in **Table 4.1** below. The primary COD data collection was done by a qualified WCM laboratory analyst; the data interpretation and analysis is carried out in the Measure Phase, in Chapter 5. The analysis of the data is required to provide answers to the first research objective of this thesis: "Investigate which stages in wet corn processing have a significant influence on the subsequent high COD in the discharged effluent".

Table 4.1: COD data collection plan from the eleven WCM effluent macro channels

Sampling Time	Number of samples per macro effluent channels
06:00	1
08:00	1
10:00	1
12:00	1
14:00	1
16:00	1
18:00	1
20:00	1
22:00	1
00:00	1
02:00	1
04:00	1

Part two of the sampling method in Improve Phase in Chapter 5 involved stratified probability sampling. Kohl, Magnussen and Marchetti (2006: 105) explain stratified sampling as a type of sampling method in which the total population is divided into smaller subgroups or elements. Brown, Suter and Churchill (2018: 211) agree that stratified sampling is a form of probability sampling in which the population is divided into mutually exclusive and exhaustive subgroups, and secondly, samples are chosen from each of the subgroups. Guided by the views of the authors mentioned above, the stratified sampling in this study involved two groups of samples whereby group one is represented by 36 acidic samples in experimental trial one and group two by 36 alkaline samples in experimental trial two. See **Figure 4.4** below for the two groups of stratified sampling and **Appendix A** for detailed CGAC filtration procedures.

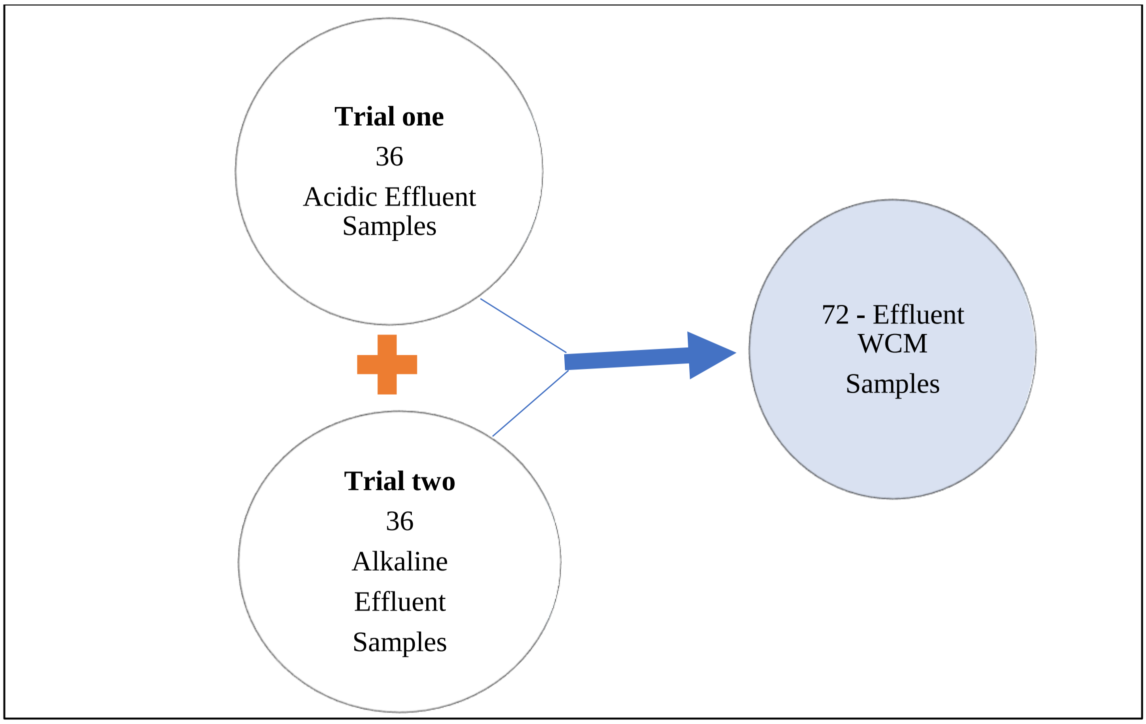

Figure 4.4: Stratified sampling plan for CGAC trials

When collecting the primary data, the pH, conductivity and COD measurements are conducted on each effluent sample, before and after the CGAC filtration procedure. The CGAC filtration procedures for each trial batch involved adjustments of the filtration weight (20 gram (g), 30 g and 40 g), and adjustment of the contact time (20 min (minutes), 30 min and 60 min). The effect of the adjustments of the independent variables mentioned in **Appendix 1** are studied in a process of investigating most optimal procedure for COD removal. The two trials are employed to study the effects of the pH using the WCM effluent, guided by the literature reviewed in section 3.3.5, the COD data collected from the eleven effluent macro channels is analysed using statistical tests, namely paired t-test, correlation studies and independent sample t-test to evaluate the effect in meeting the objectives of this study.

4.5 Data analysis methods following the DMAIC approach

The University of Pretoria Department of Library Sciences (2018) states that data analysis involves the interpretation of data to identify the trends, patterns, and relationships in the data being studied. Six Sigma DMAIC methodology was used as a framework in this study for data collection, interpretation and analysis to investigate the research problem and to find an answer to the investigative questions of this study, as outlined in section 1.6 in Chapter 1. The description of the type of data used in each DMAIC Phase and the tools or statistical tests used for data interpretation and analysis are depicted in **Table 4.2** below.

Table 4.2: Plan followed the application of DMAIC for COD concentrations removal from the WCM effluent

Six Sigma Phase	Type of data source	Tools or techniques
1. Define	WCM process map	**Flow chart** is used to describe the process flow of the WCM effluent to identify all the macro channels that make up the overall WCM effluent. The identified WCM effluent macro channels COD concentrations were measured in the Define Phase.
2. Measure	Raw or untreated effluent COD concentrations data collected from the WCM effluent macro channels.	**One tail t-test**s are employed to test which macro channels had COD concentrations means that showed a significant difference (higher) compared to the standard requirement of 5000 ppm.
3. Analyse	Incident records for the selected WCM effluent channels.	**Fishbone diagrams and 5 Whys** are employed to identify the all the possible causes of the incidents that resulted in high COD levels; with the ultimate aim of establishing their root causes.
4. Improve	The data of the trials were collected using CGAC filtration procedures.	**Paired two tailed tests** are used to examine if there is a significance different between the raw COD concentrations means versus the filtered COD concentrations. **Correlation studies** are used to identify which procedures resulted in a good positive linearity for the COD concentrations removal in the WCM effluent. **Independent two-sample t-tests** were conducted to compare if there is a significant difference between the COD concentrations means obtained from the two trials (trial one and trial two) that used similar procedures but different effluent pH contents.
5. Control	Data collected from the risks assessment of the WCM effluent process before and after the improvement.	**FMEA** tool is used to address the identified the failure risks that will hinder the effectiveness of the improved WCM process to achieve desired outcomes.

The application of these quality tools and statistical techniques mentioned in **Table 4.2** above is elaborated on in the discussion of the DMAIC phases below.

4.5.1 Application of the Six Sigma DMAIC Phases

In the Define Phase, the process flow is used to collect and map information to help to identify the areas or units of a problem being studied (Vanzant-Stern, 2012: 117). In the Define Phase, a process flow is used for mapping the WCM effluent process with the aim of identifying all the macro channels that contributed to the total COD concentrations in the WCM effluent. The information on the process flow is measured in the Measure Phase to identify the macro effluent channels that contributed to the nonconforming COD concentrations; thus, providing answers to the first research objective of this study: "Investigate which stages in wet corn processing have a significant influence on the subsequent high COD in the discharged effluent".

Bremer, Daniels, Gupta and McCarty (2005: 365) emphasize that the fundamental purpose of the Measure Phase is to gather information or data about the current status or performance of a process, and identify the areas of improvement. Antony (2014: 204) explains that the Measure Phase of DMAIC helps to study the current state "as it is", concerning to the performance of the problem being investigated. In this research study, the Measure Phase is employed to identify the macro channels with nonconforming COD concentrations. This is accomplished using the one-tailed t-test to test if the COD concentrations means obtained from each macro channel. Where the COD means were investigated if they are less or equal to the legislated standard specification of 5000 ppm. The null hypothesis of each t-test is that the COD concentrations of the effluent generated by each macro channel mean is less than or equal to the COD legislated standard specification. The alternate hypothesis is that the COD concentrations of the effluent generated by each macro channel is greater than the legislated standard specification. The critical values for the t-test analysis are found at the 95% confidence of limits (one-directional test). The results of the t-tests will determine the WCM effluent macro channels that contributed to nonconforming COD concentrations in the WCM effluent. See **Table 4.3** for the one direction t-test claims that are used in Chapter 5.

Table 4.3: T-test application plan followed for the analysis of COD data collected from the applicable WCM effluent macro channels

Effluent macro channel	**Test claim**
Name of the applicable WCM macro channel	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm

The outcomes of the t-tests results are used as inputs in the Analyse Phase when constructing the RCAs to establish the root causes of non-compliance in the selected macro channels. Gopalakrishnan (2012: 133) advocates the Analyse Phase to investigate potential causes that contribute to a research problem. Hodges (2017: 250) reports that the 5 Whys analysis is a follow-up technique used for determining the root causes of effects identified in the Fishbone diagrams. In the Analyse Phase, a Fishbone and 5 Whys analysis are performed on each macro channel that failed the t-test null hypothesis in the Measure Phase to identify the root causes of the failure. The use of the Fishbone and 5 Whys analysis helped the researcher to answer research objective number two of this study: "Establish the causes of the increasing levels of COD in the effluent generated by a WCM".

The CIToolkit (2018) states that the Fishbone diagrams is used to prevent the recurrence of problems by implementing preventative actions. After identification of the root causes of the nonconforming COD concentrations in the applicable WCM effluent macro channels, preventative actions are embarked in the Improve Phase.

Anirban (2015: 45) proposes that the target of the Improve Phase of DMAIC is to design creative solutions to the problem by using the outcome of the previous, the Analyse Phase. Rumane (2017: 120) declares that the Improve Phase involves planning and execution of an action plan that consists of innovative ideas to permanently remove the root causes of the problem being investigated. In the Improve Phase of this research study, different CGAC procedures are explored to find a method that would result in optimal removal of the COD concentrations in the WCM effluent. The experimental procedures were designed to provide an answer to the third research objective of this study: "Identify variable(s) that can be adjusted to yield the optimal removal of COD from WCM effluent".

The Improve Phase is divided into two trials, where trial one involve testing the CGAC procedures in removing the COD concentrations using acidic WCM effluent samples. Trial two involve testing the same CGAC procedures used in trial one but with alkaline WCM effluent samples. In each trial, three batches of experiments are conducted. Batch one will evaluate the effect of changing the CGAC filtration weight (20 g, 30 g and 40 g), at constant contact time of 20 min. In batch two and batch three the effect of increasing contact time to 30 min and 60 min respectively are explored. See **Appendix A** in the appendix for insight into the procedures to be used in each trial. After the collection of the data from the different experimental trials, the hypotheses tests are performed to interpret and analyse the data, as explained in point 3.4 of Chapter 3. These tests included paired t-test, correlation coefficient and independent two sample testing. An explanation of how each test is conducted is discussed below.

Paired t-test hypothesis testing is used to determine whether there is a significant difference between the COD concentrations means obtained before and after the CGAC application. The paired t-tests is conducted at 95% (α/2-0.025) confidence of limits and at n-1 degrees of freedom for each trial batch. The null hypothesis for each test is that there is no significant difference between the COD concentrations means before and after the treatment and an alternate hypothesis is that there is a difference between the COD means before and after filtration. If the null hypothesis is accepted, it implies that the tested procedure to be considered not effective enough to result in optimal removal at the 95% confidence limit. The null hypothesis and alternate hypothesis clams are illustrated in **Table 4.4** below.

Table 4.4: Application of the paired t-test to examine the mean difference between COD concentrations before and after treatment

Trial Name	Hypothesis claim	Trial Name	Hypothesis claim
Trial 1: Batch 1	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	Trial 2: Batch 1	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
Trial 1: Batch 2	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	Trial 2: Batch 2	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
Trial 1: Batch 3	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	Trial 2: Batch 3	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$

The correlation study is used to evaluate the effect of increase in contact time and CGAC filtration weight on the optimal removal of the COD concentrations. The correlation studies are performed to identify procedures that would result in a significant positive correlation for optimal COD concentrations removal from the two trials. See **Table 4.5** below for the test procedures.

Table 4.5: Procedures to be used for correlation analysis

Acidic WCM effluent samples	Alkaline WCM effluent samples
20 g: 20 min, 30 min and 60 min	20 g: 20 min, 30 min and 60 min
30 g: 20 min, 30 min and 60 min	30 g: 20 min, 30 min and 60 min
40 g: 20 min, 30 min and 60 min	40 g:2 0 min, 30 min and 60 min
20 min: 20 g, 30 g, and 40 g	20 min: 20 g, 30 g, and 40 g
30 min: 20 g, 30 g, and 40 g	30 min: 20 g, 30 g, and 40 g
60 min: 20 g, 30 g, and 40 g	60 min: 20 g, 30 g, and 40 g

The conclusion of the Improve Phase is drawn using the independent two-sample t-tests result. The t-tests results are used to determine if there was a difference in the reduced COD concentrations means obtained in trial one compared to trial two. The null hypothesis for each paired t-test is that there would be no significant difference between the reduced COD concentrations means obtained when using alkaline versus acidic samples. The alternate hypothesis will state that there would be a significant difference between the reduced COD concentrations means obtained when using alkaline samples versus acidic samples. If the null hypothesis is accepted, it will imply that the reduced COD concentrations means are the same at 95% confidence limits. If the alternate hypothesis is accepted, it will imply that there is a significant difference between the reduced COD concentrations means for the procedures. The results of the t-tests will help to answer the following research question of this study: "Identify variable(s) that can be adjusted to yield the optimal removal of COD from WCM effluent". See **Table 4.6** below for the procedure followed when conducting the paired t-tests.

Table 4.6: Independent two-sample t-test for testing mean difference in the reduced COD concentrations means obtained when using alkaline versus acidic effluent samples

Treatment	**Hypothesis claim**
20 g and 20 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
20 g and 30 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
20 g and 60 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
30 g and 20 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
30 g and 30 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
30 g and 60 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
40 g and 20 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$
40 g and 30 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 00$
40 g and 60 min	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$

The outcomes of the analysis of the t-test results are used as the inputs in the Control Phase. The Control Phase involved designing the sustainable control measures to eliminate the failure risks of the improvements or recommendations made by preceding Phases, using FMEA. In Carroll's (2013: 76) view, the FMEA tool can be used in the Control Phase to develop action plans to prevent failures that have been identified happening. The application of the FMEA in the Control Phase is aligned with the view of Anirban (2015: 45) who states that the Control Phase involves controlling the improvement interventions by documenting them for monitoring or sustainability purposes.

In this research study, the FMEA tool is used to assess the risks of having high COD concentrations in the WCM effluent after the implementation of the improvement actions. Ultimately, the Control Phase of this study is aimed at establishing the controls required for optimal removal of the COD concentrations in the WCM; refer to **Appendix O1 – OC** for the FMEA ratings. This provides answers to the last objective of this research study: "Establish an effective procedure for removal of the COD in the effluent generated by a corn wet milling process".

Berthouex and Brown (2002: 22) observe that all measurements are subject to error, and statistics play a vital role to quantify and characterize error, taking into account when data are used to make decisions. The errors considered in this study are discussed in the next point called validity.

4.6 Validity

Albery, Chandler, Field, Jones, Hammond, Messer, Moore, Sterling, Sutton and Trapp (2014: 25) state that validity is the extent to which a study produces accurate results that are widely applicable. Haradhan (2017: 59) opined that both external validity and internal validity are crucial when performing experiments. The National Institute of Health Research (2020) defines validity as the extent to which the method of measure is capable of providing measure(s) that match the true value. Reinard (2006: 137) stipulates that external validity involves the degree of generalizability of the findings to a population. The external validity in this research study were assured by selecting appropriate data collection and data analysis methods which are proven to be valid in the reviewed literature in Chapter 3. Studies conducted by Antony (2014: 204), Gopalakrishnan (2012: 133) and Rumane (2017: 120) indicate that the Six Sigma DMAIC methodology employed in this thesis is validated to provide generalizations about the population of a study.

Albery, *at el*. (2014: 25) contend that internal validity refers to the extent to which the actual test results of an experiment pass the hypothesis test claim. Anusree, Mohapatra and Sreejesh (2014: 90) add that internal validity involves proper experimental design in order to make valid conclusions about a study that are free of errors. In this study, internal validity was assured by making use of validated test procedures and externally maintained and calibrated measuring instruments, which included:

- Sampling methods – for collecting primary and secondary data,
- experimental procedures – including the operation of the instruments that were used to measure CGAC mass, effluent volume, pH, conductivity and COD concentrations, and

- external calibration of the instruments that were used, and validated using a SANA accredited supplier or service provider.

Moreover, in the Improve Phase, each measurement is done in duplicate, and the means are used for data interpretation, the experiments conducted in different environmental conditions. Speight (2015: 70), and Crouch, Holler, Skoog and West (2004: 94-95) stipulate that internal validity in experimental observations or scientific methods is verified by using accuracy testing or absolute error calculations. Absolute error calculations are performed using internal reference standard solutions; which are measured during the conduct of the trials in the Improve Phase. This provided assurance about the reliability of the data collected, and analysed in Chapter 5. The statistical tests that are used for evaluating the internal validity of in this study included absolute error, linearity, and one-way ANOVA tests.

4.6.1 Internal validity

Crouch, *et al*. (2004:94-95) mention two types of uncertainty that can affect the validity of measurements, namely random and systematic errors. De Jong, Monette and Sullivan (2011: 124) define systematic errors as troublesome errors might affect the accuracy of the results. Crouch, *et al*. (2004:94-95) mention three types of sources of systematic errors that can originate from the instruments, methods, and personnel. Chaudhry and Nakra (2004: 33) explain that random errors can be minimised by making use of standard operating procedures (SOPs). Kirkup (2002: 33) reports that random errors can measured using a precision measure of uncertainty known as the percentage relative standard deviation. Carter and Lubinsky (2016: 243) declare that relative or absolute internal validity of measurements involves assessing the precision of the individual measurements within a group. Internal validity was taken into consideration in this research study through demonstration that the measurements used are accurate and repeatable. In Chapter 5 of this study, the interpretation of random errors is provided to the reader to present the percentage of uncertainty in the obtained data.

Internal validity analysis in this study began by presenting the accuracy (%bias) and precision (%RSD) of the reference standards data, namely 2000 ppm, 5000 ppm and 10 000 ppm. Part of the reference standards data are collected by the WCM and employed to prove the validity of the data used in the Measure Phase. Part two of the reference standards data are collected during the conduct of the two trials in the Improve Phase, to demonstrate internal validity of the measurements. T-tests and ANOVA testing is performed to interpret the validity of the reference standard to demonstrate the validity of the measurements obtained during the trials.

4.6.1.1 Application of hypothesis testing within internal validity

Mwavita and Strunk (2020: 82) comment that a t-test is a trustworthy measure that helps with probability analysis of measurements in association with any given true value. Oak Ridge National Laboratory (2002: 16) states that a t-test is a suitable hypothesis test to evaluate the accuracy of the measurements against the ideal value. MockInterview.co (2018) comments that a t-test is used to statistically evaluate if there is a difference between the measurement results and the control results. Applicable in this study, the t-test is used to present the results of deviation or variance obtained from the internal reference standards in order to demonstrate the effect of the systematic error of the data collected.

The reference standard measurements obtained at each set of the trials (which include trial one - day one, trial one - day two, trial two - day one and trial two - day one) were tested using a two-tail t-test. The two-tail t-test is used to assess if all the means obtained from the sets had no significant difference compared to the true values for the three reference standards, at a 95% confidence limit. The claims for each reference standard for the trial sets are presented in **Table 4.7** below.

Table 4.7: Two-tailed t-test data interpretation plan for the internal validity of the reference standard data to be collected with the trial data in Chapter 5

Name of the reference standard	Claim
2000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$
5000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$
10 000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$

Haber, *et al*. (2013: 228) claim that ANOVA can be used to examine the difference between more than two groups. De Bievre and Gunzler (2002: 91) declare that the one-way ANOVA test is a suitable hypothesis testing tool to examine the standard of uncertainty and repeatability of the measurements. Gamst, Guarino and Meyers (2013: 521) note that the one-way ANOVA test is suitable to examine the repeatability of the measures of the same variable under different conditions. Jones, Tohen and Tsuang (2011: 77-79) used one-way ANOVA to test the repeatability of the results to provide their estimated variation error. In this research study, one-way ANOVA testing is adopted to prove repeatability of measurements taken during the conduct of the experiments.

The one-way ANOVA tests are used to examine if there is no variance in the means of the same reference standard measurements collected at different times and conditions of the conduct of the trials.

These tests are conducted at a 95% confidence limit to demonstrate that the instruments and the method used during the conduct of the experiments were able to provide reproductive measurements during the trials. The one-way test claims for each reference standard is presented in **Table 4.8** below.

Table 4.8: One-way ANOVA analysis plan for internal validity using data to be collected from the reference standards during the conduct of the trials

Name of the reference standard	Hypothesis claim
2000 ppm	H_0: $X_{Tria1\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$ H_1 : $X_{Tria1\text{-}Day1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$
5000 ppm	H_0: $X_{Tria1\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$ H_1 : $X_{Tria1\text{-}Day1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$
10 000 ppm	H_0: $X_{Tria1\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$ H_1 : $X_{Tria1\text{-}Day1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$

Oak Ridge National Laboratory (2002: 17) reports that a correlation coefficient determines a linear relationship between the actual measurements and the ideal measurements. Badgett and Christmann (2009: 116) write that a coefficient correlation is a primary statistical concept used to test the validity of measurements. Jensen, *et al*. (2014: 478) report that a correlation coefficient measure provides the researcher with an analysis of the accuracy of the measurements collected in an experiment. Abraham, *et al*. (2003: 55), agree that the correlation coefficient is used to statistically estimate the correlation of actual results compared with expected results. Badgett and Christmann (2009: 116) note that a negative coefficient at any level signifies invalid test results, and tests with a positive coefficient of +0.9 signify highly valid results. In Chapter 5, the correlation studies are employed to prove the capability of the instruments and experimental method in providing with accurate measurements.

Applicable in this study, two measurements of each reference standard were measured in duplicate to prove that both the SOP and instruments are reliable and provided accurate measurements.

4.6.1.2 Confidence intervals

Jones (2002: 135) defines confidence intervals as a range of the values in which a mean is likely to fall with a specified level of confidence or certainty. Investopedia (2018) explains the confidence interval as a probability that a measured value will fall between an upper and lower bound of a probability distribution; the author further comments that the confidence interval probability ranges from 95% to 99%. Jawlik (2006: 102-107) adds that a 95% confidence interval corresponds to a 95% confidence level and 0.05 or 5% of the level of significance: the maximum allowed percentage of error. Ellis, Ogee and Pammer (2018) note that confidence intervals are commonly used in hypothesis testing to validate the claim made about the test. In this research study, a 95% level of confidence is employed during the analysis of the COD measurements that are obtained using different GAC filtration methods (conditions).

Ary and Suen (2014: 99) note that validity, which includes internal validity or reliability and external validity, is key to assuring effective high quality quantitative research. These authors explain that external validity involves the ability of the collected data to reflect the underlying attribute of interest. Macnee and McCabe (2008: 199) note that if a study lacks internal validity, it automatically lacks external validity. Therefore, both internal and external validity are treated as a priority in this study. External validity taken into consideration in this study is discussed in the next point.

4.6.2 External validity

Warner (2008: 18) declares that external validity is the extent to which the findings of a research study can be generalized beyond the specific setting, and be applied to real world situations. Felbinger and Langbein (2006: 34) acknowledge that external validity concerns the conclusion of the inferences made in a particular study. Macnee and McCabe (2008: 199) add that external validity refers to the ability to infer that the findings for a particular sample can be applied to an entire population.

Kite and Whitley (2013: 213) mention two aspects of external validity in a study, “generalizability across” (the results can be generalized to more than one setting or population) and “generalizing to” (the results can only be generalized to a particular setting or population). Macera, Shaffer and Shaffer (2013: 151) acknowledge that external validity includes generalizability and representativeness of the results of the studied samples to different conditions. According to Albery, *et al*. (2014: 25), external validity presents the extent to which the results of the experiment can be applied to other situations. Maruyama and Ryan (2014: 39-40) expand that external validity specific to an experiment asks a question pertaining the extent of generalizability in which the findings can be applied to different groups, settings, subject, and under what conditions the experiment can be generalized. Felbinger and Langbein (2006: 35-36) also agree that external validity provides a degree to which the conclusions in one`s study can be accomplished by other persons in other places and times.

According to Cottrell and McKenzie (2005: 173), external validity includes the ability to conduct experiments and achieve similar results in different environmental conditions or settings. McKay (2008: 32) reports that external validity can be assured by assuring that the selected sample in the study is a representative of the target population. Bauman (2013: 212) comments that external validity can be maximized by a randomized sampling of the variable in a research study. McKay (2008: 32) adds that another approach to assuring external validity is to conduct multiple studies across different sample sub-groups, settings or conditions and times. External validity is provided to the reader in Chapter 5 of this study, derived from the research design that includes sampling size and a data collection design plan. It helped the researcher to make valid findings in this study in relation to the real word. It is discussed in greater detail in Chapter 5 to provide assurance using externally approved, validated instruments for testing; standard operating procedures for collecting the primary data (experimental data); and the experiments were performed on different days, times and conditions.

4.7 Considered ethics

Concerning the ethics in this study, since this is an experimental research design and not social science research, there were no human research participants. However, permission from the company to collect data was granted and the protection of the company's identity in this research was agreed upon. Moreover, this research was conducted in accordance with the CPUT Faculty of Engineering and the Built Environment Ethics Guideline.

The Department of Industrial and Systems Engineering takes student researchers through all the ethical guidelines before the conduct of the actual research. This helps the researcher to be aware of the ethical requirements expected from him or her. Upon completion of the ethical research training, the researcher is then required to complete the ethical clearance form that pinpoints all the ethics that must be adhered to. Hence the ethical clearance letter that was issued by the WCM management granting the researcher permission to use their data and pertaining to the agreement of the protection of the company. Due to the nature of this study, humans and animals were not used for collecting the data, therefore, they were excluded. All this was done to ensure that the ethical compliance agreement between the relevant parties, including the assurance of the quality and integrity of the research.

4.8 Chapter 4 summary

This chapter first provided the reader with an overarching view of different research philosophies and worldviews. Then the chapter outlined the specific research methodology used by this study, namely Six Sigma DMAIC. The population and sampling plan was highlighted and data collection methods were discussed in detail. Thereafter, a detailed data analysis plan in each DMAIC phase was presented. The description of the measures that were taken to ensure internal validity and external validity of this research study were presented. In conclusion, the ethical considerations applicable in this study were discussed. In conclusion, validity (internal and external) was discussed.

The next chapter, Chapter 5 will present to the reader with analysis and interpretation of the data, following Six Sigma DMAIC methodology.

CHAPTER 5: DATA ANALYSIS

5.1 Introduction to Chapter 5

A research data analysis involves data interpretation using five aspects, namely credibility, meaning, importance, generalization of the extent and the implications of the findings (Beck & Polit, 2008: 653). In succession of data interpretation, the researcher presents the analysis to make deductions or inductions about the data (De Chesnay, 2015: 14). Guided by these definitions of data analysis, this chapter commenced by presenting data interpretation and analysis of the results, obtained using the Six Sigma DMAIC methodology. Then conclude with the presentation of the results of validity.

5.2 Overview of Chapter 5

Data analysis is the process through which inferences are drawn about the data available after its interpretation (Sharma, 2011: 8). Data analysis serves as the foundation of the improvement cycle by providing results that illustrate the effectiveness of current methods, procedures, and structures (Depka, 2006: 3). Data interpretation and analysis are used to examine data of this study and to draw conclusions regarding the research problem of this study.

Essentially, the Define Phase presents a detailed outline of the WCM effluent process, by identifying all the process inputs and output of each WCM effluent process steps. In the Measure Phase, the current performance of the WCM effluent process are measured, by examining the performance of the WCM effluent macro channels using the hypothesis t-tests. The findings of the Measure Phase provided a foundation for the Analyse Phase. Here the identification of the root causes of the problematic effluent macro channels took place with the use of the RCAs constructed using the Fishbone and 5 Whys method. The root causes that were identified in the Analyse Phase signified the areas to improve in the Improve Phase; where the improvement solution actions are developed and tested to address the research problem. The improvement solutions in the Improve Phase include scientific interventions to determine the most effective solution. The effectiveness of the solutions are examined for sustainability in the Control Phase using the FMEA tool. Then the internal validity was presented to the readers using selected statistical techniques, such as percentage bias, t-test, one-way ANOVA testing and the coefficient of determination. This is followed by the external validity considered in this study, from planning to execution. This chapter will conclude by presenting ethics that were considered in this research.

5.3 Define Phase

A process flow can be used in Define Phase to assist the analyst to gain a better understanding of the process being studied (Meredith and Safer, 2019: 276). The process flow is used to exhibit a detailed WCM effluent process flow, to identify all its process steps or macro channels. The process flow is created during a Gemba walk in the WCM effluent plant with the purpose of gaining a better understanding of the layout of the WCM effluent process. Thereafter, an analysis of measurement was conducted on each sub-process or macro channel.

5.3.1 Wet Corn Milling effluent process flow

The process flow shown in **Figure 5.1** provides the reader with the insight into the stages of the WCM effluent process. To demonstrate the complexity of the WCM effluent process, this comprehensive effluent process flow was developed. This detailed effluent process flow was created to illustrate the complexity of the WCM effluent process. It depicts all the effluent process steps (macro channels); which had input to the total WCM effluent. The process flow shows that the process has thirteen macro effluent channels. The process spillages are also included in the process flow, as they had a direct effect on the WCM effluent plant COD.

The WCM effluent process is divided into five categories; category one includes all the effluent generated by the corn steeping, separation processes of the steeped corn, and the drying of the by-product processes. These effluent channels include the Evaporator, Wet Corn Milling Spillages, Condensate and Condensate Return. Category two includes all the effluent generated by the modification of the starch and refining stages of Glucose as a by-product. The Glucose Spillages, Hot Water, CPV Tank, and Concentrator are the effluent channels in the secondary category. Category three includes Saccharification, Spillages, Anion, Cation and the Effluent Tank; these are the effluents generated by the refining of glucose and germination of the starch. All the effluent generated by all three categories is collected in the Effluent Sump, which is the fourth category. The fifth category includes the product spillages from the loading bay.

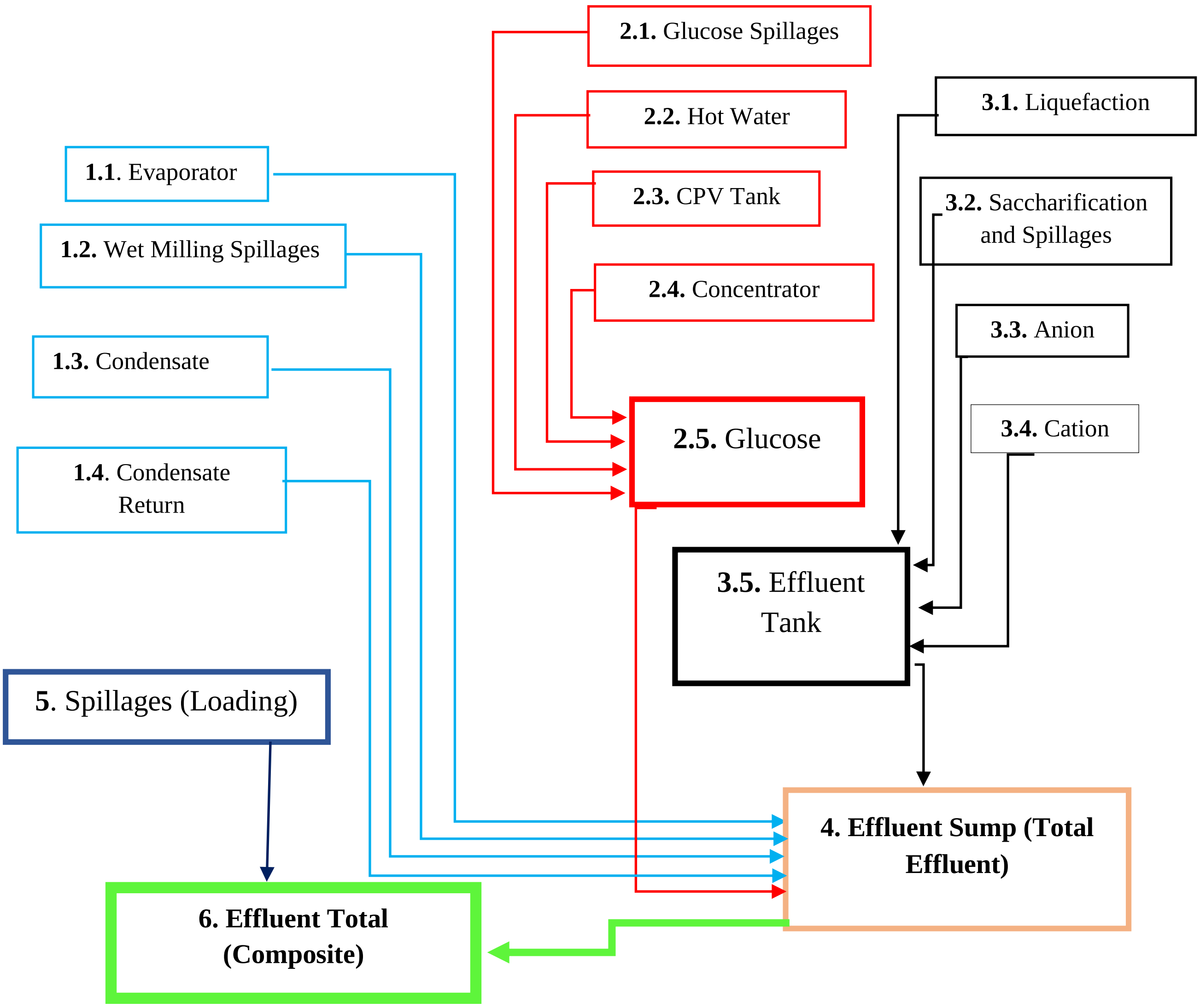

Figure 5.1: Wet Corn Milling effluent process flow

The conclusion of this phase is summarised in the next section below.

5.3.2 Define Phase outcome summary

Condensate Return, Concentrator, Evaporator, Hot Water, CPV Tank, Liquefaction, Condensate, Anion, Cation, Glucose and Effluent Tank are the eleven key effluent macro channels in the WCM. As the units of analysis in the Measure Phase, the eleven macro channels are used; following the reviewed literature in section 3.4.3, which revealed that flow chart can be used in Define Phase to help to classify major primary data collection areas (Davis & Yen, 2000: 27). The quality of the effluent COD concentrations in the effluent generated from the eleven effluent macro channels are presented in the next Phase, Measure. The objective of the measurement of the eleven effluent macro channels is to determine the macro channels that had an impact on the nonconforming CODs in the WCM effluent.

5.4 Measure Phase

The one-tail test is used in this Phase to interpret the current state of the process or sub-process (Kadambi, 2012: 40). Results of one-tail t-tests are used to compare the measurements of COD concentrations produced in each macro channel to the standard specification requirement of government controlled COD concentrations. The goal of this Phase is to identify the effluent macro channels that produce COD concentrations greater than the standard specification of 5000 ppm required by the legislation. Therefore, reply to the first investigative question of this research study: "Which stages in a WCM process contribute to increasing concentrations of COD in the discharged effluent?"

5.4.1 One-tailed t-test analysis within the Measure Phase

The null hypothesis claims for all the t-test are: The COD concentrations in the effluent generated by each effluent macro channel is less than or equal to 5000 ppm (H_0: µ COD concentrations $\leq$ 5000 ppm). The alternate hypothesis claims are: the COD concentrations of effluent generated by each macro channel is greater than 5000 ppm (H_1: µ COD concentrations > 5000 ppm).

At 95% confidence limits and at the degrees of freedom of n-1(59), the upper t-critical value was found to be 1.6710. The t-statistics of three of the eleven macro channels, namely Condensate, Effluent Tank and Glucose were found to be greater than the t-critical values. Therefore, their null hypotheses were rejected. Their alternate hypotheses were accepted, which stated that their COD concentrations means are significantly greater than 5000 ppm. See the t-tests result for all the eleven WCM effluent macro channels in **Table 5.1** below, and the calculations presented in **Appendix J**.

Table 5.1: T-test results for eleven WCM effluent macro channels

Effluent macro channel	Test claim	t-critical	t statistical	Decision (Accept or Reject)
Condensate Return	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-1012.8871	Accept the null hypothesis
Concentrator	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-524.8158	Accept the null hypothesis
Evaporator	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-129.0892	Accept the null hypothesis
Hot Water	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-127.3751	Accept the null hypothesis
CPV Tank	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-93.7038	Accept the null hypothesis
Liquefaction	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-35.5278	Accept the null hypothesis
Condensate	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	16.8249	Reject the null hypothesis
Anion	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-1.3211	Accept the null hypothesis
Cation	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	-0.8218	Accept the null hypothesis
Glucose	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	5.0150	Reject the null hypothesis
Effluent tank	H_0: $\bar{x}_{\text{COD concentrations}} \leq 5000$ ppm H1: $\bar{x}_{\text{COD concentrations}} > 5000$ ppm	1.671	7.4328	Reject the null hypothesis

The t-tests result indicated that there were three WCM effluent macro channels that contributed to the nonconforming COD concentrations in the WCM effluent plant.

5.4.2 Measure Phase summary

For the three WCM effluent macro channels, the null hypothesis was dismissed on the grounds that there was substantial statistical evidence that their mean COD concentrations were significantly larger than 5000 ppm. This was therefore the answer to the first investigative question of this study: "Which stages in a WCM process contributed to increasing concentrations of COD in the discharged effluent?" Statistically, the three WCM effluent macro channels, namely Condensate, Glucose and Effluent Tank, were found to have higher mean COD concentration than the 5000 ppm controlled standard specification.

In line with the literature reviewed Chapter 3, section 3.4.5.1; which points out that the findings of the Measure Phase act as inputs to the Analyse Phase (Vanzant-Stern, 2012: 117). Therefore, the inputs of the RCAs are carried out in the next research step (Analyse Phase) are these three WCM effluent macro channels.

5.5 Analyse Phase

The root cause analysis is performed during the process to figure out what happened in the three macro channels that were established in the previous phase as problematic. Carrell and Peterson (2010: 22), who clarified the intent of the Analyse Phase as to determine what should happen to prevent the problems recurring. Then Antony (2014: 205), expounded that the RCA is a brainstorming tool to examine the root cause of the research problem; which include the Fishbone diagrams and 5 Whys methods. In this analysis, directed by Antony, the Fishbone diagrams is used to describe the possible causes of high COD concentrations caused three WCM effluent macro channels, namely Condensate, Effluent Tank, and Glucose.

In order to comply with the regulatory requirements, the root causes of non-conforming CODs are known to present risks. Identifying the root causes will help to improve the alternatives (appropriate correction and preventative). Using the Fishbone diagrams and 5 Whys approaches, the identification of the root causes of the high COD concentrations is studied. Led by the literature review presented in Chapter 3, section 3.4.5.2 in Chapter 3; which pin pointed out that the 5 Whys approach may not actually have to include specifically 5 Whys questions, but to include at most 5 explanations why to get to the root cause of the issue to peel off the layers of (Carrell & Peterson, 2010: 22). For this reason, in this study, only 3 Whys are used to navigate to the root cause of the symptoms; to find answers to the second question of this research study, namely "What are the causes of the high COD content present in the discharged effluent of the various process stages?"

The RCA is created for the three effluent macro channels that were identified as the problematic channels in the preceding Measure Phase. As Cahyadi, *et al*. (2017: 850) stated in section 3.4.6, in Chapter 3 that the Improve Phase sets out to eliminate the root cause(s) of non-compliance identified during the Analyse Phase. The findings of this Phase are the keys to find solutions to the researched problem; the analysis of each channel are discussed in the section that follows.

5.5.1 RCA analysis for the Condensate channel

Fewer than 5 Whys can be used to classify the root causes of a nonconforming process, according to Isixsigma (2020). Consequently, only three Whys were necessary from the three macro channels to classify the root causes of high COD concentrations. The results of the Condensate channel RCA suggested that the interlinked processes were not properly monitored or regulated, resulting in high COD level caused by cross-contamination of the substance. The explanation for this is that hydrocarbons, proteins and oils that are generate by wet maize processing, and if the interlinked processes are not properly regulated, these elements may contaminate the effluent and thus give rise to higher concentrations of COD. In addition, if one or more of the processing stages of the WCM are not effectively regulated, some of the products can be channelled into the macro channel(s) of the effluent and the COD concentrations in the effluent can be elevated. The RCA also emphasized that the existing system in place are not adequate to identify accidents quickly and prevent from occurring. **Table 5.2** below presents the results of the RCA for the Condensate macro channel in tabular form.

Table 5.2: Condensate channel RCA outcomes

Category	Potential root cause	Why 1	Why 2	Why 3	Root Cause
Mother nature/material	Poly saccharides	Inefficient separation in the WCM processes	Poor process operation	Gap in competency	Skills gap/level of understanding
Machine	Spillages	Blocked pumps/tank overflows	Nature of the product being processed/ lack of tank monitoring	No system to detect these incidents sooner	No systematic approach to detect the problems sooner
Measurements	Reactive rather than proactive	Taking a long time to sample and perform the measurements	The current sampling and analysing method used	Gap in technology innovation	Current measuring system is too reactive

The RCA above identified the level of knowledge of the process operators, and ineffectiveness in the current system used to measure COD deviations and alert the process operators when there was a deviation. To overcome these risks, it is proposed that improvement on skill development, and COD detection, is required to improve the monitoring of the WCM processes to ensure that each process stage does not leak the products into the effluent channel. A more proactive measuring system should be implemented in place to reduce the risks.

5.5.2 RCA analysis for the Effluent Tank channel

The findings obtained from the RCA of the Effluent Tank macro channel suggested that the two factors are responsible for the high COD concentrations. High COD concentrations that are produced during the regeneration process of anions and cations resins used to extract ions from the glucose syrups are factor one. The management of high COD concentrations produced during resin regeneration is not adequately addressed by the current procedure and system in place. Factor two is that there is an inadequate monitoring mechanism for COD concentrations to notify the process operators rapidly when the COD concentrations begin to deviate or elevate. This leads to the late identification of high levels of COD caused by leaks or product spillages, defective pumps or blocked channels. **Table 5.3** below presents the results of the RCA for the Effluent Tank macro channel.

Table 5.3: Effluent Tank channel RCA outcomes

Category	Potential root cause	Why 1	Why 2	Why 3	Root Cause
Mother nature/ material	Regeneration outputs	Release of poly hydrocarbons from the regeneration process	No process designed to treat the contaminate effluent	Current procedure does not cater for the process	Inadequate procedure
Mother nature/ material	Poly saccharides	Product cross-contamination	Product over flow	Blocked machine	ineffective COD detection system

The RCA above identifies the inadequate effluent management procedure, ineffective COD detecting control measure and preventative maintenance risks in this macro channel. To overcome these risks, the current procedure for managing the nonconformities in the WCM effluent should be reviewed for improvement, including the current COD detection and preventative maintenance.

5.5.3 RCA for the Glucose channel

The results obtained from the RCA Glucose channel indicated that there is a gap in the defined processes stipulating the actions that must be taken when to correct and prevent high COD concentrations when detected. Moreover, the existing system used for measuring or detecting high COD is not proactive or vigilant because it does not provide the results early enough to predict or anticipate the next deviation from the process deviation. Rather, it only waits and detects after the process has deviated from the normal. The Glucose channel`s RCA findings are presented in **Table 5.4** below.

Table 5.4: Glucose channel RCA outcomes

Category	Potential root cause	Why 1	Why 2	Why 3	Root Cause
Methods	No effective procedures to monitor the tank levels	It was not included in the process risk assessment	At the time of the establishment of the process risks, the effluent was not rated as a major risk	The quality of the effluent was not as important as it is now	Inadequate procedures for COD effluent risk identification and management
Machines	Product spillages	Blocked product channels, and faulty tank level indicators	Cold product left on the paths solidifies	The methods in place are not adequate and they are not followed	Inadequate operating procedures to address operational risks and preventative measure that need to be followed when there is an incident
Measurements	Product cross-contamination low detection	Late detection of contaminated effluent	Sampling and testing take too long	Current system implemented	Inadequate resources

The RCA above identifies a lack of procedures and resources as risks in this process. To overcome these risks, it is proposed that effective control measures should be implemented, such as establishment of procedures that stipulate how to correct and prevent pre-identified risks to ensure that the effluent discharged into the municipal drains adheres to regulated COD concentrations.

5.5.4 Analyse Phase summary

The results of the RCAs presented on the three identified nonconforming WCM macro effluent channels illustrate that there is a gap in the skills, ineffective COD control measures or a system and inadequacy in the implemented procedures in place. Suitable control measures are not available to detect, correct and prevent risks which might trigger an elevation in the COD concentrations in the WCM effluent.

This necessitates the establishment of a procedure that will help to improve the monitoring of the COD concentrations in the WCM effluent, to consistently meet the legislated standard specification, which is explored in the next phase.

5.6 Improve Phase

Following Cahyadi, *et al*. (2017: 850) statement in section 3.4.6 in Chapter 3, that say, the solutions of the problems identified in the Analyse Phase are tested in Improve Phase. In this Phase, the CGAC adsorption experiments are conducted to explore the most suitable procedure for removing COD concentrations in the WCM effluent. The goal of this Phase is to execute the action solution plans to eliminate the root causes of a problem (Tibbetts & Williams, 2006: 6). In the previous Phase, the root causes of high COD concentrations in the identified three WCM effluent macro channels and this Phase focuses on establishing an effective procedure for reducing the COD concentrations in the WCM effluent using the CGAC adsorption process.

In this Phase, the researcher tested different experimental procedures of CGAC to reduce the COD concentrations in the WCM effluent samples, with the aim of answering the third investigative question of this research study, which states: "Can a process variable(s) be identified that when adjusted yields the optimal removal of COD from WCM effluent?"

This is accomplished by means of conducting experimental trials to collect primary data, which is later interpreted and analysed using paired t-tests, correlation studies and independent-sample t-tests.

The primary data is collected from two experimental trials, namely trial one and trial two. Each trial consisted of three batches with different procedures used for evaluating the effectiveness of the CGAC procedure to achieve optimal COD removal. The two trials are conducted using the WCM effluent samples collected from the Condensate, Effluent Tank and Glucose non-conforming COD macro channels. The two trial procedures are presented in **Appendix 1:** Where trial one employed acidic range of the effluent samples, and trial two involved alkaline range effluent samples. The trial batches are outlined as follows:

1. **Trial one & two: batch one –** Evaluating the effect of an increase in contact time (20, 30 and 60 minutes) at constant CGAC filtration weight of 20 gramss.
2. **Trial one & two: batch two** – Evaluating the effect of an increase in contact time (20, 30 and 60 minutes) at constant CGAC filtration weight of 30 gramss.
3. **Trial one & two: batch three** – Evaluating the effect of an increase in contact time (20, 30 and 60 minutes) constant CGAC filtration weight of 40 gramss.

The interpretation of the results obtained in trial one and trial two batches are conducted using paired t-tests and correlation studies. The interpretation tactic is guided by Butler, *et al*. (2016: 163), as they described a paired t-test as a test that is used to determine significant differences in the changes before and after the improvement implementations. Furthermore, Javier (2011: 144) used a paired t-test result at 95% confidence limits to examine the differences between two results obtained from independent variable(s). Therefore, in this Phase, initially, the paired t-tests are used to explore mean differences between the untreated COD concentrations and the treated COD concentrations. Ramu (2017: 51) states that correlation studies are employed in the Improve Phase to estimate the significant of the improvement; applicable in this study, the correlation studies are used to identify a CGAC filtration procedure that would result in good and positive correlation. Lastly, Martinez, *et al*. (2020: 181) reported that the two independent sample t-tests are used to compare two groups in the Improve Phase to determine if there was a statistically significant relationship. In this study, the t-tests are conducted to explore the statistical relationship between the reduced COD concentrations obtained when using two different pH content samples, namely acidic and alkaline samples.

5.6.1 Trial one paired t-test analysis within the Improve Phase

The paired t-tests are used to examine if there is a significant difference in the COD concentrations means before filtration versus after filtration in the acidic effluent samples. The purpose of this test is to determine if the treatment had a significant effect on removing the COD concentrations in acidic effluent samples.

All the paired t-tests results indicated that there are significant differences in the COD concentrations means before and after the CGAC filtration. This is demonstrated by the rejection of all the null hypotheses, which stated that there is no significant difference between the COD means before and after the filtration at confident limits of 95% and degrees of freedom of 2 (n-1). All the calculated t-tests values fell outside the non-rejection area; as a result, the alternate hypotheses are favoured for all the tests. Therefore, there is enough statistical evidence to prove that there are significant differences between the COD concentrations means before and after the CGAC filtrations. The calculations are documented in **Appendix K1** and the results are summarised in **Table 5.5** below.

Table 5.5: Paired t-tests result for the mean difference analysis between the COD concentrations before and after treatments for trial one

Trial Name	Hypothesis claim	t-statistical	t-critical	Decision (Accept or Reject)
Trial 1: Batch 1	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.0151	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis
Trial 1: Batch 2	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.0167	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis
Trial 1: Batch 3	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.0824	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis

It is deduced from the results obtained using the paired t-tests that CGAC did reduce the COD concentrations in the acidic WCM effluent samples. This deduction is supported by the rejection of all the null hypotheses which stated that there is no difference in the COD concentrations before and after the filtration.

5.6.2 Trial one correlation analysis within the Improve Phase

The correlation analysis is conducted to identify which suitable CGAC procedure that would result in a good positive relationship when applied in acidic effluent samples. This analysis is conducted to investigate the most stable and consistence procedure for the optimal removal of the COD concentrations in acidic WCM effluent samples. By exploring the effect of the variables, namely filtration time and CGAC filtration weight.

Statistically, based on the sample size of this study, a better positive correlation is obtained when the CGAC weights are increased to a constant contact time of 60 minutes, the maximum time used in this study. This interpretation is guided by Brewer and Picus (2014: 826) as they acknowledged a coefficient determination of 0.7 to 1 as good relationship. Therefore, a good correlation value of 0.7565 is achieved; which statistically implies that 75.65% of the relationship in the removal of the COD concentrations can be explained by the linear graph. The remaining 24.35% was subjected to other external factors which include the pH content of the samples used and the chemical composition of the effluent samples used. The results obtained during this trial are presented in **Table 5.6** below using the experimental data and the calculations that are presented in **Appendix K2.**

Table 5.6: Coefficient of determination results for trial one

Treatment	Coefficient of determination
20 g: 20 min, 30 min and 60 min	0.05633
30 g: 20 min, 30 min and 60 min	0.05033
40 g: 20 min, 30 min and 60 min	0.38120
20 min: 20 g, 30 g, and 40 g	0.3971
30 min: 20 g, 30 g, and 40 g	0.1275
60 min: 20 g, 30 g, and 40 g	0.7565

In trial one, the data interpretation indicated that when using acidic samples, positive linearity is obtained when the filtration time and CGAC filtration mass are increased. A good correlation for the COD removal is achieved when the contact time is 60 minutes using increasing CGAC weights. Therefore, it can be deduced that more consistent COD removal from acidic samples was achieved when the CGAC was increased gradually at the highest contact time of 60 minutes. The increase in contact time allowed molecules in the solute to react and adhered with the molecules on the surfaces of the CGAC. Consequently, resulted in more COD removal from the filtered effluent samples and this occurrence is supported with the reviewed literature in section 3.3.4.

Trial two experiments are carried out following the same CGAC filtration procedures used in trial one to explore the effect of alkaline samples on optimal COD removal in the WCM effluent. The effect of is examined using the paired t-test to evaluate if there was a statistically significant difference between the COD concentrations means before and after the filtrations.

5.6.3 Trial two paired t-test analysis within the Improve Phase

The purpose of conducting the paired t-tests is to determine if there would be a significant difference in the COD concentrations result obtained before and after CGAC filtration, using alkaline samples. All the paired t-tests result indicated that is a significant difference in the COD concentrations before and after the CGAC filtration of the alkaline samples. This is indicated by the rejection of the null hypothesis that stated that there is no significant difference between the COD means at confident limits of 95% and degrees of freedom of 2(n-1). All the calculated t-tests values fell inside the rejection area; as a result, the alternate hypotheses are favoured. Therefore, there is enough statistical evidence that there are significant differences between the COD concentrations before and after the filtrations when alkaline samples are used. The paired t-tests calculations are presented in **Appendix M1** and are summarised in **Table 5.7** below.

Table 5.7: Paired t-tests result for the mean difference analysis between the COD concentrations before and after treatment for trial two

Trial Name	Hypothesis claim	t-statistical	t-critical	Decision (Accept or Reject)
Trial 2: Batch 1	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.0408	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis
Trial 2: Batch 2	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.0987	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis
Trial 2: Batch 3	H_0: $\bar{X}_{\text{Difference}} = 0$ H_1: $\bar{X}_{\text{COD Difference}} \neq 0$	4.1048	2.1098 and -2.1098	Reject the null hypothesis and accept the alternate hypothesis

From the outcomes of this test it can be deduced using the paired t-test that all the CGAC adsorption procedures are able to reduce the COD concentrations from the alkaline WCM effluent samples. This is indicated by the rejection of all the null hypotheses from the nine different CGAC procedures, which stated that the COD concentrations before filtrations not different compared to the COD concentrations after the CGAC filtration.

5.6.4 Trial two correlation analysis within the Improve Phase

The correlation analysis in this trial is used to examine the effect of CGAC filtration weight and contact time on obtaining a good positive correlation, when using alkaline effluent samples. A positive correlation of 0.8801 is obtained when the contact time is kept constant at 60 minutes, while the CGAC weight was increased accordingly. The obtained coefficient of determination value statistically indicated 75.20% removal of the COD concentrations from the alkaline effluent samples. This is explained by the linearity obtained in this study. The results of the correlation analysis are presented in **Appendix M2**; see the results in **Table 5.8** below.

Table 5.8: Coefficient of determination results for trial two

Treatment	Coefficient of determination
20 g: 20 min, 30 min and 60 min	0.0017
30 g: 20 min, 30 min and 60 min	0.5849
40 g: 20 min, 30 min and 60 min	0.7166
20 min: 20 g, 30 g, and 40 g	0.4609
30 min:20 g, 30 g, and 40 g	0.7392
60 min: 20 g, 30 g, and 40 g	0.8801

The correlation analysis results of trial two demonstrated that when using alkaline samples, the best correlation for the COD removal was achieved when the contact time was kept constant at 60minutes for the increasing CGAC filtration weights. Therefore, this indicated that more COD removal in alkaline samples is achieved when the CGAC filtration weights are increased to a high contact time of 60 minutes. These results are similar to the one obtained when acidic effluent samples were used and the literature reviewed in this research; which indicated that an increase in time or GAC weight results in more removal of the COD in the effluent. Therefore, the outcomes of the correlation studies also demonstrated that the use of alkaline samples conceded to similar outcomes compared to the use of acidic samples pertaining to effect of filtration time and CGAC weight.

Paired t-tests results showed that when the CGAC treatment procedures are applied in acidic or alkaline samples, they are capable of removing the COD concentrations in the WCM effluent. Analysis of the results of trial one and trial two indicated that good linearity is achieved when the effluent samples (acidic or alkaline) are filtered through increasing CGAC weights for 60 minutes. Laerd Statistics (2018) defined the independent t-test as test used for determining a statistically significant difference between two means of two unrelated groups. Guided by this definition, the results of the independent t-tests used to explore the difference between the reduced COD data obtained using two pH content samples, acidic versus alkaline are discussed in the next point.

5.6.5 T-test for equal mean variances: A deduction

Nine t-tests are performed to statistically evaluate if there is a variance between the means of the reduced COD concentrations obtained using two different pH content samples, acidic and alkaline. The results of the t-tests indicated that there is no statistically significant variance between the reduced COD concentrations obtained, when eight similar CGAC procedures used to filter acidic and basic effluent samples. This claim is illustrated by the acceptance of the null hypotheses and rejection of the alternate hypotheses for the eight CGAC filtration procedures, at 95% confidence limits.

Initially, the null hypothesis was rejected for the last treatment, namely 40 g and 60 minutes, favouring the alternate hypothesis which stated that there is a significant variance between the reduced COD concentrations. Further analysis was conducted on the treatment with the rejected null hypothesis to validate if there was a variance using t-prime. The results of the t-test prime presented in **Appendix N** indicated that there is no significant variance between the reduced COD concentrations obtained for this treatment. Therefore, the rejection of the null hypothesis was reversed, and it is concluded that there is no difference between the reduced COD concentrations obtained when using the acidic effluent samples compared to alkaline effluent samples. See calculations of the summarised results in **Table 5.9** below in **Appendix N**.

Table 5.9: T-tests result for equal mean variance in reduced COD concentrations means obtained between alkaline and acidic effluent samples

Treatment	Hypothesis claim	t-statistic	Non-rejection area	Decision (Accept or Reject)
20 g & 20 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	0.1533	3.4954 and -3.4954	Accept the null hypothesis and reject the alternate hypothesis
20 g & 30 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	1.2705	3.4954 and -3.4954	Accept the null hypothesis and reject the alternate hypothesis
20 g & 60 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	1.5283	3.4954 and -3.4954	Accept the null Hypothesis and reject the alternate hypothesis
30 g & 20 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	0.7217	3.4954 and -3.4954	Accept the null hypothesis and reject the alternate hypothesis
30 g & 30 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	0.3253	3.4954 and -3.4954	Accept the null hypothesis and reject the alternate hypothesis
30 g & 60 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	1.5021	3.4954 and -3.4954	Accept the null Hypothesis and reject the alternate hypothesis
40 g & 20 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	1.2117	3.4954 and -3.4954	Accept the null Hypothesis and reject the alternate hypothesis
40 g & 30 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	0.9867	3.4954 and -3.4954	Accept the null Hypothesis and reject the alternate hypothesis
40 g & 60 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	5.2545	3.4954 and -3.4954	Reject the null Hypothesis and accept the alternate hypothesis
t-prime results for unequal mean variance				
40 g & 60 min	H_0: $\bar{X}_A = \bar{X}_B$ H_1: $\bar{X}_A \neq \bar{X}_B$	5.2545	4.1765 and -4.1765	Accept the null Hypothesis and reject the alternate hypothesis

All the t-tests result analysis indicated that there are no mean variances between the reduced COD concentrations, obtained when acidic and alkaline WCM effluent samples are treated in similar conditions. This claim is validated by the t-test outcomes, which imply similar results in the reduction of COD concentrations in the WCM effluent are obtained when using either acidic or basic effluent samples.

The results in **Appendix K1** and **Appendix K2** illustrated that more reduced COD means are obtained from the acidic samples; when acidic effluent samples are filtered through similar conditions compared to alkaline samples. This phenomenon is in agreement to what was reviewed in section 3.3.3.2 in

Chapter 3 of this study. Where the authors, namely, Ushakumary (2013: 7), Goswami and Kulkarni (2013: 181), El-Gawad and EL-Aziz (2018: 228) reasoned that the decrease in the pH content of the filtered solution results in more COD removal. However, the results of the t-tests presented in **Table 5.9** indicated that the differences between the reduced COD means are not big enough to result in a statistical significant difference at 95% confidence intervals in this study.

5.6.6 Improve Phase summary

The paired t-test outcomes indicated that there is a significant reduction in the COD concentrations before and after CGAC filtration when using either acidic or alkaline effluent samples. The results of the correlation studies showed positive good correction responses to the increased filtration times and CGAC filtration weights. Lastly, the t-test for equal and unequal mean variance indicated that there are no variances between the COD removal obtained using acidic effluent samples versus using alkaline effluent samples. Therefore, the results of the Improve Phase indicated that the CGAC adsorption procedures are capable of removing the COD from the WCM effluent.

Sobh (2008: 169) commented that the effective sustainability of the solutions of the Improve Phase are reviewed and validated in the Control Phase. For this reason, in the Control Phase, FMEA is adopted to explore all the risks of having high COD concentrations generated in the effluent current process flow. The use of FMEA is aimed to identify a procedure that could assist the WCM to ensure that the COD concentrations of the effluent discharged are consistently within legislated standard specification. Thus, provide an answer to the last investigative question of this research study: "Can a modified procedure be employed to reduce COD concentrations in effluent generated by the WCM process to comply with the regulatory specification consistently?"

5.7 Control Phase

The goal of the Control Phase is to ensure that improvement is maintained once effective solutions are identified (Larson, 2014: 134). Failure Modes Effects Analysis is applied in the Control Phase to assess the significance of the risks, and identify the control measures to eliminate them (DeCarlo, *et al*., 2012: 288). Failure Modes Affects Analysis is a systematic tool for establishing a procedure to eliminate identified risks to control the process (Arivalager & Naagarazan, 2005: 120). Guided by the explanations above, the FMEA tool is employed in this study to establish a suitable procedure for improving the monitoring of the COD concentrations in the WCM in accordance with the standard regulation specification. This is carried out to answer the last question of this research study in section 1.6.4 in Chapter 1.

The FMEA is used to first assess all the risks in the current effluent process, and to seek a mitigating action plan to eliminate the identified risks. Then the last part in this phase involved a re-evaluation of the effect of the mitigating action plan in eliminating the identified risks in order to create a modified procedure to address the preventative actions.

5.7.1 Failure mode effects analysis risk assessment of the current process

The high risk failures are indicated with red shading, medium risks with amber shading and low or insignificant failure risks are indicated with green shading. Two risks are identified as medium, namely a skills gap, and spillages that are caused by over-filled tanks. Four are identified as high risk, which include poor control of the inter-linked process stages, blocked channels or pipes, faulty valves, and regeneration of the deionized beds. The outcomes of the risks assessment are tabled in **Table 5.10** below, using the rating presented in **Appendices: O1, O2 and O3**.

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Table 5.10: FMEA risks assessment result of the WCM effluent process "*as it is*"

Risk - Bad events	**Consequences**	**Severity**	**Occurrence**	**Current process control**	**Detection**	**Risk Priority Number**	**Recommendation**
Skills gap	Ineffective process monitoring	9	5	Once-off entrance training and evaluations	3	135	Not applicable
Over-filled tanks that result in product spillages	Effluent contamination (non-conforming COD)	9	10	No control	4	350	Not applicable
Poor control WCM processing stages	High COD concentrations	8	10	Manual check	9	720	Install an inline COD detector at the total effluent and a warning alarm that will go off 4800 ppm.
Blocked channel flow or pipes	Leakages. overflows that cause effluent contamination (high COD concentrations)	9	8	Visual check	10	720	Install sensors on the critical process areas to detect if no flow of product
Faulty system (valves and detectors)	Effluent contamination/ product spillages	9	8	Visual check	10	720	Test valves using a systematic self-test programsme (maintenance), calibrate the detectors and perform daily verification of the validity of the warning alarms.
Regeneration of the anion and cation beds	Contamination of the effluent tank and overall WCM effluent	10	5	Manual check	5	250	Design a process to pre-treat the effluent generated during the regeneration of the beds.

The risks associated with the inability of the WCM effluent to consistently comply with the legislated standard requirement of COD concentrations are presented in the above FMEA analysis. In agreement with the views of De Carlo, *et al*. (2012: 288), in section 3.4.7, who opine that FMEA to eliminate risks by evaluating the effectiveness of each control measure, and Dhillon (2007: 60), who commented that FMEA is commonly used in the industrial sector to analyse engineering systems to improve their reliability; the corrective and preventative actions to reduce and eliminate the identified risks are discussed in the next point.

5.7.2 Risk mitigation action plan

The corrective and preventative actions for the four risks identified were used to finalise the development of a procedure to address the optimisation required to eliminate or reduce the risks to acceptable levels. This procedure included an action plan to redesign the current WCM effluent process to introduce measures that will eliminate the risks (see **Appendix P** for the proposed procedure). A recommendation from this analysis is that employees need to be re-trained and certified competent at least once per annum; and the plan must encourage the teams to improve performance by implementing team appraisals.

The current WCM effluent process needs to be redesigned to include measures that will control the identified risks. The redesigned WCM effluent process will help to improve the monitoring of the tank levels, thus preventing product spillages or wastage, and eliminating product wastage caused by faulty valves. Moreover, it will prevent high COD concentrations caused by regeneration of the resins. The redesigned process involves installing tank level indicators and installing a programsme with warning alarms to indicate when a tank is approximately 95% full. This programsme should automatically close the tank inlet valves when the tanks are full. This will eliminate product spillages caused by faulty valves and blocked channels, and eliminate non-conforming effluent COD concentrations caused by product spillage contamination.

In cases where the products are leaked due to procedures in the current processes that are not adequately controlled, automated inline COD detectors should be installed in all three problematic macro channel tanks and in the main effluent tank. These COD detectors must be programsmed to automatically give a warning alarm when the COD concentrations reaches 4800ppm, and completely stop the flow of the incoming contaminated channel(s), then open an alternate valve to channel the effluent with the contaminated COD concentrations into the COD pre-treatment tank until the COD concentrations are corrected.

There must be a high COD pre-treatment tank divided into two parts. The first part is to accommodate the non-conforming effluent, with an outlet valve into the second part of the tank. The second part should have a CGAC layer on top and on the bottom to increase the CGAC contact time with the effluent, as the correlation studies showed that an increase in contact time results in more COD removal.

The inline COD detector too must be installed inside the second part of the tank to measure the COD of the effluent during filtration; as Liptak (2003: 1229) and Rhosonics (2017) stated that on-line COD detector provide with quick COD results at source; therefore, in-line detection will help to improve the detection and management of nonconforming CODs. The tank should be designed with an outlet valve connected to the total WCM effluent tank. The level tank indicators should be installed in the second part at a certain level, and close the inlet valve from the first part of the tank once the tank reaches a certain level. The COD detector meter must be programsmed to close the outlet valve that allows the effluent to flow into the total effluent tank if the COD concentrations of the effluent is below 5000ppm. Inside the tank, a pump must be installed to circulate the effluent through the two CGAC beds.

This redesigned WCM effluent process will help to prevent high COD concentrations from contaminating the overall WCM effluent, thus preventing the discharge of nonconforming COD concentrations into the WCM effluent. The WCM effluent treatment tank design for COD is presented in **Figure 5.2** below, and the procedure is presented in **Appendix P**.

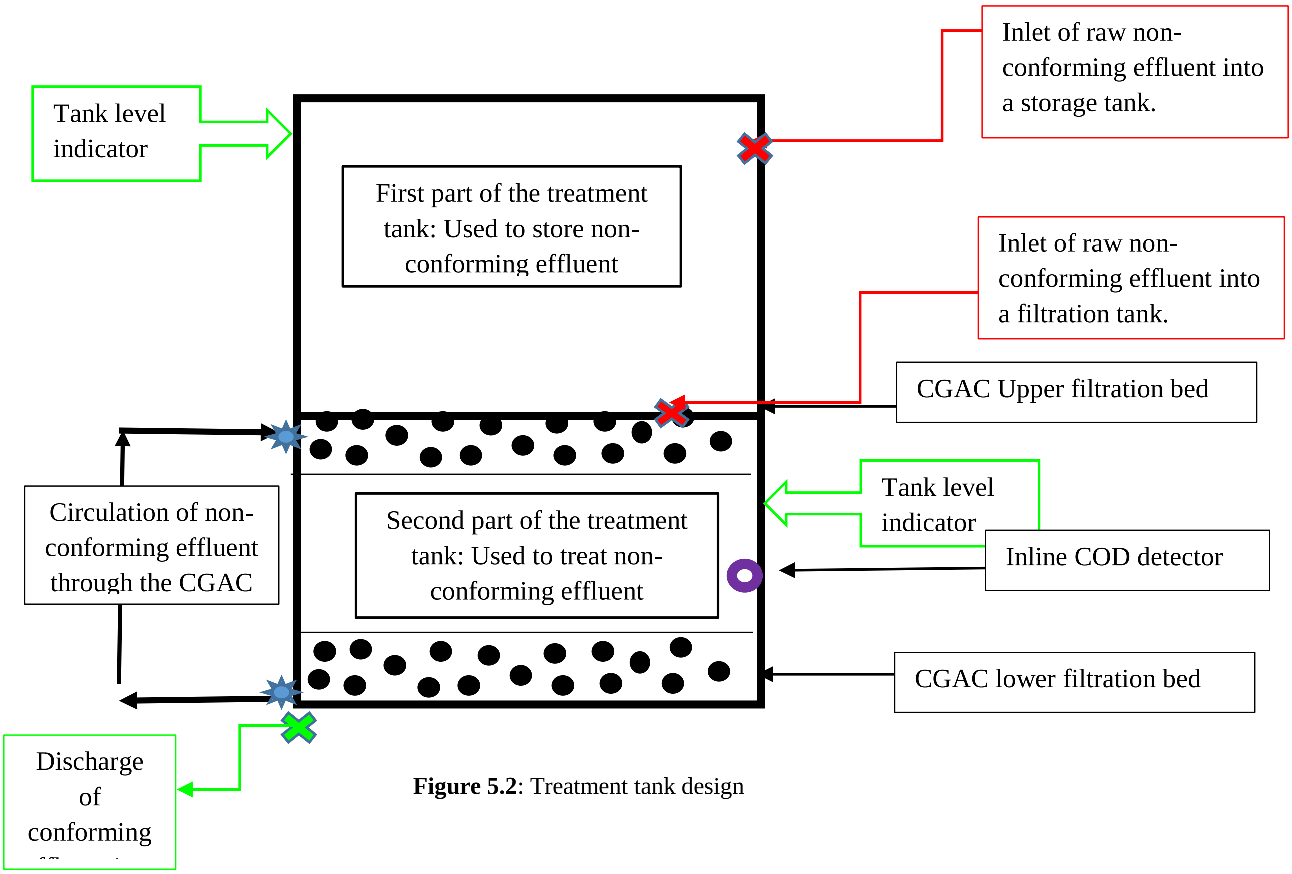

Figure 5.2: Treatment tank design

The new process flow presented in **Figure 5.3** below illustrates all the changes that could be made to improve the conformity of the COD concentrations in the WCM effluent.

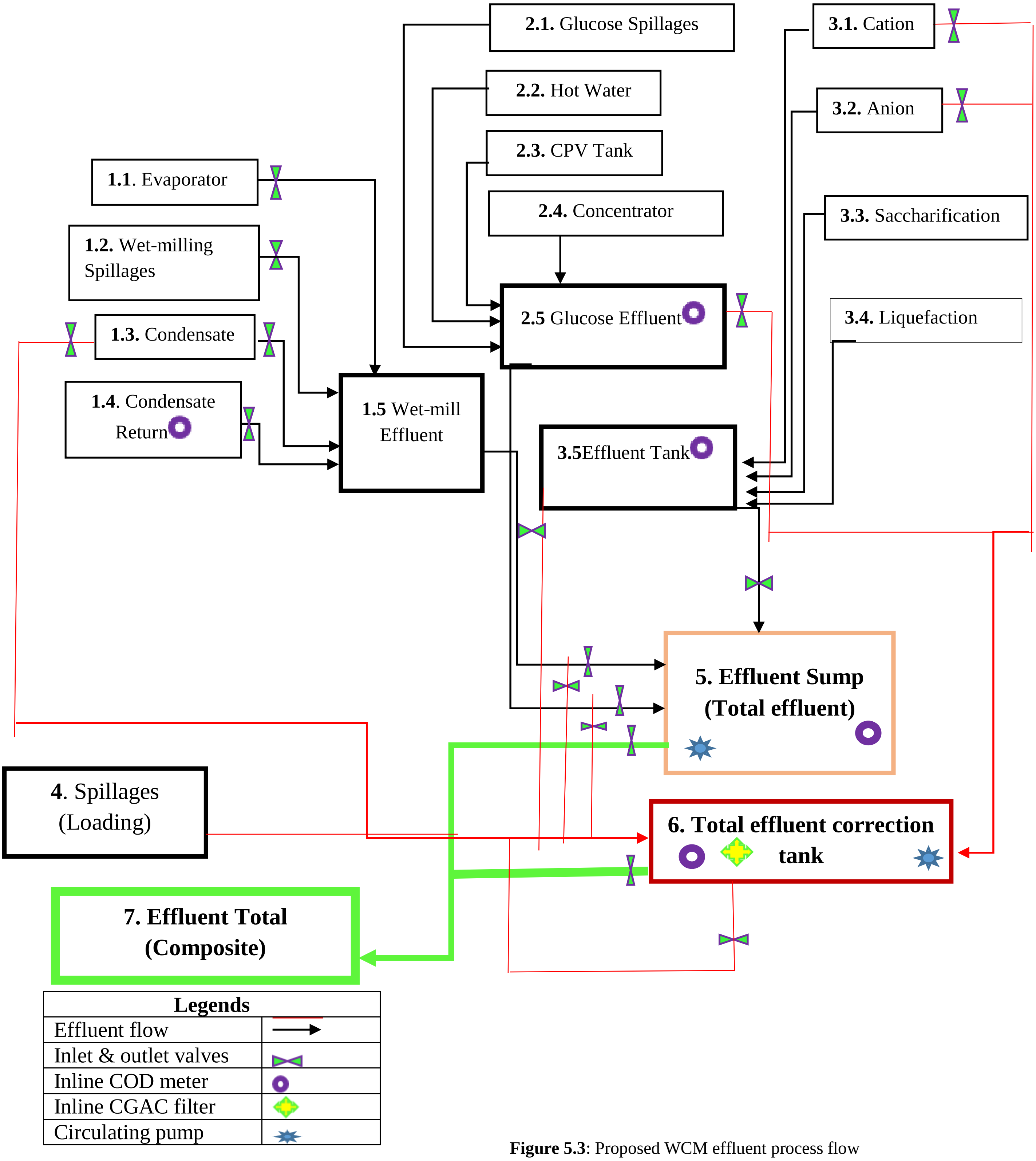

Figure 5.3: Proposed WCM effluent process flow

In alignment with the view of Beauregard, McDermott and Mikulak (2008: 1), acknowledged the FMEA as a tool used to identify and prevent incidents before they occur, FMEA risk assessment reviewed in the redesigned WCM effluent process to evaluate the effectiveness of the recommended actions in reducing or eliminating the pre-identified risks that are mentioned in **Table 5.10** above. The new assessment process flow is designed to validate an answer the last investigative question, which states: “Can a modified procedure be employed to reduce COD concentrations in the effluent generated by the WCM process to comply with the regulatory specification consistently?”.

The FMEA results for the recommended WCM process flow showed a very low risk priority number. This indicates that the recommended action plans will improve and sustain COD concentrations in the effluent discharged by the WCM are always compliant with the requirement. The FMEA risk assessment on the new recommended process flow is presented in **Table 5.11** below; the rating descriptions are documented in **Appendix O1-O3**.

Table 5.11: FMEA risks assessment result of the proposed WCM effluent process flow

Risk - Bad events	**Consequences**	**Severity**	**Occurrence**	**Proposed process control**	**Detection**	**RPN**	**Actions**
Skills gap	Poor control of the interlinked process stages	2	1	Automated inline COD detector in all the effluent macro channels	1	2	Conduct planned maintenance as per established frequency, calibrations and verifications of the detector and maintain the records
Poor control WCM processing stages	High COD concentrations	1	1	Automated inline COD detector in all the effluent macro channels	1	1	Conduct planned maintenance as per established frequency, calibrations and verifications of the detector and maintain the records
Blocked channel flow or pipes	Spillages and leakages that causes effluent contamination (high COD concentrations)	1	1	Automated inline sensor detectors	1	1	Implement a planned maintenance and testing schedule (verification) and maintain the records
Faulty system (valves and detectors)	Effluent contamination/ product spillages	1	1	Systematic automated full system check	1	1	Implement a planned maintenance and testing schedule (verification) and maintain the records
Regeneration of the anion and cation beds	Contamination of the effluent tank and overall WCM effluent	1	1	Automated	1	1	Design a process to pre-treat the effluent that gets generated during the regeneration of the beds

The risks assessment performed in the new proposed process showed very insignificant risks should the detection of risks be improved. These improvements in detection measures will impact positively on both the incident recurrences and on the impact or severity. Therefore, re-designing the risks away can be adopted by improving the technology and moving away from manual detection, and becoming more proactive in detecting a problem before it occurs and preventing it from causing severe impact.

5.7.3 Control Phase summary

The FMEA tool identified the potential failure modes and risks that could result in COD concentrations contamination. The corrective and preventative solutions indicated a significant reduction on the pre-identified failure risk, thus resulting in the establishment of a procedure that could be used to manage the COD concentrations in the WCM effluent to consistently adhere to the standard regulatory requirement.

The National Institute of Health Research (2020) made a statement that validity is the extent to which the method of measure is capable of providing measure(s) that match the true value and enable accurate generalizations about the study. Keeping this statement in mind, the validity of this study is presented in the next section.

5.8 Validity

Validity is the extent to which a study produces accurate results (internal validity) and results that are widely applicable (external validity) (Albery, *et al*., 2014: 25). Ary and Suen (2014: 99) report that internal validity includes dependability, consistency, predictability and stability; while external validity involves the ability of the collected data to reflect the underlying attribute of interest. Internal validity is a prerequisite for validity even though it does not guarantee validity (Jolley & Mitchell, 2010: 143). Internal validity is the measurement of error which entails two meanings, systematic or instrumental errors and unsystematic error, also known as random error of measurement (Hunter & Schmidt, 2004: 252). Relative or absolute internal validity of the measurements involves assessing the precision of the individual measurements within a group (Carter & Lubinsky, 2016: 243). Bias affects external validity and the conclusions that are drawn about the target population (National Institute of Health Research., 2020). Percentage bias and standard relative error measurement analysis are performed to prove the reliability of the procedures and instruments employed during the conduct of this study.

Osborn (2006: 168) supports the view that the two-tailed t-test is a non-directional test that can be used to examine whether there is a significant difference between the sample mean and the true value. Following the analysis of percentage bias (also referred to as %Bias) and relative error, a two-tailed test are used to provide internal validity using reference standard sample measurements and their true values. According to Pham (2006: 262), one-way ANOVA hypothesis testing can be used to measure bias, to support the interval validity of the collected data analysis. Following the two-tailed t-test analysis, analysis using one-way ANOVA to evaluate the validity of the repeatability of study using reference standards is presented. Thereafter, correlation of the internal reference standards analysis is presented, as Badgett and Christmann (2009: 116) reason that correlation is a primary statistical concept used to test the validity of the measurements.

In conclusion to this discussion of validity, external validity considered in this study was presented and discussed following the view of Carter and Lubinsky (2016: 244), who wrote that external validity is the quality associated with how results are applied. Felbinger and Langbein, (2006: 35-36) added that the external validity refers to the generalizability of research results, which relies on the selection of the unit of analysis and selection of larger random samples. Random sampling and use of recognized and approved methods for data collection and analysis are adopted in this study for external validity purposes.

5.8.1 Internal validity

Internal validity is the extent to which the actual experiment tests are the same as the hypothesis claim tests (Albery, *et al*., 2014: 25). In this research, the accuracy of the measurements was assured by conducting measurements using the internal reference standardized solutions. The measurements of the reference standards were carried out to ensure that the instruments were capable of producing reliable data during the conduct of the experimental trials. Moreover, the standard operating procedures (SOPs) documented in **Appendix A** were used to eliminate the random errors.

To ensure the precision and accuracy of the experimental data used in this research, and to ensure that it was free of bias, ANOVA test and linearity test are performed using the data of the reference standards. These tests are carried out to give assurance that the conclusions made from the data used in this research study are true.

5.8.1.1 WCM reference standard data precision and accuracy

Bias of the measures poses very serious threats to the validity of the result in a study (Jolley & Mitchell, 2010: 142). Precision (%RSD) and accuracy (%Bias) are ensured using the reference standard data collected by the WCM laboratory. With regard to the data of the reference standards collected from 06 May 2017 until 30 July 2017 by the WCM laboratory analysts, see **Appendix D1** for the raw data and in **Appendix D2** for the calculations. All the precision results of the reference standards measurements conducted by the WCM laboratory ranged from 0.05996% to 0.1996%. This range illustrates that the method of measurements and the instruments are able to provide reproducible measurements. The bias measurements obtained on the three reference standards had the highest value of 0.08%, which indicated a minimum accuracy of 99.92%. See a summary of the results in **Table 5.12** below.

Table 5.12: Precision and accuracy results obtained from the reference standard data collected by the WCM laboratory from 06 May 2017 until 30 July 2017

Name of the Reference standard	Precision in % (%RSD)	Accuracy in % (%Bias)
2000 ppm	0.1996	0.2
5000 ppm	0.05996	0.06
10 000 pm	0.1099	0.08

5.8.1.2 WCM reference standard linearity

The coefficient of determination for the reference standard measurements obtained using the reference standard data collected by the WCM at the start is found to be 1.0000 (refer to **Appendix D3** for the calculations). This implies that 100% of the total variation in reference standard measurements can be explained by the linear curve.

5.8.1.3 Trial data precision and accuracy

The precision (%RSD) and accuracy (%Bias) trial results are conducted from the data of the reference standards that were measured when the trial measurements were taken. The detailed calculations for trial one and trial two are documented in **Appendix 5A** and **Appendix 5B** respectively.

The precision results of the reference standard measurements conducted for trial one experiments ranged from 0.3984% to 1.4115%, which is less than 5%. The measure of errors obtained on the three reference standards ranged from 0.3% to 1.12%, which indicates that a minimum accuracy of 98.88%.

The precision results of the reference standard measurements of trial two experiments ranged from 0.2197% to 2.2540%, which is less than the analytical tolerance of 5%. The measure of errors obtained on the three reference standards ranged from 0.12% to 2.04%; which indicates minimum accuracy of 97.96%, and achievement of high accuracy of 99.88%. See **Table 5.13** below for the outcomes of the results.

Table 5.13: Precision and accuracy results for the reference standards trial measurements

Trial one – day 1		
Name of the reference standard	**Precision in % (%RSD)**	**Accuracy in % (%Bias)**
2000 ppm	0.3984	0.4
5000 ppm	1.4115	2.03
10 000 ppm	0.9889	1.12
Trial one – day 2		
Name of the reference standard	**Precision in % (%RSD)**	**Accuracy in % (%Bias)**
2000 ppm	0.3988	0.3
5000 ppm	0.8723	0.88
10 000 ppm	0.3779	0.55
Trial two – day 1		
Name of the reference standard	**Precision in % (%RSD)**	**Accuracy in % (%Bias)**
2000 ppm	0.6924	1.1
5000 ppm	2.2540	2.04
10 000 ppm	1.8953	1.83
Trial two – day 2		
Name of the reference standard	**Precision in % (%RSD)**	**Accuracy in % (%Bias)**
2000 ppm	0.4475	0.55
5000 ppm	0.2197	0.12
10 000 ppm	0.4976	0.49

The analysis results of the random and precision measurements indicated in the above **Table 5.3** demonstrate that the bias and precision measures are within the accepted criteria of 95%. This proves that measurements obtained on different days under different conditions are similar. The two tailed t-tests for each reference standard for the COD concentrations are discussed in the next section.

5.8.1.4 Two tailed test for accuracy testing of the reference standards run during the trials

The T-test is a statistical significance test that can be used to examine significant differences between two independent groups of study, such as experimental and control groups (Riazi, 2016: 333).

There is enough significant statistical evidence based on the t-test results that the instruments and procedures used in this research study are capable of providing accurate measurements. This conclusion is supported by the results of the two-tailed test which is demonstrated the acceptance of the null hypothesis for the three reference standards, namely 2000 ppm, 5000 ppm and 10 000 ppm at 95% confidence. All the calculated t-test values fall within the non-rejection criteria, which illustrates that there are no significant differences between the measurements of the reference standard means compared with the true values. The calculations are documented in **Appendix F1** and **Appendix F2**, and a summary of the results is presented in **Figure 5.14** below.

Table 5.14: Two-tailed t-test results from the reference standard collected from the trial data

Trial one – day 1				
Name of the reference standard	**Claim**	**t-statistical**	**Non-rejection region**	**Decision (accept or reject claim)**
2000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	2.0000	-3.1820 to 3.1820	Accept the claim
5000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	2.8056	-3.1820 to 3.1820	Accept the claim
10 000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	2.2400	-3.1820 to 3.1820	Accept the claim
Trial one – day 2				
Name of the reference standard	**Claim**	**t-statistical**	**Non-rejection region**	**Decision (Accept or reject claim)**
2000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.5000	-3.1820 to 3.1820	Accept the claim
5000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	2.0000	-3.1820 to 3.1820	Accept the claim
10 000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.7739	-3.1820 to 3.1820	Accept the Claim
Trial two – day 1				
Name of the reference standard	**Claim**	**t-statistical**	**Non-rejection region**	**Decision (accept or reject claim)**
2000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	3.1429	-3.1820 to 3.1820	Accept the claim
5000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.7739	-3.1820 to 3.1820	Accept the claim
10 000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.8964	-3.1820 to 3.1820	Accept the claim
Trial two – day 2				
Name of the reference standard	**Claim**	**t-statistical**	**Non-rejection region**	**Decision (accept or reject claim)**
2000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	2.4444	-3.1820 to 3.1820	Accept the claim
5000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.0909	-3.1820 to 3.1820	Accept the claim
10 000 ppm	$H_0: \bar{x} = \mu$ $H_1: \bar{x} \neq \mu$	1.9600	-3.1820 to 3.1820	Accept the claim

There is enough statistical evidence that proved the procedures and the instruments used during data collection provided similar results at 95% confidence limits. One-way ANOVA analysis is conducted to prove accuracy of the reference standard results obtained through the conduct of the experiments.

5.8.1.5 One-way ANOVA for the reference standard measurements collected during the trials

One-way ANOVA testing provides analysis results for internal consistency in order to prove stability and reliability of the testing equipment and procedure (Salmons & Wilson, 2009: 368).

At the confidence limit of 95%, there is enough statistical evidence to accept the null hypothesis for all the reference standard, i.e. there is no significant difference in the variation of the reference standard means during the time of data collection, since all the calculated F-statistics values are found to be less than f-upper(Fu). The conclusion is reached that there is no significant difference between means of the reference standard measurements conducted in trial one – day one, trial one – day two, trial two – day one, and trial two – day two. See **Table 5.15** below for the summarized one-way ANOVA results for the reference standards, refer to **Appendix G** for measurements and calculations.

Table 5.15: One-way ANOVA tests result using reference standards data collected during the trials

Name of the reference standard	Hypothesis claim	F-statistical	F-critical	Decision (accept or reject the claim)
2000 ppm	H_0: $X_{Tria11-Day1} = X_{Trial1-Day2} = X_{Trial2-Day1} = X_{Trial2-Day2}$ H_1: $X_{Trisl1-sDay1} \neq X_{Trial1-Day2} \neq X_{Trial2-Day1} \neq X_{Trial2-Day2}$	1.8381	3.4903	Accept the claim
5000 ppm	H_0: $X_{Tria11-Day1} = X_{Trial1-Day2} = X_{Trial2-Day1} = X_{Trial2-Day2}$ H_1: $X_{Trisl1-sDay1} \neq X_{Trial1-Day2} \neq X_{Trial2-Day1} \neq X_{Trial2-Day2}$	1.7064	3.4903	Accept the claim
10 000 ppm	H_0: $X_{Tria11-Day1} = X_{Trial1-Day2} = X_{Trial2-Day1} = X_{Trial2-Day2}$ H_1: $X_{Trisl1-sDay1} \neq X_{Trial1-Day2} \neq X_{Trial2-Day1} \neq X_{Trial2-Day2}$	1.2282	3.4903	Accept the claim

There is enough statistical evidence at 95 confident limits to demonstrate that there is no difference in the reference standard reading obtained when the experiments were conducted. It can thus be concluded that the method and instrument used in this study are capable of providing consistent measurements. The linearity of the reference standard is discussed in the next section.

5.8.1.6 Trial reference standard linearity

A negative coefficient at any level signifies invalid test results while a test with a positive coefficient of +0.9 signifies highly valid results (Badgett and Christmann, 2009: 116).

The correlation coefficient value for the reference standards measurements obtained for trial one at the start is found to be 0.9996. This implies that 99.96% of the total variation in y-intercept can be explained by the linear relationship between the independent and the dependent variables. The remaining 0.04% variation in the dependent variable cannot be explained due to certain other factors. The actual calculations are presented in **Appendices E1** and **E2**; see the results in **Table 5.16** below.

Table 5.16: Linearity results for the reference standards (2000ppm, 5000ppm, and 10 000ppm) data collected during the trials

Name of trial	Coefficient of determination value	Coefficient of determination value in %
Trial one – day 1	0.9996	99.96
Trial one – day 2	0.9999	99.99
Trial two – day 1	0.9989	99.89
Trial two – day 2	0.9999	99.99

There is enough statistical evidence obtained from the results of the random error tests, hypothesis tests, and linearity tests conducted in the reference standard measurements collected during the primary data collection in this study to prove that the data used in this study is valid.

5.8.2 External validity

External validity indicates the extent to which the results of experiment can be applied to other situations (Albery, *et al*., 2014: 25). All the procedures that were used to collect primary data were validated in accordance with ISO 17025 requirements. This assures that the sampling procedures can be repeated and similar results obtained, as the experiments were conducted for a period of two months under different conditions, and as these procedures provide guidance on how to prevent sampling errors and testing errors that could result in misleading data and inaccurate generalization of the data.

The use of the externally calibrated and approved analytical measuring apparatus by SANAS certified bodies also provided assurance on the integrity of the data. This means that when the procedures applied in this study are applied elsewhere, similar outcomes are attained, taking into consideration the applicable confidence of limits.

5.8.3 Validity summary

All the tests conducted to demonstrate internal validity of this study indicate that this study met the applicable internal validity criteria. External validity, which is illustrated by the methods followed during the conduct of this research, also rests on valid protocols that were followed using the reviewed literature. Ethical considerations that are applicable in this study are presented in the next section.

5.9 Chapter 5 conclusion

This chapter presented the data interpretation and analysis of the findings of this research study. It identified the macro effluent channels that contributed to the nonconforming COD concentrations in the WCM, namely Concentrator, Effluent Tank and Glucose. Then, the root causes of the nonconforming effluent with high COD concentrations derived from the three macro channels were identified with the use of the Fishbone and 5 Whys method. The result of the RCAs indicated the skills gap, lack of process control, ineffective manual detection of process deviations and inadequacy of procedures for correcting nonconforming COD concentrations.

The experimental trials were performed to determine a parameter-adjusted procedure that will result in greater reduction of the COD concentrations in the WCM effluent. The results of analysis obtained from the trials using paired t-tests, correlation, and independent sample t-test studies indicated that the increase in the filtration time and CGAC surface area enhances reduction of the COD concentrations. The FMEA risk assessment was then used to identify all the failure risks in the proposed flow to identify preventative measures to eliminate or minimize the risks. This chapter concluded by presenting analyses of data validity in this study and the applicable ethics that were taken into consideration.

The next chapter, Chapter 6 will include a recap of this study before presenting the final research findings, conclusions, and recommendations of this study. The outcomes of this study will also be reviewed to see if the objectives of this research study are met.

CHAPTER 6: CONCLUSION AND RECOMMENDATIONS

6.1 Introduction to Chapter 6

This study is geared towards assisting a WCM plant in the WC Province in SA, to reduce COD content in the effluent discharged by the plant, so that it could consistently conform to the legislated standard specification for COD. The purpose of this research study is to develop a procedure that would be suitable for optimal reduction of the COD concentrations in the effluent generated by the specific WCM. This research study has found that the reduction of the COD concentrations in the WCM effluent can be achieved by applying a suitable scientific method and improving the current systems used by the plant. This involved the alteration of selected variable(s) within the specific effluent and evaluation of the adjustment of parameter in terms of effectiveness on COD removal. Thereafter, it involved improving the current process by incorporating the successfully tested procedure within the WCM effluent process and taking into consideration the failure risks to achieving a sustainable process.

This chapter presents a brief summary of each preceding chapter, then revisits the research problem statement, research aim, primary research question, investigation questions, and the research objectives. Following this, the final research findings are presented and recommendations and research conclusion are provided at the end of this chapter.

6.2 Summary of preceding chapters

A brief explanation of the context lay out of each chapter is explained in the next point below.

6.2.1 Summary of Chapter 1

The introduction and motivational background of this study is presented in Chapter 1 to articulate the background to the researched problem. The research problem statement, aim of the study, primary research question, investigative questions and research objectives to be embarked upon are also presented, accompanied by a brief introduction to the conceptual framework, methodology and research design, data collection and analysis, data validity, ethics, research assumptions and research constraints. Chapter 1 concluded by outlining the chapters which served as a research process followed in this study.

6.2.2 Summary of Chapter 2

In Chapter 2, an introduction to the research environment is presented, which included the importance of water management in SA in the context of this research study. This is followed by discussion on the importance management of effluent in SA and contraventions associated with its ineffective management. The background to WCM plant in which this research is based was explained, including certified international organizational standard systems. Detailed explanation of wet corn processing is provided, and the environmental implications of COD concentrations in the effluent generated by the WCM, as well as the impact of poor COD concentrations management in the effluent. Chapter 2 concluded with the overview of the WCM plant's quality control implemented measures.

6.2.3 Summary of Chapter 3

Existing literature pertaining to the research problem is discussed in detail to provide the reader with a clear understanding of contextual variable(s) which played a role in the research problem. The implications of the research problem and its significance were presented, followed with the discussion of the possible solutions, and most effective solution applicable to the researched problem. The Chapter concluded by discussing relevant tools used to find solutions to the research problem followed by possible solutions.

6.2.4 Summary of Chapter 4

Chapter 4 presented the research plan of this study. It commenced with an introduction to general research methodology, followed by an overview of research worldviews. The reader is then introduced to the research approach selected for this study, namely Six Sigma DMAIC, including data collection methods and data analysis methods. The specific methods that are planned to be used in the Six Sigma DMAIC phases were discussed, which entailed providing details on data analysis and interpretation using the specified quality tools and techniques. Furthermore, plans to ensure internal validity and external validity of this study; followed by a presentation of the considered ethical practices.

6.2.5 Summary of Chapter 5

Chapter 5 presented an analysis of empirical data within the DMAIC framework; in each phase, interpretation and analysis of the results of data were presented in the order outlined in Chapter 4. Data analysis is performed with the use of the appropriate quality tools or techniques, and taking into account the investigative questions that needed to be answered. The quality tools included a process flow, Fishbone diagramss, 5Whys and FMEA. Statistical techniques are employed to make inferences about data of interest, include the t-test hypothesis, paired t-test hypothesis and correlation studies. This Chapter concluded by explaining data validity; where the calculations and explanations are presented to provide assurance regarding the repeatability of this study.

6.2.6 Summary of Chapter 6

Chapter 6 commences with an overview of the preceding chapters before revisiting the research problem statement, research aim, primary research question, investigation questions and the research objectives. This is to review if the all the research questions and objectives are met. The chapter concludes by presenting the final research findings and proposed research recommendations for optimal COD concentrations reduction in the WCM effluent.

6.3 Revisited research questions, objectives and findings

The revisiting of the research questions and objectives are presented to review if they are met and the purpose of this study is accomplished. The research findings and recommendations are presented in accordance with the research objectives to reduce the COD concentrations in the WCM effluent to consistently meet the legislated standard specification.

6.3.1 Research problem statement

The research problem statement of this research study is "The effluent discharged from a WCM does not consistently adhere to the required regulatory specification limit". The research problem did not only impact on the environment but also affected costs in the business. Linked with the revisited research problem statement, the aim of the research study is revisited in the next section.

6.3.2 Aim of the study

The aim of this research study is "to develop and document a processing step which will improve the quality of the effluent (reduce COD concentrations) discharged by the WCM of a specific WCM organisation that will adhere to the legislated discharge standard. In addition, the process introduced must be easily controlled at the operational level and consider environmental concerns". This is aimed at reducing environmental risks to aquatic species and reducing the cost associated with effluent charges, and furthermore eliminating the hidden additional cost of treating the effluent in municipal effluent treatment plants prior to discharge. The primary research question applicable in this study to meet the research aim is provided below.

6.3.3 Primary research question

The primary research question proposed for this study is "Can a quality management procedure be developed for a WCM plant to optimise the removal of COD in the effluent discharged to meet regulatory specifications consistently?" This question was posed to stop and think about the modification of the existing methods to achieve optimal removal of the COD in the effluent to consistently conform to the COD standard regulator, and eliminate the negatively effect. The revisited investigative question is provided on the next point.

6.3.4 Investigative questions

The investigative questions within the ambit of this thesis read as follows:

1. Which stages in a WCM process contribute to increasing concentrations of COD in the discharged effluent?
2. What are the causes of the high COD content present in the discharged effluent of the various process stages?
3. Can a process variable(s) be identified that when adjusted yields the optimal removal of COD from WCM effluent?
4. Can a modified procedure be employed to reduce COD concentrations in effluent generated by the WCM process to comply with the regulatory specification consistently?

6.3.4.1 Findings related to investigative question 1

6.3.4.1.1 Analogies drawn from literature: According to Ndlovu (2013: 69), WCM processes emit high concentrations of the COD in the effluent, due to the nature of this process. Garcia Einschlag (2011: 8) mentions that the high organic components in the WCM effluent gives rise to high COD concentrations. Akpor and Muchie (2011: 2379) comment that increasing levels of the highly contaminated COD concentrations due to urbanization result in high COD concentrations.

6.3.4.1.2 Analogies drawn from data analysis: The data illustrated that high COD concentrations in the WCM is caused by three macro processes, namely Condensate, Glucose, and Effluent Tank. The lack in the control and management of these three macro effluent channels resulted in cross-contamination of the COD in the effluent.

6.3.4.2 Findings related to investigative question 2

6.3.4.2.1 Analogies drawn from literature: Ndlovu (2013: 69) pointed out that the nature of the WCM process results in generation of high COD concentrations in the effluent. Ahsan and Ismail (2019: 2) added that high levels of carbohydrates in the effluent result in high COD concentrations. Brouckaert, *et al*. (2002) also support the view that high levels of soluble proteins in WCM effluent is a major contributor to high COD concentrations.

6.3.4.2.2 Analogies drawn from data analysis: The data illustrated that high COD concentrations in the WCM is caused by ineffective management of the effluent process. Contributors to poor management are highlighted to be a skills gap, inadequate procedures, delayed detection of problems, and preventative maintenance.

6.3.4.3 Findings related to investigative question 3

6.3.4.3.1 Analogies drawn from literature: Moreno-Castilla (2004: 5) states that the composition of the GAC surface area plays the most crucial role with regard to achieving the optimal reduction of COD concentrations. Bhise, *et al*. (2013: 67) note that the composition of the adsorbate surface area helps to improve the adsorption rate if selected appropriately. Wu (2004:7) explains that the size of the pores in the GAC can maximize reduction of the COD concentrations. Nekoo and Shohreh (2013:87) report that availability GAC surface area, the pH content of the filtered solution, and the GAC contact time with the solute can result in greater reduction of the COD concentrations. El-Gawad and EL-Aziz (2018: 228) reason that a decrease in pH of the solute can result in greater reduction of the COD concentrations.

6.3.4.3.2 Analogies drawn from data analysis: The paired t-test and correlation study indicated that an increase in contact time and CGAC filtration weight resulted in optimal reduction of the COD concentrations in the WCM effluent. Even though the reduced COD means indicated that acidic effluent samples had better COD removal, compared to using alkaline samples. The independent two-samples test results indicated that at 95% confidence limits there is no difference in the removal of COD concentrations achieved when acidic effluent samples are used compared to alkaline samples. Thus implying the pH content had no significant effect on removal of the COD concentrations during the conduct of this study.

6.3.4.4 Findings related to investigative question 4

6.3.4.4.1 Analogies drawn from literature: Islam, *et al*. (2020: 2006) comment that the WCM generates a high volume of effluent with nonconforming COD concentrations. Icon Water (2015) notes that additional interventions are required to ensure that the quality of the COD concentrations in the effluent is controlled accordingly. Botes, *et al*. (2004) agree that integrated improved systems are needed to effectively manage the effluent.

6.3.4.4.2 Analogies drawn from data analysis: The results obtained from the FMEA tool highlighted the risks associated with the current WCM effluent process which can affect proper management of the COD concentrations. This helped to identify the solutions needed to consistently manage the COD concentrations in accordance with the legislated requirements. The proposed solution includes establishment of a modified procedure for reducing the COD concentrations to enable the COD concentrations in the WCM effluent to consistently comply with the legislated requirements.

6.3.5 Revisited research objectives

The revisited research objectives applicable in this thesis are:

- Investigate which stages in wet corn processing have a significant influence on the subsequent high COD of the discharged effluent,
- establish the causes of the increasing levels of COD in the effluent generated by a WCM,
- identify variable(s) that can be adjusted to yield the optimal removal of COD from WCM effluent, and
- establish an effective procedure for removal of the COD in the effluent generated by a corn wet milling process.

All the research objectives are met as discussed in the revisited research questions which are linked to each research objective. Therefore, the aim of this research study was fully achieved. The recommendations are discussed below for the WCM to optimize the removal of the COD concentrations to consistently comply with the regulations, and eliminate the consequences thereafter of not doing so.

6.4 Conclusion to objectives and recommendations

6.4.1 Conclusion to objective 1 and recommendations: The conclusion to the first objective is that three macro channels contributed to the nonconforming COD in the WCM effluent. The three nonconforming WCM macro channels are: Condensate, Glucose and Effluent Tank. Recommendations on how to improve the performance of these macro channels are elaborated in the next section.

6.4.2 Conclusion to objective 2 and recommendations: The conclusion to the second objective is that the root causes of high COD concentrations from the effluent generated by the three macro effluent channels included, poor control of process management caused by the skills gap, ineffective monitoring of the COD concentrations, unavailability of appropriate procedures for correcting and preventing nonconforming COD concentrations in the effluent, and lack of preventative maintenance to reduce the failure rate. The recommendation is to provide training and retraining for the process operators, perform procedure reviews to improve performance and review the systems in place to continuously better their performance to benefit the plant.

6.4.3 Conclusion to objective 3 and recommendations: The conclusion to the third objective is that CGAC surface area and filtration time can be adjusted to achieve more COD removal from the WCM effluent. Thus, to achieve optimal COD removal, more exposure of the effluent samples is required. The recommendation is to introduce a process step, where these two variable(s) are controlled accordingly to achieve more reduction of the COD concentrations.

6.4.4 Conclusion to objective 4 and recommendations: The conclusion to the fourth objective of this study is that there is a skill gap on the process operators; which affects the effective manner in which the process is controlled. Another factor, is the time it takes to detect the problem, which directly affects the time to correct the problem and its severity; and the fact that there are no process steps and procedures for correcting nonconforming effluent to comply with the requirements. The proposed recommendations to find solutions for the reported problems are as follows:

- The WCM plant is recommended to conduct an effective effluent process management workshop with the employees who are directly involved with the effluent process. The purpose of this workshop will be firstly to educate the process operators about the importance of generating conforming effluent with COD concentrations. Secondly, the process operators must be educated in the consequences of generating and discharging effluent with non-conforming COD concentrations for the environment and for their business. Thirdly, they must be involved in the process of identifying the risks (risk assessment) that causes COD concentrations in the effluent to deviate from the manufacturing standard specification. Lastly, the process operators must form part of the team, when creating procedures or reviewing procedures, to ensure that adequate procedures are developed and understood at all levels, and are implemented accordingly. Moreover, they should undergo structured training to prevent the same failures from recurring. They must be certified as competent to independently conduct an effective RCA, using a structured approach designed by the plant management.

- It is recommended that on-line or in-line COD detectors which are able to detect when COD concentrations deviate from the predetermined specifications must be installed, to improve the performance of the current system. The current system is not designed to quickly detect the process when it starts to deviate, but takes a long time to detect deviation, after the defect has spread and contaminated the total effluent. As the current measuring method takes about an hour or more to provide results of the COD generated by each channel. Significantly, the COD concentrations can deviate from the legislated limits within a minute. Therefore, if the detection method is not quick enough, it will be difficult for the plant to control the process in accordance with the required specifications. These on-line detectors should be installed in the main macro channel tanks and be programsmed to plot a moving range chart, using data points measured every 10 minutes. The integrated process control software will to help the process operator to predict process deviation before it goes out of the defined specification limits. The detectors must be programsmed to raise an alarm when the COD concentrations exceed a certain limit. A procedure should be established to stipulate actions that must be taken when the process has deviated from the norm. These interventions will react to and correct the process before it exceeds the standard specification. A system such as real time analysis of live COD concentrations data will help the plant isolate the main source of contamination until the problem is solved.

- The nature of the products that are manufactured by the WCM consist of polysaccharides; which gives rise to COD levels in the effluent once the product is mixed with the effluent (Ndlovu, 2013: 69-70). The Effluent Tank RCA analysis in Chapter 5 indicated that COD concentrations are also prompted during the regeneration of anions and cations, which is naturally part of the WCM process. Therefore, the WCM must establish a proper procedure of treating nonconforming COD concentrations, an additional effluent treatment step is needed. This step will consist of CGAC; which will enable the WCM to treat all the nonconforming COD to consistently meet standard specification requirements. As the literature reviewed in section 3.3.1 in Chapter 3 and the findings presented in section 5.5 in Chapter 5 of this study illustrated that the CGAC adsorption method can reduce the COD concentrations in the WCM effluent. Therefore, CGAC adsorption method can be used to correct the nonconforming COD in WCM effluent when contaminated.

- Block valves that causes product overflows and effluent contamination can be mitigated by generating a plant start-up and plant shut-down procedures. The start-up procedure should be designed to cater the start-up inspections to ensure that there is free flow of the product through the process valves, thus, prevent product spillages or overflow on start-ups. The shut-down procedure should be designed to address product blockages; which could be accomplished through proper emptying the products out of the process channels. This will eliminate the products overflows that are cause by the blockages of the valves. The product flow check points should be identified and clearly labelled, and the line inspectors should be trained to be competent to perform the inspections accordingly.

6.5 Recommendations for future research

The final recommendation of this research for future research is to investigate the most cost-effective and environmentally effective way of managing or recycling the waste generated by the CGAC adsorption process. Further investigation could include the lifespan of CGAC when applied in effluent generated by a WCM, and finding a cost-effective and environmentally friendly procedure for reactivating or regenerating exhausted CGAC beds.

6.6 Conclusion

This research is carried out in a WCM plant located in the WC. The purpose this research is to establish a suitable procedure that can be employed to optimally reduce the COD concentrations in the WCM effluent, to consistently comply with the legislated standard requirement of 5000 ppm. The reviewed literature guided the data collection, interpretation and its analysis; which resulted in the conclusions and recommendations of this study. All the research questions are answered and research objectives are achieved.

The aim of this research study is to establish a procedure that will help the WCM plant to optimally remove COD concentrations from the effluent, and a procedure for achieving the most effective COD reduction in the effluent is established.

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8 APPENDICES

Appendix A: RESEARCH STANDARD OPERATING PROCEDURE FOR COD ANALYSIS

Cape Peninsula University of Technology	**Faculty of Engineering: Department Quality**
	Method for analysis of COD in the effluent

1. Introduction

This method was used for the analysis of the COD concentrations in the WCM effluent samples, which were collected from the WCM that is located in the WC.

2. Personnel

The person performing the procedure deemed competent through the test and the plan job observation.

3. Limitation, precision (random and systematic errors) of the method

a. The method is applied effluent samples collected from the WCM plant in WC.

b. The COD concentrations range that can be measured is 2000 ppm – 10 000 ppm.

4. Equipment, Apparatus and Materials

a. 1ml pipette
b. Coconut GAC
c. 120 degrees' heat reactor
d. Beakers (500 ml),
e. 250 ml Erlenmeyer or conical flask
f. Spatula,
g. Analytical balance,
h. Filter paper (90 mm).
i. Stopwatch,
j. COD meter (Benchtop Photometer)
k. High Range (HR) COD vial reagents that range from 0 to 15 000 ppm/mg/L.

Appendix A: RESEARCH STANDARD OPERATING PROCEDURE FOR COD ANALYSIS

Cape Peninsula University of Technology	**Faculty of Engineering: Department Quality**
	Method for analysis of COD in the effluent

5. **Reagents (Good Quality Analytical Graded Reagents)**

i. H93754-25 COD High Range Reagent Dichromate Vials – Follows the dichromate method for the high range determination of chemical oxygen demand using a compatible benchtop photometer.
ii. Coconut Shell Granular Activated Carbon – A good adsorption performance, high strength, easy regeneration, economy and durability.

6. **Laboratory Reference Standards**

This product is prepared on a weight/volume basis, holds IEC/ISO 17025 accreditation for laboratory balances, and pipettes (Ref-265C). Product specifications, the accuracy of ±0.5%, a liquid form, Packaging - HDPE Twin Neck bottle, matrix –water, physical form - liquid form.

i. 2000 ppm – Total Organic Carbon Standard suitable for calibrating a wide range of TOC analysers to high accuracy.
ii. 5000 ppm - Total Organic Carbon Standard suitable for calibrating a wide range of TOC analysers to high accuracy.
iii. 10 000 ppm– Total Organic Carbon Standard suitable for calibrating a wide range of TOC analysers to high accuracy.

7. **Safety Precautions**

i. A bump cap, safety shoes, goggles, gloves and acid resistant overalls must be worn when collecting the effluent samples.
ii. A Laboratory coat, safety goggles, safety shoes and gloves must be worn when applying the procedures.
iii. Pre-reading of the Material Safety Data Sheets is mandatory before using any of the chemicals.
iv. Adherence to the disposal of the chemical waste specified in the Material Safety Data Sheets is mandatory.

Appendix A: RESEARCH STANDARD OPERATING PROCEDURE FOR COD ANALYSIS

Cape Peninsula University of Technology	**Faculty of Engineering: Department Quality**
	Method for analysis of COD in the effluent

8. <u>Sample and Standard Test Procedure</u>

a. Mix the sample and transfer 0.2 ml into the HR reagent vial using a 1 ml pipette.

b. Keep the vial at an angle of 45 degrees relative to the pipette while transferring the sample.

c. Invert the reagent vial containing the sample 20 times to mix the solution thoroughly.

d. Transfer another 0.2 ml of deionised water into a new HR reagent vial using 1ml pipette.

e. Keep the angle of the vial at an angle of 45 degrees relative to the pipette, while transferring deionised water.

f. Invert the vial containing the deionised water 20 times to mix the solutions thoroughly.

g. Insert the two vials into a 150 degrees Celsius pre-heated reactor for 2 hours.

h. Switch of the heat reactor at the end of the tow hours (digestion period), and after 10 minutes, invert the vials 20 times.

i. Place the HR reagent vials in a steel rack to allow them to cool down at room temperature for 30 minutes.

j. After 30minutes, the samples were analysed using a Benchtop Photometer, NB: a careful handling of the samples is need to prevent forming bubbles and humidity from the samples (NB – Do not shake the samples).

k. Switch on the Benchtop Photometer and use the vial with the blank solution (Deionised water) to zero the instrument.

l. After zeroing the instrument, measure the sample and record the measurement. NB: No shaking of the sample after cooling and when taking the measurements.

m. Each measurement was performed in duplicate.

Appendix A: RESEARCH STANDARD OPERATING PROCEDURE FOR COD ANALYSIS

Cape Peninsula University of Technology	**Faculty of Engineering: Department Quality**
	Method for analysis of COD in the effluent

9. <u>Effluent Sampling Procedure</u>

a. Label the sampling container with the sample name to be sampled.

b. Open the sampling tank for at least 10 seconds, before collecting a sample.

c. Rinse the sampling container at least three times with the sample to be collected before collecting a sample.

d. Then collect the sample and close the sampling container led.

10. <u>Effluent Sample Preparation Procedure</u>

a. Weigh CGAC into 200 ml beaker using a four digital analytical balance.

b. Measure 100ml of the effluent sample using a 100 ml measuring cylinder.

c. Transfer the 100ml volume of the effluent sample into the beaker that contain CGAC, and start the timer/clock.

d. Take 250 ml conical flask and insert a funnel with the filter paper.

e. When the timer goes off, filter the effluent in the beaker.

f. Use the filtered samples for conducting the COD measurements.

11. The trial batches that were used in this study are illustrated in the table below:

Trial 1				
Batch Number	**CGAC Weight in gramss**	**Contact Time in Minutes**	**Sample Name**	**pH Content**
1	20	20	Condensate, Effluent tank, & Glucose	Acidic
		30		Acidic
		60		Acidic
2	30	20	Condensate, Effluent tank, & Glucose	Acidic
		30		Acidic
		60		Acidic
3	40	20	Condensate, Effluent tank, & Glucose	Acidic
		30		Acidic
		60		Acidic
Trial 2				
Batch Number	**CGAC Weight in gramss**	**Contact Time in Minutes**	**Sample Name**	**pH Content**
1	20	20	Condensate, Effluent tank, & Glucose	Alkaline
		30		Alkaline
		60		Alkaline
2	30	20	Condensate, Effluent tank, & Glucose	Alkaline
		30		Alkaline
		60		Alkaline
3	40	20	Condensate, Effluent tank, & Glucose	Alkaline
		30		Alkaline
		60		Alkaline

Appendix B: DATA ANALYSIS METHOD, DESCRIPTION AND CALCULATION FORMULAS

Name of the method	**Description**	**Calculation Formula**
Bias or random error	Used to calculate the difference between the measured and actual value to measure the accuracy of the measurements.	$\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ Mean formula: $\bar{x} = \frac{\sum_{i=1}^{n} \bar{x}}{n}$ Where: μ is the true value $\bar{x}$ is the mean
Precision	Used to calculate closeness of the readings.	$\% \, RSD = \left[\frac{Std}{\bar{x}}\right] 100$ **Where:** Std is the standard deviation. $\bar{X}$ is the mean. $Std = \sqrt{\frac{\sum(x - xbar)2}{n}}$ $\bar{x} = \frac{\sum_{i=1}^{n} \bar{x}}{n}$
Regression: Slope of the variable(s)	Used to determine internal calibration uncertainty in	$\beta_0 = Y_{bar} - \beta_1 X_{bar}$ β_I is the slope and β_0 is the Y-intercept. Where β_I: $b_1 = \frac{SSXY}{SSX}$ Where SSXY is the cumulative sum of the X multiplied by the cumulative sum of variable y, divided by the number of observations and then subtracted from the accumulative product of X and Y. Where SSXY is calculated using: $\sum_{i-1}^{n} X_i Y_i - \frac{(\sum_{i-1}^{n} Xi)(\sum_{i-1}^{n} Yi)}{n}$ Where SSX is the squared value of the cumulative sum of variable x, divided by the number of observations and then subtracted from the accumulative sum of all the squared values of variable x. $= \sum_{i=1}^{n} X_i^2 - \frac{(\sum_{i=1}^{n} X_i)^2}{n}$
Coefficient of determination	To measures the proportion of variation that is explained by the independent variable X in the regression model. The value of r^2 can range between 0 and 1, and the higher its value the more accurate the regression model is. A large r^2 value indicates a strong linear relationship between the two variable(s). A perfect correlation of ± 1 occurs only when the data points all lie exactly on a straight line. If $r = +1$, the slope of this line is positive. If $r = -1$, the slope of this line is negative.	$r^2 = \frac{\text{Regression sum of squares}}{\text{Total sum of squares}} = \frac{SSR}{SST} \text{ or } = \frac{SSR}{SSR + SSE}$ Where r is: $r = \sqrt{\frac{SSR}{SST}}$ where r takes the sign of b

Name of the method	Description	Calculation Formula
One Tail T-test	A sample statistic is used to make inferences about the entire population. It begins with the hypothetical claim: null hypothesis (H_0) and the opposite of the null hypothesis, called alternate (H_1). And make use of the t-statistical and t-critical to make decisions on the claim. Decision making: When a calculated t-value is found to be less than the critical value (t_{upper}), the null hypothesis is accepted and the alternate is rejected. However, if the calculated value is found to be greater than the critical value, the null hypothesis is rejected and the alternate hypothesis is accepted. Confidence limits at 95% for one direction (α-0.05), at n-1 degrees of freedom.	$t = \frac{\bar{x} - \mu}{s / \sqrt{n}}$ Where: t is t-statistic calculated), x is the sample mean (calculated in excel), n is the number of samples, s is the sample standard deviation (calculated in excel). μ is the true value NB: for one direction one ail t-test, the null hypothesis is accepted when the calculated t-statistics is less than the t-critical (t-statistic< t-critical).
Paired t-test	A statistical test used to calculate if there is a difference between two groups of measurements or set of items. It begins with the hypothetical claim: null hypothesis, symbol H_D and the opposite of the null hypothesis, called alternate, H_D. Where D represents the difference between two means. It makes the use of the t-statistical and t-critical values to make decisions. If the calculated statistical value falls within the non-rejection criteria obtained from the table at specified confidence limits and degrees of freedom, the null hypothesis is accepted. However, if the statistical value falls outside of the non-rejected area, the alternate hypothesis is accepted instead of the null hypothesis. Confidence limits at 95% for two tail. Confidence limits at 95% for one directions (α/2-0.025), at n-1 degrees of freedom.	$t = \frac{\overline{D} - \mu_D}{s_D / \sqrt{n}}$ Where: $\overline{D} = \frac{\sum_{i=1}^{n} D_i}{n}$ And $s_D = \sqrt{\frac{\sum_{i=1}^{n} (D_i - \overline{D})^2}{n-1}}$ Hypothesis: $H_o : \mu_D = 0 \, (\text{where} \, \mu_D = \mu_1 - \mu_2)$ Alternate $H_1 : \mu_D \neq 0$ Decision rule: Reject H_0 if t-statistical fall outside of a non-rejection criterion. Reject H_1 if t-statistical falls within the non-rejection criterion.

<table>
<tr><th>Name of the method</th><th>Description</th><th>Calculation Formula</th></tr>
<tr>
<td>One-way ANOVA</td>
<td>Analysing if there is are variation among and within groups, to statistically determine if there are differences in the group means

Decision rule is to reject H_0 if Statistics is greater than F-critical (F>Fu), otherwise accept H_0. If the null hypothesis is rejected, the conclusion that there is a significant difference in the mean values of the groups considered.

Confidence limits at 95%</td>
<td>SST is the total variation.
$$SST = \sum_{j=1}^{c} \sum_{i=1}^{n_j} (X_{ij} - \overline{\overline{X}})^2$$
Where:
c is the number of groups,
X_{ij} is the value in the group j,
n is the total number of values in all groups
nj is the number of values in group j,

SSA is among-group variation
$$SSA = \sum_{j=1}^{c} n_j (\overline{X}_j - \overline{\overline{X}})^2$$
where:
c = number of groups
n_j = number of values in group j
$\overline{\overline{X}}$ = grand mean
$\overline{X}_j$ = mean of sample group j

Where SSW is the variation within-group
$$SSW = \sum_{j=1}^{c} \sum_{i=1}^{n_j} (X_{ij} - \overline{X}_j)^2$$
Where:
X_{ij} = the ith value in the group j
$\overline{X}_j$ = the sample mean in the group j
<table>
<tr><th>Source</th><th>Degrees Of Freedom</th><th>Sums of Squares</th><th>Mean Square (Variance)</th></tr>
<tr><td>Among groups</td><td>$c-1$</td><td>SSA</td><td>$MSA = \frac{SSA}{C-1}$</td></tr>
<tr><td>Within groups</td><td>$n-c$</td><td>SSW</td><td>$MSW = \frac{SSW}{N-C}$</td></tr>
<tr><td>Total</td><td>$n-1$</td><td>SST</td><td></td></tr>
</table>

F statistics:
$$F = \frac{MSA}{MSW}$$
To obtain the $F_{crit\alpha}$ statistic value, the degrees of freedom; which follows c-1 in numerator and n-c in denominator are used at α of 0.05 or 95% confidence of limits.</td>
</tr>
</table>

Name of the method	Description	Calculation Formula
Pooled-variance t-test	This hypothesis testing allows to compare the means of two independent samples. This test calculates the F test statistic is calculated by dividing the variance of a larger sample (numerator) divide by that of the smaller sample variance (denominator). The null hypothesis states that the sample means are equal and the alternate hypothesis states that the sample means are not equal; H_0: $\mu_1 = \mu_2$ H_1: $\mu_1 \neq \mu_2$ The critical values that are obtained on the F table are obtained by using two degrees of freedom of the two samples Decision rule: Accept the null hypothesis if F-statistics is less than the F-upper critical value and greater than F lower, F < Fu and F > F_L. If the F statistics falls in a rejection region, when F > Fu or F < F_L, the null hypothesis is rejected and the alternate is accepted. If the F-test concluded that the variances observed from the two samples; then a different test, called separate variance t-test or t prime test is conducted.	F test: $t = \frac{(\overline{X}_1 - \overline{X}_2) - (\mu_1 - \mu_2)}{\sqrt{s_p^2(\frac{1}{n_1} + \frac{1}{n_2})}}$ S^2p: $s_p^2 = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{(n_1 - 1) + (n_2 - 1)}$ $df = n_1 + n_2 - 2$ Critical values: $\frac{df\,(n-1)}{df\,(n-1)}$ $\frac{\text{explanation: (of\_sample\_1\_in\_the\_numerator)}}{\text{(of\_sample\_2\_in\_the\_denominator)}}$ When the samples are equal, $n_1 = n_2$; then use: $s_p^2 = \frac{s_1^2 + s_2^2}{2}$ When the samples are not equal, $n_1 \neq n_2$; then use: $t' = \frac{(\bar{x}_1 - \bar{x}_2) - (\mu_1 - \mu_2)}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}}$ $df = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\frac{(s_1^2/n_1)^2}{n_1 - 1} + \frac{(s_2^2/n_2)^2}{n_2 - 1}}$

Appendix C1: T-TABLE- USED FOR T-TESTS (DOUGHERTY, 2002)

t Distribution: Critical Values of *t*

Degrees of freedom	Two-tailed test: / One-tailed test:	Significance level 10% / 5%	5% / 2.5%	2% / 1%	1% / 0.5%	0.2% / 0.1%	0.1% / 0.05%
1		6.314	12.706	31.821	63.657	318.309	636.619
2		2.920	4.303	6.965	9.925	22.327	31.599
3		2.353	3.182	4.541	5.841	10.215	12.924
4		2.132	2.776	3.747	4.604	7.173	8.610
5		2.015	2.571	3.365	4.032	5.893	6.869
6		1.943	2.447	3.143	3.707	5.208	5.959
7		1.894	2.365	2.998	3.499	4.785	5.408
8		1.860	2.306	2.896	3.355	4.501	5.041
9		1.833	2.262	2.821	3.250	4.297	4.781
10		1.812	2.228	2.764	3.169	4.144	4.587
11		1.796	2.201	2.718	3.106	4.025	4.437
12		1.782	2.179	2.681	3.055	3.930	4.318
13		1.771	2.160	2.650	3.012	3.852	4.221
14		1.761	2.145	2.624	2.977	3.787	4.140
15		1.753	2.131	2.602	2.947	3.733	4.073
16		1.746	2.120	2.583	2.921	3.686	4.015
17		1.740	2.110	2.567	2.898	3.646	3.965
18		1.734	2.101	2.552	2.878	3.610	3.922
19		1.729	2.093	2.539	2.861	3.579	3.883
20		1.725	2.086	2.528	2.845	3.552	3.850
21		1.721	2.080	2.518	2.831	3.527	3.819
22		1.717	2.074	2.508	2.819	3.505	3.792
23		1.714	2.069	2.500	2.807	3.485	3.768
24		1.711	2.064	2.492	2.797	3.467	3.745
25		1.708	2.060	2.485	2.787	3.450	3.725
26		1.706	2.056	2.479	2.779	3.435	3.707
27		1.703	2.052	2.473	2.771	3.421	3.690
28		1.701	2.048	2.467	2.763	3.408	3.674
29		1.699	2.045	2.462	2.756	3.396	3.659
30		1.697	2.042	2.457	2.750	3.385	3.646
32		1.694	2.037	2.449	2.738	3.365	3.622
34		1.691	2.032	2.441	2.728	3.348	3.601
36		1.688	2.028	2.434	2.719	3.333	3.582
38		1.686	2.024	2.429	2.712	3.319	3.566
40		1.684	2.021	2.423	2.704	3.307	3.551
42		1.682	2.018	2.418	2.698	3.296	3.538
44		1.680	2.015	2.414	2.692	3.286	3.526
46		1.679	2.013	2.410	2.687	3.277	3.515
48		1.677	2.011	2.407	2.682	3.269	3.505

Appendix C2: F-TABLE – USED FOR F-TESTS (HEAGERTY, 2004)

Table 3: Critical values (percentiles) for the F distribution. Upper one-sided 0.025 significance levels; two-sided 0.05 significance levels; 97.5 percent percentiles.

	Numerator degrees of freedom																		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
1	647.8	799.5	864.2	899.6	921.8	937.1	948.2	956.7	963.3	968.6	976.7	984.9	993.1	997.2	1001	1006	1010	1014	1018
2	38.51	39.00	39.17	39.25	39.30	39.33	39.36	39.37	39.39	39.40	39.41	39.43	39.45	39.46	39.47	39.48	39.49	39.50	
3	17.44	16.04	15.44	15.10	14.88	14.73	14.62	14.54	14.47	14.42	14.34	14.25	14.17	14.12	14.08	14.04	13.99	13.95	13.90
4	12.22	10.65	9.98	9.60	9.36	9.20	9.07	8.98	8.90	8.84	8.75	8.66	8.56	8.51	8.46	8.41	8.36	8.31	8.26
5	10.01	8.43	7.76	7.39	7.15	6.98	6.85	6.76	6.68	6.62	6.52	6.43	6.33	6.28	6.23	6.18	6.12	6.07	6.02
6	8.81	7.26	6.60	6.23	5.99	5.82	5.70	5.60	5.52	5.46	5.37	5.27	5.17	5.12	5.07	5.01	4.96	4.90	4.85
7	8.07	6.54	5.89	5.52	5.29	5.12	4.99	4.90	4.82	4.76	4.67	4.57	4.47	4.42	4.36	4.31	4.25	4.20	4.14
8	7.57	6.06	5.42	5.05	4.82	4.65	4.53	4.43	4.36	4.30	4.20	4.10	4.00	3.95	3.89	3.84	3.78	3.73	3.67
9	7.21	5.71	5.08	4.72	4.48	4.32	4.20	4.10	4.03	3.96	3.87	3.77	3.67	3.61	3.56	3.51	3.45	3.39	3.33
10	6.94	5.46	4.83	4.47	4.24	4.07	3.95	3.85	3.78	3.72	3.62	3.52	3.42	3.37	3.31	3.26	3.20	3.14	3.08
11	6.72	5.26	4.63	4.28	4.04	3.88	3.76	3.66	3.59	3.53	3.43	3.33	3.23	3.17	3.12	3.06	3.00	2.94	2.88
12	6.55	5.10	4.47	4.12	3.89	3.73	3.61	3.51	3.44	3.37	3.28	3.18	3.07	3.02	2.96	2.91	2.85	2.79	2.72
13	6.41	4.97	4.35	4.00	3.77	3.60	3.48	3.39	3.31	3.25	3.15	3.05	2.95	2.89	2.84	2.78	2.72	2.66	2.60
14	6.30	4.86	4.24	3.89	3.66	3.50	3.38	3.29	3.21	3.15	3.05	2.95	2.84	2.79	2.73	2.67	2.61	2.55	2.49
15	6.20	4.77	4.15	3.80	3.58	3.41	3.29	3.20	3.12	3.06	2.96	2.86	2.76	2.70	2.64	2.59	2.52	2.46	2.40
16	6.12	4.69	4.08	3.73	3.50	3.34	3.22	3.12	3.05	2.99	2.89	2.79	2.68	2.63	2.57	2.51	2.45	2.38	2.32
17	6.04	4.62	4.01	3.66	3.44	3.28	3.16	3.06	2.98	2.92	2.82	2.72	2.62	2.56	2.50	2.44	2.38	2.32	2.25
18	5.98	4.56	3.95	3.61	3.38	3.22	3.10	3.01	2.93	2.87	2.77	2.67	2.56	2.50	2.44	2.38	2.32	2.26	2.19
19	5.92	4.51	3.90	3.56	3.33	3.17	3.05	2.96	2.88	2.82	2.72	2.62	2.51	2.45	2.39	2.33	2.27	2.20	2.13

Appendix D1: REFERENCE STANDARD MEASUREMENTS CONDUCTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

The data was collected by the laboratory analyst using the procedure that is outlined in **Appendix A** above. A total number of twelve samples for twenty-four hours, at even hours, were measured from 06 May 2017 to 30 July 2017. The means and the standard deviation were calculated for a total period of 60 days, and the results are presented in Appendix below. This data was used to calculate the t-statistical value in the t-test that was conducted using raw effluent COD data obtained from the eleven macro effluent channels. The validity of this data was assured by running the daily reference standards from 6 May 2017 to 30 July 2017.

Number of days	2000 IRS in ppm	5000 IRS in ppm	10000 IRS in ppm	Number of days	2000 IRS in ppm	5000 IRS in ppm	10000 IRS in ppm
1	2009	5007	10005	31	2005	5003	10003
2	2007	5003	10001	32	2002	5004	10000
3	2002	5009	10019	33	2002	5001	10010
4	2005	5000	10002	34	2001	5000	10000
5	2010	5002	10009	35	2000	5002	10005
6	2003	5002	10030	36	2003	5003	10003
7	2003	5009	10018	37	2004	5005	10004
8	2007	5007	10013	38	2005	5007	10007
9	2002	5002	10005	39	2002	5002	10005
10	2001	5000	10001	40	2005	4999	10001
11	1998	5005	10008	41	2000	5003	10006
12	1999	5007	10005	42	1998	4996	10004
13	2001	5010	10007	43	2001	5000	10003
14	2005	5013	10013	44	2002	5003	10003
15	2007	5008	10001	45	2005	5006	10005
16	2002	5006	10020	46	2000	5006	10010
17	2022	5003	10000	47	2012	5001	10000
18	2001	5002	10010	48	2003	5002	10005
19	2008	5005	10080	49	2008	5002	10003
20	2003	5001	10020	50	2005	5001	10005
21	2005	5002	10013	51	2005	5002	10003
22	2013	5009	10008	52	2009	5004	10005
23	2007	5006	10006	53	2007	5006	10002
24	2005	5008	10001	54	2003	5005	10003
25	2001	5002	10005	55	2002	5002	10005
26	2000	4998	10012	56	2002	4998	10010
27	2002	5001	10009	57	2004	5003	10006
28	2003	5000	10013	58	2003	5002	10003
29	2011	5001	10005	59	2001	5005	10005
30	2004	5001	10002	60	2004	5001	10003

	2000 ppm IRS	5000 ppm IRS	10 000 ppm IRS
Mean	2004	5003	10008
Std	4	3	11

Appendix D2: WCM REFERENCE STANDARD CALCULATIONS FOR %BIAS AND PRECISION

Precision calculations for 2000 ppm reference standard measurements:

Std = 4

$\bar{x}$ is 2004 ppm

$$\% RSD = \left[\frac{Std}{\bar{x}}\right]100$$

$$\% RSD = \left[\frac{4}{2004}\right]100$$

%RSD = 0.1996%

The state of the COD concentrations analysis when the COD measurements were conducted by the WCM laboratory on 2000 ppm reference standard was 0.1996 % imprecise.

% Bias (Random Error) calculations for 2000 ppm reference standard measurements

Where:

$\bar{x}$ is 2004 ppm

μ is 2000 ppm

$$\% Bias = \left[\frac{\bar{x} - \mu}{\mu}\right]100$$

$$\% Bias = \left[\frac{2004 - 2000}{2000}\right]100$$

$$\% Bias = \left[\frac{4}{2000}\right]100$$

% Bias = 0.2%

The state of COD concentrations analysis for 2000 ppm reference standard when the analysis of the COD concentrations conducted by the WCM laboratory showed a deviation of 0.2% from the true value of this reference standard.

Precision calculations for 5000ppm reference standard measurements:

Std = 3

$\bar{x}$ is 5003 ppm

$$\% RSD = \left[\frac{Std}{\bar{x}}\right]100$$

$$\% RSD = \left[\frac{3}{5003}\right]100$$

%RSD = 0.05996%

The state of the COD concentrations analysis when the COD measurements were conducted by the WCM laboratory at 5000 ppm reference standard was 0.06 % imprecise.

Random error calculations for 5000 ppm reference standard measurements:

Where:

$\bar{x}$ is 5003 ppm

μ is 5000 ppm

$$\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$$

$$\% \, Bias = \left[\frac{5003 - 5000}{5000}\right] 100$$

$$\% \, Bias = \left[\frac{3}{5000}\right] 100$$

% Bias = 0.06%

The state of COD concentrations analysis for 5000 ppm reference standard when the analysis of the COD concentrations conducted by the WCM laboratory had a deviation of 0.06% from the true value of this reference standard.

Precision calculations for 10 000 ppm reference standard measurements:

Std = 11

$\bar{x}$ is 10 008 ppm

$$\% \, RSD = \left[\frac{Std}{\bar{x}}\right] 100$$

$$\% \, RSD = \left[\frac{11}{10008}\right] 100$$

%RSD = 0.1099%

The state of the COD concentrations analysis when the COD measurements were conducted by the WCM laboratory on 10 000 ppm reference standard was 0.1099 % imprecise.

Random error calculations for 10 000 ppm reference standard measurements:

Where:

$\bar{x}$ is 10 008 ppm

μ is 10 000 ppm

$$\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$$

$$\% \, Bias = \left[\frac{10008 - 10000}{10000}\right] 100$$

$$\% \, Bias = \left[\frac{8}{10000}\right] 100$$

% Bias = 0.08%

The state of COD concentrations analysis for 10 000 ppm reference standard when the analysis of the COD concentrations conducted by the WCM laboratory had a deviation of 0.08% from the true value of this reference standard.

APPENDIX D3: WCM LABORATORY REFERENCE STANDARDS CALCULATIONS FOR LINEARITY

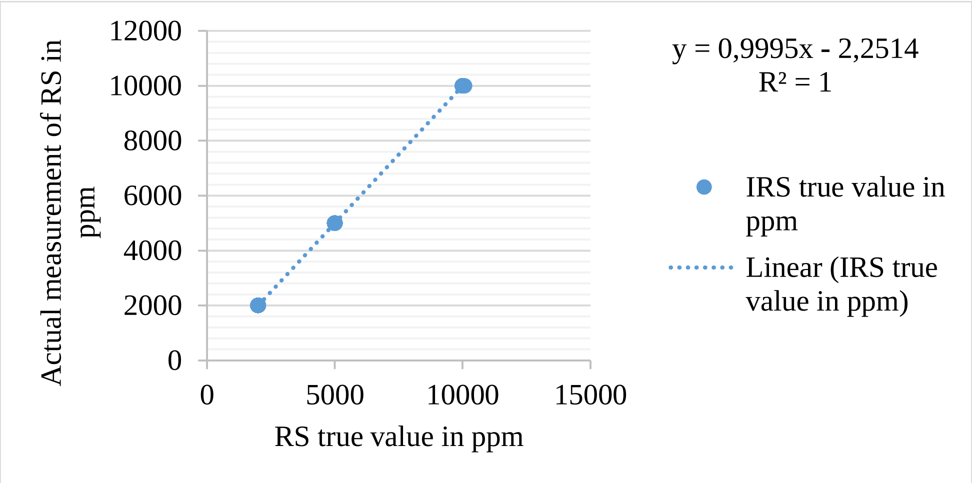
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,99999769
R Square	0,99999537
Adjusted R Square	0,99999535
Standard Error	7,14225874
Observations	180

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1961959823	1961959823	38460856,52	0
Residual	178	9080,111054	51,01185986		
Total	179	1961968903			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2,2787415	1,057893067	2,15403765	0,032581895	0,19111554	4,36636746	0,19111554	4,36636746
Reference Standard true value in ppm	1,00049983	0,000161327	6201,68175	0	1,00018147	1,00081819	1,00018147	1,00081819



r^2 of 1.0000 and r of 1.0000 indicates good positive correlation or linearity.

F-ANOVA = 38460856.52 – the large number implies significant correlation.

Appendix E1: %BIAS AND PRECISION CALCULATIONS ON THE REFERENCE STANDARDS DATA COLLECTED DURING THE EXPERIMENTAL TRIAL ONE

Trial one – day one (28 August 2018)

	2000 ppm	**5000 ppm**	**10 000 ppm**
Start	2000 2001	5006 5180	10008 10045
End	2015 2015	5118 5100	10205 10190
Mean	**2008**	**5101**	**10112**
Std	**8**	**72**	**100**

Trial one – day two (10 September 2018)

	2000 ppm	**5000 ppm**	**10 000 ppm**
Start	2005 2018	5085 5079	10027 10033
End	2000 2001	5002 5010	10049 10110
Mean	**2006**	**5044**	**10055**
Std	**8**	**44**	**38**

Trial one – day one

% Bias for 2000 ppm IRS	**% Bias for 5000 ppm IRS**	**% Bias for 10 000 ppm IRS**
$\bar{x}$ is 2008 ppm μ is 2000 ppm $\% Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% Bias = \left[\frac{2008 - 2000}{2000}\right] 100$ $\% Bias = \left[\frac{8}{2000}\right] 100$ **% Bias = 0.4%** The state of COD concentrations analysis for 2000 ppm reference standard conducted in trial one at day 1 has a deviation of 0.4% from the true value of this reference standard.	$\bar{x}$ is 5101 ppm μ is 5000 ppm $\% Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% Bias = \left[\frac{5101 - 5000}{5000}\right] 100$ $\% Bias = \left[\frac{101}{5000}\right] 100$ **% Bias = 2.03%** The state of COD concentrations analysis for 5000 ppm reference standard conducted in trial one day 1 has a deviation of 2.03% from the true value of this reference standard.	$\bar{x}$ is 10 112 ppm μ is 10 000 ppm $\% Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% Bias = \left[\frac{10112 - 10000}{10000}\right] 100$ $\% Bias = \left[\frac{112}{10000}\right] 100$ **% Bias = 1.12%** The state of COD concentrations analysis for 10 000 ppm reference standard conducted in trial one at day 1 has a deviation of 1.12% from the true value of this reference standard.
Precision analysis: 2000 ppm IRS	**Precision analysis: 5000 ppm IRS**	**Precision analysis: 10 000ppm IRS**
Std = 8 $\bar{x}$ is 2008 ppm $\% \text{RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{RSD} = \left[\frac{8}{2008}\right] 100$ **%RSD = 0.3984%** The state of the COD concentrations analysis when the COD measurements conducted on 2000 ppm reference standard in Trial one day 1 is 0.4 % imprecise.	Std = 72 $\bar{x}$ is 5101 ppm $\% \text{RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{RSD} = \left[\frac{72}{5101}\right] 100$ **%RSD = 1.4115%** The state of the COD concentrations analysis when the COD measurements conducted on 5000 ppm reference standard in trial one day 1 is 2.03 % imprecise.	Std = 100 $\bar{x}$ is 10 112 ppm $\% \text{RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{RSD} = \left[\frac{100}{10112}\right] 100$ **%RSD = 0.9889%** The state of the COD concentrations analysis when the COD measurements conducted on 10 000 ppm reference standard in trial one day 1 at the start is 1.12 % imprecise.

Trial one – day two		
% Bias for 2000 ppm IRS	**% Bias for 5000 ppm IRS**	**% Bias for 10 000ppm IRS**
$\bar{x}$ is 2006 ppm µ is 2000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{2006 - 2000}{2000}\right] 100$ $\% \, Bias = \left[\frac{6}{2000}\right] 100$ **% Bias = 0.3%** The state of COD concentrations analysis for 2000 ppm reference standard conducted in trial 1 day 1 has a deviation of 0.3% from the true value of this reference standard.	$\bar{x}$ is 5044 ppm µ is 5000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{5044 - 5000}{5000}\right] 100$ $\% \, Bias = \left[\frac{44}{5000}\right] 100$ **% Bias = 0.88%** The state of COD concentrations analysis for 5000 ppm reference standard conducted in trial 1 day 1 has a deviation of 0.88% from the true value of this reference standard.	$\bar{x}$ is 10 055 ppm µ is 10 000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{10055 - 10000}{10000}\right] 100$ $\% \, Bias = \left[\frac{55}{10000}\right] 100$ **% Bias = 0.55%** The state of COD concentrations analysis for 10 000 ppm reference standard conducted in trial 1 day 1 has a deviation of 0.55% from the true value of this reference standard.
Precision analysis: 2000 ppm IRS	**Precision analysis: 5000 ppm IRS**	**Precision analysis: 10 000 ppm IRS**
Std = 8 $\bar{x}$ is 2006 ppm $\% \, RSD = \left[\frac{Std}{\bar{x}}\right] 100$ $\% \, RSD = \left[\frac{8}{2006}\right] 100$ **%RSD = 0.3988%** The state of the COD concentrations analysis when the COD measurements conducted on a 2000 ppm reference standard in trial two day 1 is 0.3988 % imprecise.	Std = 44 $\bar{x}$ is 5044 ppm $\% \, RSD = \left[\frac{Std}{\bar{x}}\right] 100$ $\% \, RSD = \left[\frac{44}{5044}\right] 100$ **%RSD = 0.8723%** The state of the COD concentrations analysis when the COD measurements conducted on a 5000 ppm reference standard in trial two day 1 is 0.8723 % imprecise.	Std = 38 $\bar{x}$ is 10 055 ppm $\% \, RSD = \left[\frac{Std}{\bar{x}}\right] 100$ $\% \, RSD = \left[\frac{38}{10055}\right] 100$ **%RSD = 0.3779%** The state of the COD concentrations analysis when the COD measurements conducted on a 10 000 ppm reference standard in trial two day 2 is 0.3779 % imprecise.

Appendix E2: %BIAS AND PRECISION CALCULATIONS ON THE REFERENCE STANDARDS DATA COLLECTED DURING THE EXPERIMENTAL TRIAL TWO.

Trial two – day 1 (30 September 2018)

	2000 ppm	5000 ppm	10 000 ppm
Start	2008	5002	10018
	2012	5003	10015
End	2028	5203	10349
	2038	5200	10351
Mean	**2022**	**5102**	**10183**
Std	**14**	**115**	**193**

Trial two – day 2 (29 October 2018)

	2000 ppm	5000 ppm	10 000 ppm
Start	2000	4994	10010
	2006	5001	10003
End	2015	5013	10103
	2021	5017	10079
Mean	**2011**	**5006**	**10049**
Std	**9**	**11**	**50**

Trial two – day one		
% Bias for 2000 ppm IRS	**% Bias for 5000 ppm IRS**	**% Bias for 10 000 ppm IRS**
$\bar{x}$ is 2022 ppm μ is 2000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{2022 - 2000}{2000}\right] 100$ $\% \, Bias = \left[\frac{22}{2000}\right] 100$ **% Bias = 1.1%** The state of COD concentrations analysis for 2000 ppm reference standard conducted in trial two day 1 has a deviation of 1.1% from the true value of this reference standard.	$\bar{x}$ is 5102 ppm μ is 5000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{5102 - 5000}{5000}\right] 100$ $\% \, Bias = \left[\frac{101}{5000}\right] 100$ **% Bias = 2.04%** The state of COD concentrations analysis for 5000 ppm reference standard conducted in trial two day has a deviation of 2.04% from the true value of this reference standard.	$\bar{x}$ is 10 183 ppm μ is 10 000 ppm $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{10183 - 10000}{10000}\right] 100$ $\% \, Bias = \left[\frac{183}{10000}\right] 100$ **% Bias = 1.83%** The state of COD concentrations analysis for 10 000 ppm reference standard conducted in trial two day 1 has a deviation of 1.83% from the true value of this reference standard.
Precision analysis: 2000 ppm IRS	**Precision analysis: 5000 ppm IRS**	**Precision analysis: 10 000 ppm IRS**
Std = 14 x̄ is 2022 ppm $\% \text{ RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{ RSD} = \left[\frac{14}{2022}\right] 100$ **%RSD = 0.6924%** The state of the COD concentrations analysis when	Std = 115 x̄ is 5102 ppm $\% \text{ RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{ RSD} = \left[\frac{115}{5102}\right] 100$ **%RSD = 2.2540%** The state of the COD concentrations analysis when	Std = 193 x̄ is 10 183 ppm $\% \text{ RSD} = \left[\frac{\text{Std}}{\bar{x}}\right] 100$ $\% \text{ RSD} = \left[\frac{193}{10183}\right] 100$ **%RSD = 1.8953%** The state of the COD concentrations analysis when the

the COD measurements conducted on 2000 ppm reference standard in trial two day 1 is 0.6924 % imprecise.	the COD measurements conducted on 5000 ppm reference standard in trial two day 1 is 2.2540% imprecise.	COD measurements conducted on 10 000 ppm reference standard in trial two day 1 is 1.8953 % imprecise.
Trial two – day two		
% Bias for 2000 ppm IRS	**% Bias for 5000 ppm IRS**	**% Bias for 10 000 ppm IRS**
***x* is 2011 ppm** **µ is 2000 ppm** $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{2011 - 2000}{2000}\right] 100$ $\% \, Bias = \left[\frac{11}{2000}\right] 100$ **% Bias = 0.55%** The state of COD concentrations analysis for 2000 ppm reference standard conducted in trial two day 2 has a deviation of 0.55% from the true value of this reference standard.	***x* is 5006 ppm** **µ is 5000 ppm** $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{5006 - 5000}{5000}\right] 100$ $\% \, Bias = \left[\frac{6}{5000}\right] 100$ **% Bias = 0.12%** The state of COD concentrations analysis for 5000 ppm reference standard conducted in trial two day 2 at the end has a deviation of 0.12% from the true value of this reference standard.	***x* is 10 049 ppm** **µ is 10 000 ppm** $\% \, Bias = \left[\frac{\bar{x} - \mu}{\mu}\right] 100$ $\% \, Bias = \left[\frac{10049 - 10000}{10000}\right] 100$ $\% \, Bias = \left[\frac{49}{10000}\right] 100$ **% Bias = 0.49%** The state of COD concentrations analysis for 10 000 ppm reference standard conducted in trial two day 2 has a deviation of 0.49% from the true value of this reference standard.
Precision analysis: 2000 ppm IRS	**Precision analysis: 5000 ppm IRS**	**Precision analysis: 10 000 ppm IRS**
Std = 9 x̄ is 2011 ppm $\% \, \mathrm{RSD} = \left[\frac{\mathrm{Std}}{\bar{x}}\right] 100$ $\% \, \mathrm{RSD} = \left[\frac{9}{2011}\right] 100$ **%RSD = 0.4475%** The state of the COD concentrations analysis when the COD measurements conducted on a 2000 ppm reference standard in trial two day 2 is 0.4475 % imprecise.	Std = 11 x̄ is 5006 ppm $\% \, \mathrm{RSD} = \left[\frac{\mathrm{Std}}{\bar{x}}\right] 100$ $\% \, \mathrm{RSD} = \left[\frac{11}{5006}\right] 100$ **%RSD = 0.2197%** The state of the COD concentrations analysis when the COD measurements conducted on a 5000 ppm reference standard in trial two day 2 is 0.2197 % imprecise.	Std = 50 x̄ is 10 049 ppm $\% \, \mathrm{RSD} = \left[\frac{\mathrm{Std}}{\bar{x}}\right] 100$ $\% \, \mathrm{RSD} = \left[\frac{50}{10049}\right] 100$ **%RSD = 0.4976%** The state of the COD concentrations analysis when the COD measurements conducted on a 10 000 ppm reference standard in trial two day 2 is 0.4976 % imprecise.

Appendix F1: TWO-TAILED T-TEST CALCULATIONS OF TRIAL ONE

Two-tail t-test: Reference standards mean differences - trial one-day one:

This test is carried out to examine if there was no significant difference between the reference standards measurements that were conducted when the experimental trials were carried out.

	Trial one – day one		
	IRS 2000p pm	**IRS 5000 ppm**	**IRS 10 000 ppm**
Mean	2008	5101	10112
Std	8	72	100

The test claim:

Null hypothesis (H_0): $X_{\text{Internal standard}} = \mu$

Alternate hypothesis (H_1): $X_{\text{Internal standard}} \neq \mu$

Trial one – day one	**Trial one – day one**	**Trial one – day one**
μ = 2000 ppm Mean = 2008 ppm S= 8 n= 4 $t = \frac{\bar{x}-\mu}{s/\sqrt{n}}$ $t = \frac{2008-2000}{8/\sqrt{4}}$ $t = \frac{8}{4}$ ***t* = 2.0000** T-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 5000 ppm Mean = 5101 ppm S= 72 n= 4 $t = \frac{\bar{x}-\mu}{s/\sqrt{n}}$ $t = \frac{5101-5000}{72/\sqrt{4}}$ $t = \frac{101}{36}$ ***t* = 2.8056** T-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 10 000 ppm Mean = 10 112 ppm s = 100 n= 4 $t = \frac{\bar{x}-\mu}{s/\sqrt{n}}$ $t = \frac{10112-10000}{100/\sqrt{4}}$ $t = \frac{112}{50}$ **t = 2.2400** T-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.

At 95% confidence ($\alpha/2 = 0.025$), the accepted criteria values are found to be -3.1820 and +3.1820. Since all the calculated t-statistical values found to fall within the non-rejection criteria, the null hypothesis is accepted for all the reference standards and the alternate is rejected. Therefore, a conclusion is reached based on the statistical evidence that the reference standard measurements conducted in trial one-day one are not significantly different compared to the true values of the reference standards.

Two-tail t-test: Reference standards mean differences - trial one – day two:

	Trial one – day two		
	IRS 2000 ppm	**IRS 5000 ppm**	**IRS 10 000 ppm**
Mean	2006	5044	10055
Std	8	44	38

The test claim:

Null hypothesis (H_0): $\bar{X}_{\text{Internal standard}} = \mu$

Alternate hypothesis (H_1): $\bar{X}_{\text{Internal standard}} \neq \mu$

Trial one – day two	**Trial one – day two**	**Trial one – day two**
μ = 2000 ppm Mean = 2006 ppm S= 8 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{2006 - 2000}{8/\sqrt{4}}$ $t = \frac{6}{4}$ **t = 1.5** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 5000 ppm Mean = 5044 ppm S= 44 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{5044 - 5000}{44/\sqrt{4}}$ $t = \frac{44}{22}$ **t = 2.0000** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 10 000 ppm Mean = 10 055 ppm s = 38 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{10055 - 10000}{38/\sqrt{4}}$ $t = \frac{55}{19}$ **t = 1.7739** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.

There is enough statistical evidence at 95% confidence limits ($\alpha/2 = 0.025$), to accept the null hypothesis and reject the alternate hypothesis. As the t-statistical values for all the reference standards measurements conducted in trial one - day two, are found to fall within the non-rejection region, it is concluded that there is no significant difference between the reference standard measurements means and the true values of the reference standards.

Appendix F2: TWO-TAILED T-TEST CALCULATIONS OF TRIAL TWO

Two-tail t-test: Reference standards mean differences - trial two-day one:

	Trial two – day two		
	IRS 2000 ppm	**IRS 5000 ppm**	**IRS 10 000 ppm**
Mean	2022	5102	10183
Std	14	115	193

The test claim:

Null hypothesis (H_0): $X_{\text{Internal standard}} = \mu$

Alternate hypothesis (H_1): $X_{\text{Internal standard}} \neq \mu$

Trial two – day one	**Trial two – day one**	**Trial two – day one**
μ = 2000 ppm Mean = 2022 ppm S=14 n= 4 $t = \frac{\bar{x} - \mu}{s / \sqrt{n}}$ $t = \frac{2022 - 2000}{14 / \sqrt{4}}$ $t = \frac{22}{7}$ ***t* = 3.1429** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 5000 ppm Mean = 5102 ppm S= 115 n= 4 $t = \frac{\bar{x} - \mu}{s / \sqrt{n}}$ $t = \frac{5102 - 5000}{115 / \sqrt{4}}$ $t = \frac{102}{57.5}$ ***t* = 1.7739** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 10 000 ppm Mean = 10 183 ppm s = 193 n= 4 $t = \frac{\bar{x} - \mu}{s / \sqrt{n}}$ $t = \frac{10183 - 10000}{193 / \sqrt{4}}$ $t = \frac{183}{96.5}$ **t = 1.8964** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.

There is enough statistical evidence at 95% confidence limits ($\alpha/2 = 0.025$), to accept the null hypothesis and reject the alternate hypothesis. As the t-statistical values for all the reference standards measurements conducted at the start of trial two are found to fall within the non-rejection region, it is concluded that at the time of the conduct of the experiments on this day, that there was no significant difference between the reference standard measurements means and the true values of the reference standards.

Two-tail t-test: Reference standards mean differences - trial two-day two:

	Trial two – day two		
	IRS 2000 ppm	**IRS 5000 ppm**	**IRS 10 000 ppm**
Mean	2011	5006	10049
Std	5	11	50

The test claim:

Null hypothesis (H_0): $X_{\text{Internal standard}} = \mu$

Alternate hypothesis (H_1): $X_{\text{Internal standard}} \neq \mu$

Trial two – day two	**Trial two – day two**	**Trial two – day two**
μ = 2000 ppm Mean = 2011 ppm S= 9 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{2011 - 2000}{9/\sqrt{4}}$ $t = \frac{11}{4.5}$ ***t* = 2.4444** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 5000 ppm Mean = 5006 ppm S= 11 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{5006 - 5000}{11/\sqrt{4}}$ $t = \frac{6}{5.5}$ ***t* = 1.0909** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.	μ = 10 000 ppm Mean = 10 049 ppm s = 50 n= 4 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{10049 - 10000}{50/\sqrt{4}}$ $t = \frac{49}{25}$ **t = 1.9600** t-statistical falls within the non-rejection region; therefore, the null hypothesis is accepted and the alternate is rejected.

There is enough statistical evidence at 95% confidence limits ($\alpha/2 = 0.025$), to accept the null hypothesis and reject the alternate hypothesis. As all the t-statistical values for the reference standards measurements conducted at the end of trial two (day 2) are found to fall within the non-rejection region. Therefore, it was concluded at the reference standard measurements conducted on this time had no significant difference compared to that of the true values of the reference standards.

All t-tests results statistically proved that at 95% confidence of limits, there is no significant difference between the reference standard means and the true values of the reference standards.

Appendix G: REFERENCE STANDARDS VALIDITY CALCULATIONS – ONE-WAY ANOVA

<u>One-way ANOVA test for: 2000 ppm internal reference standard mean variance test:</u>

Number of Sub-group	**Trial 1 –day 1 IRS in ppm**	**Trial 2 – day 2 IRS in ppm**	**Trial 2 – day 1 IRS in ppm**	**Trial 2 – day 2 IRS in ppm**
1	2000	2005	2008	2000
2	2001	2018	2028	2006
3	2015	2000	2012	2015
4	2015	2001	2038	2021
Mean	**2008**	**2006**	**2022**	**2011**
std	8	8	14	9

Test claim:

H_0: $X_{Tria11\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$

H_1: $X_{Trisl1\text{-}sDay1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Trial 1 -Start	4	8031	2007,75	70,25
Trial 1 - End	4	8024	2006	68,66667
Trial 2 - Start	4	8086	2021,5	195,6667
Trial 2 - End	4	8042	2010,5	87

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	581,1875	3	193,7292	1,83811	0,193962	3,490295
Within Groups	1264,75	12	105,3958			
Total	1845,938	15				

The upper tail critical value of 3.4903 was obtained from an F distribution at 95% confidence limits, at the degrees of freedom of c-1(3) and n-c(12). Therefore, since the calculated F-statistics of 1.8381 is less than the F-critical value (F-statistic <Fu/F-critical), the null hypothesis is accepted and the alternate hypothesis is rejected. Therefore, the conclusion is that, based on the statistical evidence, there is no significant difference between means of the 2000 ppm reference standard measurements conducted in trial 1 – day 1, trial 1 – day 2, trial two – day 1, and trial two – day 2.

One-way ANOVA test for: 5000 ppm Reference standard mean variance test:

Number of Sub-group	Trial 1– day1 IRS in ppm	Trial 1 – day2 IRS in ppm	Trial 2– day 1 IRS in ppm	Trial 2 – day 2 IRS in ppm
1	5006	5085	5001	4994
2	5180	5079	5004	5001
3	5118	5002	5207	5013
4	5100	5010	5195	5017
Mean	**5101**	**5044**	**5102**	**5006**
std	72	44	115	11

Test claim:

H_0: $X_{Tria11\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$

H_1: $X_{Trisl1\text{-}sDay1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Trial 1 -Start	4	20404	5101	5185,333
Trial 1 - End	4	20176	5044	1942
Trial 2 - Start	4	20407	5101,75	13159,58
Trial 2 - End	4	20025	5006,25	112,9167

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	26107,5	3	8702,5	1,706386	0,21862	3,490295
Within Groups	61199,5	12	5099,958			
Total	87307	15				

The upper tail critical value of 3.4903 obtained from an F distribution at 95% confidence limits, at the degrees of freedom of c-1(3) and n-c(12). Therefore, since the calculated F-statistics of 1.7064 is less than the F-critical value (F-statistic <Fu/F-critical), the null hypothesis is accepted and the alternate hypothesis is rejected. Therefore, the conclusion is that, based on the statistical evidence, there is no significant difference between means of the 5000 ppm reference standard measurements conducted in trial 1 – day1, trial 1 – day 2, trial 2 – day 1, and trial 2 – day 2.

One-way ANOVA test for: 10 000 ppm Reference standard mean variance test:

Number of Sub-group	**Trial 1 – day1 IRS in ppm**	**Trial 1 – day2 IRS in ppm**	**Trial two – day1 IRS in ppm**	**Trial two – day2 IRS in ppm**
1	10008	10027	10018	10010
2	10045	10033	10015	10003
3	10205	10049	10349	10103
4	10190	10110	10351	10079
Mean	**10112**	**10055**	**10183**	**10049**
std	100	38	193	50

Test claim:

H_0: $X_{Tria11\text{-}Day1} = X_{Trial1\text{-}Day2} = X_{Trial2\text{-}Day1} = X_{Trial2\text{-}Day2}$

H_1: $X_{Trisl1\text{-}sDay1} \neq X_{Trial1\text{-}Day2} \neq X_{Trial2\text{-}Day1} \neq X_{Trial2\text{-}Day2}$

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Trial 1 -Start	4	40448	10112	10012,67
Trial 1 - End	4	40219	10054,75	1442,917
Trial 2 - Start	4	40733	10183,25	37076,25
Trial 2 - End	4	40195	10048,75	2484,25

ANOVA

Source of *Variation*	SS	df	MS	F	P-value	F *crit*
Between Groups	46993,19	3	15664,4	1,228193	0,342154	3,490295
Within Groups	153048,3	12	12754,02			
Total	200041,4	15				

The upper tail critical value of 3.4903 obtained from an F distribution at 95% confidence limits, at the degrees of freedom of c-1(3) and n-c(12). Therefore, since the calculated F-statistics of 1.2282 is less than the F-critical value (F-statistic <Fu/F-critical), the null hypothesis is accepted and the alternate hypothesis was rejected. Therefore, the conclusion is that, based on the statistical evidence, there is no significant difference between means of the 10 000 ppm reference standard measurements conducted in trial 1 – day1, trial 1 – day 2, trial two – day1, and trial two – day 2. All the one-way ANOVA tests results demonstrate that there is no statistical evidence of variation from the measurements results of the same reference standards conducted at different intervals during the conduct of this research study.

Appendix H1: REFERENCE STANDARDS CALCULATIONS – LINEARITY FOR TRIAL ONE

Trial 1 – day 1: 28 August 2018.

	2000 ppm	**5000 ppm**	**10 000 ppm**
Start	2000	5006	10008
	2001	5180	10045
End	2015	5118	10205
	2015	5100	10190
Mean	**2008**	**5101**	**10112**
Std	**8**	**72**	**100**

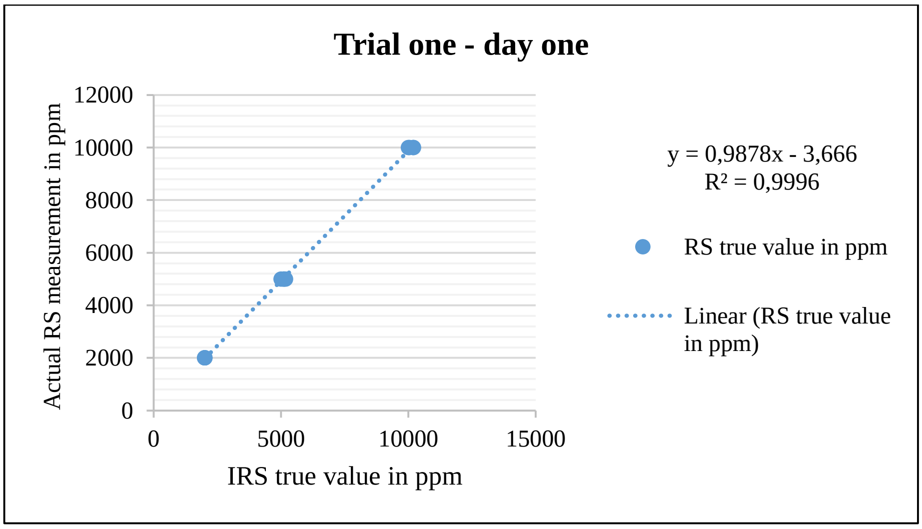
SUMMARY

Regression Statistics	
Multiple R	0,9998
R Square	0,999601
Adjusted R Square	0,999561
Standard Error	73,12059
Observations	12

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1,34E+08	1,34E+08	25025,51	2,5E-18
Residual	10	53466,2	5346,62		
Total	11	1,34E+08			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	6,002551	41,94609	0,143102	0,889052	-87,4592	99,46426	-87,4592	99,46426
RS true value in ppm	1,011926	0,006397	158,1945	2,5E-18	0,997673	1,026179	0,997673	1,026179



r^2 of 0.9996 and r of 0.9996 indicates good positive correlation or linearity.

F-ANOVA = 25025, 51; large number implies significant correlation.

The coefficient of determination for the reference standards measurements obtained for trial 1 day 1 is found to be 0.9996. This implies that 99.96% of the total variation in y-intercept can be explained by the linear relationship between the independent and the dependent variable(s). The remaining 0.04% variation in the dependent variable cannot be explained due to other factors.

Trial 1 – day 2: 10 September 2018.

	2000 ppm	**5000 ppm**	**10 000 ppm**
	2005	5085	10027
Start	2018	5079	10033
	2000	5002	10049
End	2001	5010	10110
Mean	**2006**	**5044**	**10055**
Std	**8**	**44**	**38**

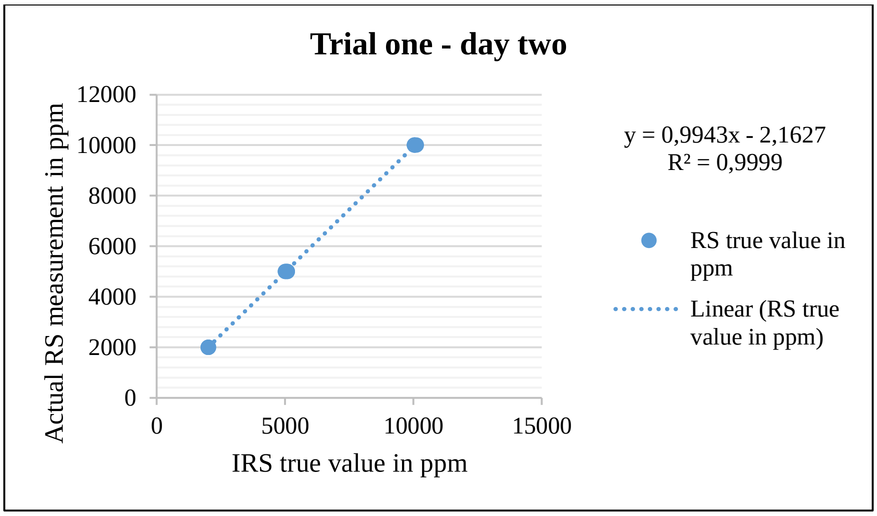
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,999957
R Square	0,999914
Adjusted R Square	0,999905
Standard Error	33,72902
Observations	12

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1,32E+08	1,32E+08	116168,1	1,16E-21
Residual	10	11376,47	1137,647		
Total	11	1,32E+08			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2,665816	19,34887	0,137776	0,893152	-40,4461	45,77778	-40,4461	45,77778
RS true value in ppm	1,005691	0,002951	340,8344	1,16E-21	0,999117	1,012266	0,999117	1,012266



r^2 of 0.9999 and r of 0.9999 indicates good positive correlation or linearity.

F-ANOVA = 25025, 51; large number implies significant correlation.

The coefficient of determination for the reference standard measurements obtained for trial one day 2 was found to be 0.9999. This implies that 99.99% of the total variation in y-intercept can be explained by the linear relationship between the independent and the dependent variable(s). The remaining 0.01% variation in the dependent variable cannot be explained due to other factors.

Appendix H2: REFERENCE STANDARDS CALCULATIONS – LINEARITY FOR TRIAL TWO

Trial 2 – day 1: 30 September 2018.

	2000 ppm	**5000 ppm**	**10 000 ppm**
	2008	5002	10018
Start	2012	5003	10015
	2028	5203	10349
End	2038	5200	10351
Mean	**2022**	**5102**	**10183**
Std	**14**	**115**	**193**

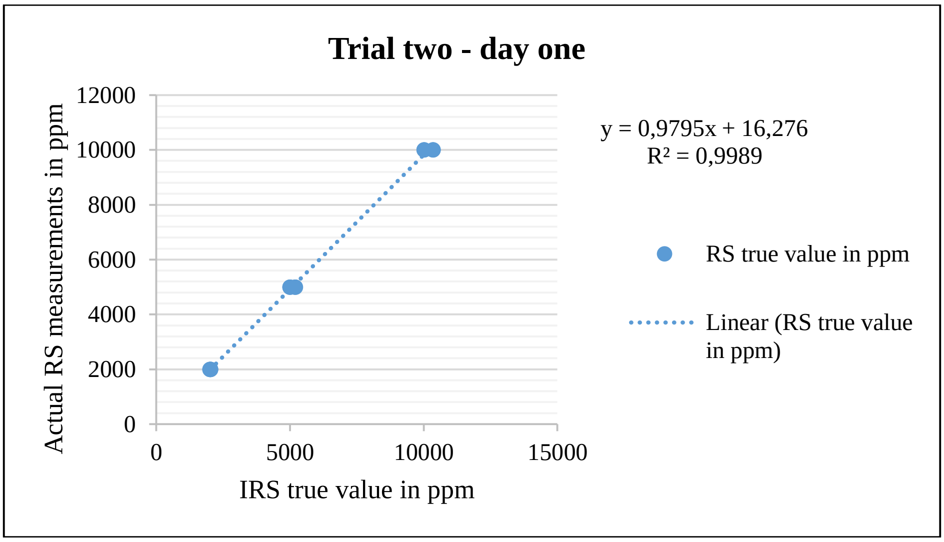
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,99944
R Square	0,998881
Adjusted R Square	0,998769
Standard Error	123,4088
Observations	12

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1,36E+08	1,36E+08	8923,156	4,33E-16
Residual	10	152297,4	15229,74		
Total	11	1,36E+08			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	-10,1403	70,79426	-0,14324	0,888949	-167,88	147,5991	-167,88	147,5991
RS true value in ppm	1,019819	0,010796	94,46246	4,33E-16	0,995764	1,043874	0,995764	1,043874



r^2 of 0.9989 and r of 0.9989 indicates good positive correlation or linearity.

F-ANOVA = 8923,156; large number implies significant correlation.

The coefficient of determination for the reference standards measurements obtained for trial 2 day 1 was found to be 0.9989. This implies that 9989% of the total variation in y-intercept can be explained by the linear relationship between the independent and the dependent variable(s). The remaining 0.11% variation in the dependent variable cannot be explained due to some other factors.

Trial 2 – day 2: 29 October 2018.

	2000 ppm	**5000 ppm**	**10 000 ppm**
Start	2000 2006	4994 5001	10010 10003
End	2015 2021	5013 5017	10103 10079
Mean	**2011**	**5006**	**10049**
Std	**9**	**11**	**50**

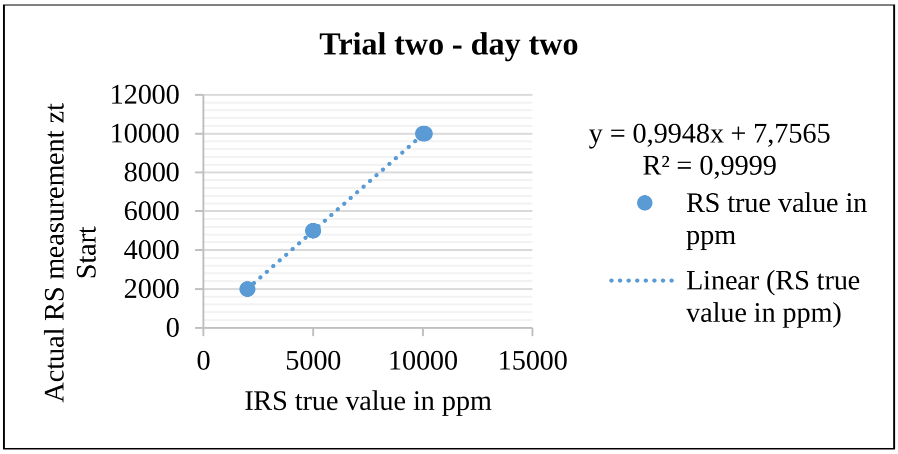
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,999966
R Square	0,999932
Adjusted R Square	0,999925
Standard Error	29,92595
Observations	12

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1,32E+08	1,32E+08	147414,4	3,53E-22
Residual	10	8955,625	895,5625		
Total	11	1,32E+08			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	-7,41071	17,16721	-0,43168	0,675134	-45,6616	30,84022	-45,6616	30,84022
RS true value in ppm	1,005161	0,002618	383,9458	3,53E-22	0,999328	1,010994	0,999328	1,010994



r^2 of 0.9999 and r of 0.9999 indicates good positive correlation or linearity.

F-ANOVA = 147414,4; large number implies significant correlation.

The coefficient of determination for the reference standards measurements obtained using trial 2 day 2 was found to be 0.9999. This implies that 9999% of the total variation in y-intercept can be explained by the linear relationship between the independent and the dependent variable(s). The remaining 0.01% variation in the dependent variable cannot be explained due to some other factors.

Appendix I1: ANION COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

	Anion														
	Days of production														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	851	5084	889	119	4384	1372	2832	143	88	72	1659	307	984	1378	915
2	2970	6307	1203	56	8584	1629	2040	146	57	53	1929	483	14478	2089	23954
3	3518	6062	10444	24	3598	1299	2138	127	97	66	19042	247	1695	7943	273
4	3909	3176	1824	41	3241	1324	42	109	62	74	3327	284	1135	33422	288
5	3218	3206	1795	41	3434	1199	119	138	69	982	1665	726	1228	2993	1968
6	3831	3224	575	27	1301	2672	116	96	72	738	1883	742	1188	2251	1940
7	5193	3892	801	57	447	4937	85	1400	80	14181	1725	800	1011	2329	1982
8	3833	6533	14673	2705	1525	4382	83	1655	96	841	1666	3532	865	2164	1740
9	5051	1280	3858	2368	1481	1554	1895	1521	76	49	1692	2622	2581	2167	1134
10	5033	1187	10810	6406	632	1516	1883	1390	86	46	10778	168	2192	2041	952
11	4670	997	1497	5431	434	2435	451	1420	334	52	8396	29	9283	1927	1222
12	5234	1144	1455	5376	2033	831	554	5376	620	44	7738	27	4914	11170	1251
Average	**3943**	**3508**	**4152**	**1888**	**2591**	**2096**	**1020**	**1127**	**145**	**1433**	**5125**	**831**	**3463**	**5990**	**3135**
std	**1258**	**2103**	**4893**	**2516**	**2310**	**1306**	**1043**	**1499**	**167**	**4031**	**5447**	**1099**	**4235**	**9132**	**6583**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	2810	229	2160	3370	1698	2073	831	1757	1506	27016	530	212	1368	6132	771
2	2964	119	13050	5533	17389	8293	1041	1631	1457	9309	1320	244	924	841	363
3	3264	452	14100	5623	4782	6383	4993	1888	1497	632	273	178	1099	494	20279
4	4471	3370	4800	5854	57183	2178	1759	1467	1681	192	526	6616	786	530	31491
5	3231	5533	3500	2059	2078	1294	1568	1627	765	180	1618	2368	635	6261	2796
6	340	5623	6640	2123	1738	1225	1204	1031	3633	150	2258	2131	647	1766	1194
7	240	6675	1565	11402	1623	7776	5274	1628	3965	89	326	1237	5855	307	1259
8	101	6651	2046	8435	35	19047	11092	6036	1614	72	202	1023	2824	380	1191
9	31271	533	3015	2043	854	3878	4284	2038	2015	93	187	12137	3273	340	1035
10	28990	2585	3406	1617	881	931	3681	2632	1849	443	162	3595	731	288	495
11	2622	340	4420	1582	1058	1091	2802	4545	503	1339	212	1840	644	6185	859
12	1111	240	3165	1112	1681	1161	3085	2360	9900	552	328	1854	674	1250	1605
Average	**6785**	**2696**	**5156**	**4229**	**7583**	**4611**	**3468**	**2387**	**2532**	**3339**	**662**	**2786**	**1622**	**2065**	**5278**
std	**11004**	**2740**	**4168**	**3214**	**16302**	**5294**	**2853**	**1453**	**2529**	**7894**	**687**	**3445**	**1601**	**2527**	**9937**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	4943	511	1287	541	1265	1090	139	149	3936	5608	1278	320	271	791	9863
2	647	320	1388	514	1760	14899	207	131	3919	4285	1263	294	264	721	3343
3	701	315	1373	18	1695	21151	297	625	3853	4106	1208	311	4021	1578	2979
4	790	311	1222	572	2608	5624	19227	4716	520	1048	5015	286	3222	11462	13135
5	758	948	1150	330	4309	1303	18882	2312	657	4264	982	185	2710	25444	2224
6	646	641	1587	347	3725	11476	561	1648	634	7506	978	183	1608	17083	3931
7	600	4502	781	11853	917	1852	432	1448	601	17584	983	279	937	4347	4395
8	546	2686	766	18105	668	243	574	1474	625	14370	947	1893	915	3443	1664
9	701	4141	639	2556	890	300	5403	1383	295	2341	888	21021	996	3881	1536
10	583	19398	598	1433	813	366	158	18680	270	1568	827	2544	884	3534	1980
11	543	2531	587	1440	677	395	148	10757	290	1310	1700	476	797	18515	1035
12	418	1617	583	1314	9352	347	139	953	1556	945	397	282	826	3737	3852
Average	**990**	**3160**	**997**	**3252**	**2390**	**4921**	**3847**	**3690**	**1430**	**5411**	**1372**	**2340**	**1454**	**7878**	**4161**
std	**1249**	**5325**	**373**	**5680**	**2503**	**7065**	**7254**	**5552**	**1528**	**5370**	**1189**	**5932**	**1208**	**8227**	**3652**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	3264	1480	752	1425	8044	2839	2714	16471	3484	88	1529	1777	101	1336	16
2	2246	14637	3450	3961	6651	2648	34622	554	1820	54	3975	3691	289	304	8
3	2423	12779	3634	3301	6984	2323	2250	36834	1639	18101	1668	2206	28368	20093	255
4	32631	30901	3418	6615	581	47009	2660	24395	1707	3395	17330	12035	3773	23093	296
5	12153	24568	705	4210	2817	47996	3272	3290	2533	3886	14227	3106	1788	571	245
6	22295	28099	850	31557	2748	37915	2148	3421	32892	2552	18959	25011	1876	454	1148
7	10369	3136	22801	22884	2442	3687	1602	2985	3120	2607	5203	20656	4539	492	2811
8	2187	1454	21390	1767	33098	15910	1292	3615	2469	928	3495	3357	1361	37	18
9	3004	977	3967	37436	2047	2745	694	62855	2628	914	3177	33830	824	45	15
10	9394	1122	2665	8934	2565	1002	5444	18543	1028	8356	2610	23414	401	9139	1303
11	2370	1829	45645	2814	2237	947	2010	38565	108	4344	2413	13757	3516	837	32899
12	4334	1308	3800	2583	3374	904	9074	3708	137	4311	2707	3609	2978	486	13539
Average	**8889**	**10191**	**9423**	**10624**	**6132**	**13827**	**5649**	**17936**	**4464**	**4128**	**6441**	**12204**	**4151**	**4741**	**4379**
std	**9615**	**11680**	**13722**	**12628**	**8795**	**18960**	**9397**	**19547**	**9016**	**4973**	**6429**	**11067**	**7767**	**8282**	**9754**

Appendix I2: CATION COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

	Cation														
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	123	6115	3295	272	4397	970	2730	380	164	77	1440	213	2362	1108	590
2	1972	6108	2247	102	5235	1026	2618	260	106	69	9919	4130	985	1267	4258
3	4718	6289	2430	57	3793	1088	2371	223	144	81	2186	2262	941	1688	261
4	3131	3026	2781	93	3395	933	152	284	100	69	1760	2213	931	13187	258
5	2989	3054	6307	70	3644	11018	105	280	111	1181	1702	3563	974	5317	1787
6	3363	17383	2040	55	15110	8574	100	232	100	791	1458	3448	917	2362	1698
7	3090	1953	9580	47	12449	8122	147	1730	116	2741	1463	1017	1192	2487	1628
8	3283	4360	5340	155	5296	1798	152	1815	109	941	1605	5047	1965	2396	1667
9	3485	2185	4240	1937	4157	1481	2704	1542	109	43	7266	2865	9479	2367	1898
10	4718	1435	4300	1894	4096	931	37309	1461	88	50	6293	158	1808	2300	1644
11	3582	1426	3230	11830	2471	1692	9742	965	67	42	1658	46	34091	2169	1522
12	3666	1159	3160	20000	34246	3718	3982	2578	2770	58	4575	46	6586	2311	2940
Average	**3177**	**4541**	**4079**	**3043**	**8191**	**3446**	**5176**	**979**	**332**	**512**	**3444**	**2084**	**5186**	**3247**	**1679**
std	**1210**	**4477**	**2153**	**6298**	**9071**	**3636**	**10482**	**817**	**768**	**815**	**2889**	**1767**	**9493**	**3300**	**1110**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	38157	2487	4346	3907	1475	3926	49374	19300	3880	1710	1967	23697	836	7190	1035
2	4612	2460	223	9991	1919	1958	33058	3860	4188	5076	2034	3842	1531	2138	1008
3	18859	2839	3160	4982	1552	1907	40858	1449	3845	7572	2193	992	17718	1144	803
4	3333	2735	2695	2267	1634	669	7861	6439	23585	2391	1922	20101	3648	835	352
5	4008	35726	2501	4735	1028	573	4968	1637	33809	1002	1483	10471	1954	435	785
6	2880	40233	28419	7830	26834	550	6581	2394	32918	2933	1967	837	6799	547	817
7	3417	40207	1934	4819	1641	641	7298	488	7166	1800	2999	1537	5225	655	855
8	3462	13002	2587	4890	11370	7995	2557	30200	8109	19767	32633	2379	3621	466	6931
9	2862	1049	7331	4831	3944	2037	2361	13243	33989	17832	33754	31640	3277	555	2248
10	26574	3553	3808	5303	2330	1783	1534	6350	3454	4836	2635	3565	1366	26411	4151
11	4553	11771	3846	8308	1713	2110	1198	2404	2714	3042	2741	9385	1195	9312	613
12	2565	14555	20566	8857	14711	2876	1236	1313	2573	2054	3215	1861	27780	1384	1070
Average	**9607**	**14218**	**6785**	**5893**	**5846**	**2252**	**13240**	**7423**	**13353**	**5835**	**7462**	**9192**	**6246**	**4256**	**1722**
std	**11779**	**15512**	**8603**	**2297**	**7944**	**2078**	**17312**	**9114**	**13447**	**6336**	**12032**	**10406**	**8193**	**7559**	**1933**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	501	1167	1604	1575	2121	5978	2032	8688	3919	4060	592	873	1980	42	22113
2	2102	964	14146	1529	931	1252	4447	211	370	5564	455	644	1658	40	22835
3	3103	1315	5101	1324	373	916	4985	653	17301	2200	630	799	294	42	559
4	1230	3174	2283	3410	717	1586	1101	493	2222	1243	369	668	2012	146	26742
5	960	1385	2811	19542	545	920	15279	2528	1218	1387	1101	577	1819	1202	1735
6	1187	965	1709	3529	11592	986	3186	19705	1585	1856	541	736	804	1378	556
7	810	1089	1018	1564	1557	3241	2231	4116	1873	5107	912	444	929	1018	514
8	720	633	918	1370	587	6735	1977	2106	1902	830	1146	1327	351	960	1327
9	881	1403	981	1371	861	2868	3050	429	13264	1902	756	1462	1304	1635	1559
10	957	8712	1089	1036	887	1791	5909	5418	2380	21973	45401	1260	1211	7604	1910
11	854	1681	3517	1138	820	1500	5338	4215	1444	3484	1367	1260	52	2530	1757
12	542	1304	1290	16594	877	1009	3468	3505	780	1572	1234	1242	43	1338	492
Average	**1154**	**1983**	**3039**	**4499**	**1822**	**2399**	**4417**	**4339**	**4022**	**4265**	**4542**	**941**	**1038**	**1495**	**6842**
std	**742**	**2211**	**3717**	**6422**	**3113**	**2000**	**3730**	**5443**	**5403**	**5790**	**12871**	**347**	**738**	**2072**	**10353**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	478	27575	210	683	404	5190	1854	36946	14902	13129	737	659	14904	568	22481
2	363	28311	1706	745	641	3608	1154	5041	4350	2672	669	723	645	530	8516
3	6666	78	2201	2295	19625	3634	721	3610	2071	2537	6154	2095	19373	557	1947
4	4334	303	728	9455	1121	2848	767	2687	1478	390	886	1686	1005	205	2600
5	482	221	910	1448	934	3552	731	2493	1749	372	754	1241	227	219	2163
6	870	119	562	1296	994	8618	902	2085	2459	390	10880	1098	197	169	1741
7	677	328	194	13321	4367	10543	12035	1801	1866	312	2153	1159	188	327	1907
8	922	70	8306	2619	2973	846	3143	2532	27212	10836	1239	979	193	330	13064
9	17134	201	1770	2018	2725	2281	3111	32921	3615	2290	881	31394	200	237	20535
10	2449	2951	2331	3359	3058	2132	3729	2723	2035	1611	916	1242	17098	777	5585
11	1855	5851	654	412	1217	1120	555	2277	2041	695	668	412	498	190	2459
12	1777	400	760	399	5763	1836	35703	5608	1692	668	884	13504	557	1621	2402
Average	**3167**	**5534**	**1694**	**3171**	**3652**	**3851**	**5367**	**8394**	**5456**	**2992**	**2235**	**4683**	**4590**	**478**	**7117**
std	**4783**	**10608**	**2208**	**4032**	**5293**	**2961**	**10067**	**12480**	**7790**	**4320**	**3128**	**9144**	**7622**	**409**	**7552**

Appendix I3: GLUCOSE COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Glucose															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	21943	10501	13356	5887	5999	6217	8540	4270	8942	7038	6298	4286	4373	4117	5813
2	11803	6668	9443	6780	9143	4762	10090	8241	5351	2657	4282	10330	3590	13443	8852
3	16613	13691	5857	9002	8481	7501	2590	3524	4972	2251	6182	6888	5602	9738	4097
4	28603	15701	7326	12768	6528	3796	1536	8253	5631	2046	10128	6213	1063	19549	5553
5	1843	11706	11366	13363	8390	5792	2980	4549	4080	2095	3904	4614	6778	3433	5748
6	15029	11156	8875	12921	5230	6525	864	5062	3856	2205	4854	4004	6207	5155	8818
7	12771	10475	6663	12749	8439	5187	819	6186	3526	3073	4290	24000	3965	5856	8518
8	10539	9728	7888	19287	6059	4097	1219	4415	4721	2874	6569	3912	2903	4822	8743
9	10429	10040	6493	19924	5982	5262	1162	2487	3959	4136	5873	3433	3064	4869	8074
10	7093	9366	5124	16855	5737	4663	1305	5301	3411	5024	7012	3764	3621	4204	7880
11	7229	5703	4456	17806	8647	8310	5710	3828	5085	4490	6523	1845	4358	4093	7501
12	6664	6525	5424	11392	9542	8910	4065	8461	6899	3303	5683	2460	4660	4895	5016
Average	**12547**	**10105**	**7689**	**13228**	**7348**	**5919**	**3407**	**5381**	**5036**	**3433**	**5967**	**6312**	**4182**	**7015**	**7051**
Std	**7293**	**2905**	**2668**	**4601**	**1549**	**1633**	**3141**	**1995**	**1586**	**1502**	**1664**	**6008**	**1551**	**4882**	**1696**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	8210	1638	45216	5061	4952	6571	10818	2155	10049	15231	14705	8608	4098	8008	28314
2	585	1296	26954	9596	4126	11396	10464	2036	13954	10519	17587	9440	3133	6374	3549
3	2190	1420	17321	6040	16293	7064	10130	16242	9591	10264	1525	7376	6891	12833	6708
4	1727	1853	59691	6425	12818	5347	10285	16551	4802	9807	4579	16232	8664	15869	4524
5	1649	739	125625	5633	11531	5943	13612	10251	3952	10040	29943	12456	9611	11913	7407
6	1490	740	8117	5688	6119	3343	26950	10023	5037	13858	9383	5172	8656	10208	2553
7	9090	8280	2687	8271	6281	10799	8120	5371	4388	24331	22967	4690	9916	11110	19875
8	5820	6720	2397	8735	5975	2181	6809	10562	8914	21010	23303	5132	9490	13401	19968
9	3500	6416	2128	7420	5132	2222	4420	12741	8440	16141	23826	4094	8656	9591	24061
10	3130	14930	9749	7087	5569	3704	5369	9401	8487	13610	18977	5762	9915	4160	16700
11	2230	10242	10486	6584	3937	6484	11263	7277	8021	11825	11655	4575	13987	4591	9678
12	1765	8252	8654	6453	3664	7741	9331	8588	4155	92542	10125	3859	9553	12108	7090
Average	**3449**	**5211**	**26585**	**6916**	**7200**	**6066**	**10631**	**9267**	**7483**	**20765**	**15715**	**7283**	**8548**	**10014**	**12536**
Std	**2769**	**4631**	**36052**	**1366**	**4058**	**2991**	**5760**	**4666**	**3067**	**23053**	**8564**	**3821**	**2833**	**3619**	**8795**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	5582	8745	12931	11571	10119	11371	10119	9360	9270	11278	2423	8911	5894	7491	17542
2	3247	8145	11234	12055	6905	12055	6905	10882	7090	13360	3156	9246	7040	7466	24587
3	2161	8889	9360	18113	19032	18113	19032	17225	4990	11050	3785	7675	5746	5961	24512
4	3141	8353	10822	22665	21730	22665	21730	10600	4240	11135	5664	12378	6032	19031	14255
5	22068	15125	17225	10251	19476	10251	19246	3837	3930	10317	5112	18807	6491	5190	12455
6	3603	14507	10600	5630	9312	5630	9312	3600	3550	10276	4730	10032	5148	7787	10125
7	3882	14463	3837	5395	6214	5395	6214	4350	6600	13227	4310	5326	13085	7176	12451
8	11291	13528	3600	5018	7040	5018	7040	6191	5410	12532	3092	6606	10948	8774	14755
9	4375	13557	4350	16295	4754	16295	4754	6858	12790	6568	2951	4769	6776	8802	16854
10	13725	13140	6191	12969	4562	12969	4562	6140	7485	3129	1739	2826	7970	6953	19514
11	18125	12912	6858	8961	5241	8961	12931	1794	6898	3334	4164	4648	7974	13063	8245
12	16098	5252	7245	9726	4985	9726	11234	10250	4016	3807	10769	3424	6659	9920	25485
Average	**8942**	**11385**	**8688**	**11554**	**9948**	**11537**	**11090**	**7591**	**6356**	**9168**	**4325**	**7887**	**7480**	**8968**	**16732**
Std	**6971**	**3281**	**4111**	**5383**	**6375**	**5383**	**5960**	**4281**	**2672**	**3882**	**2326**	**4481**	**2322**	**3759**	**5550**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	7007	14826	7998	12365	27102	4809	13781	18421	10125	4772	9781	9347	14104	11570	3036
2	6260	11828	14839	26107	16973	4524	9908	12896	15257	2885	9175	7742	14065	10269	2902
3	7142	5701	11042	17194	16619	3708	8015	11459	16657	3057	9627	2475	11387	25879	2859
4	5703	9883	4499	30461	8353	6065	6707	9339	12790	2109	8654	15580	11364	20556	570
5	5355	5453	2474	28454	8397	10143	6461	8235	8775	14169	5201	11271	4949	16277	1843
6	12352	8241	4403	6080	6208	9928	15219	8168	9496	10741	1979	11110	5393	8962	6914
7	9460	8489	5058	6053	5427	14565	13398	1717	8689	11439	3732	22590	7025	7636	4806
8	6908	6034	6451	9015	11715	13409	11839	1782	9758	12515	2639	16997	9713	8189	9204
9	6474	5770	4289	7523	7670	12159	19388	4622	11956	12797	3154	14352	9897	7870	10986
10	6265	2352	7853	5506	9430	12325	23410	3444	7925	8304	4384	18648	9801	7470	7877
11	10127	6810	6314	9776	15709	10632	16352	11065	6939	11422	6575	15323	10560	3785	10830
12	14346	6089	9364	18651	5077	11994	21887	10710	7869	12842	9476	13549	9843	2107	4875
Average	**8117**	**7623**	**7049**	**14765**	**11557**	**9522**	**13864**	**8488**	**10520**	**8921**	**6198**	**13249**	**9842**	**10881**	**5559**
Std	**2849**	**3316**	**3444**	**9227**	**6480**	**3768**	**5675**	**4960**	**3046**	**4488**	**3022**	**5322**	**2906**	**6862**	**3544**

Appendix I4: EFFLUENT TANK COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Effluent tank															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	24892	12604	12583	5688	7233	6146	4764	4297	8583	6357	5013	4034	3989	9149	5061
2	2713	8345	12784	7666	5717	6937	4640	7894	5477	6156	5758	4777	4000	15713	9596
3	10632	12219	15234	8596	9426	8250	3846	7813	5047	2304	4821	7046	7200	44680	6040
4	32987	13684	13645	12846	8495	7225	3686	7777	5153	3016	10438	6013	3946	26215	6425
5	31438	11547	10814	16655	8475	5603	4576	4876	4002	2243	4233	4105	9931	7380	5633
6	15417	12229	15071	12346	7321	6710	6540	5758	3684	2214	5123	4315	5934	5409	5688
7	13135	9481	6273	12052	8296	8800	6587	3234	3601	2387	4917	3593	3557	5209	8271
8	10017	8916	7575	22122	6005	8876	7994	3768	4429	2435	6016	3544	2849	5250	8735
9	10515	9909	7188	21853	6573	4844	4264	2290	3956	2657	5737	3504	3295	4749	7420
10	6993	9297	8924	20712	5958	4143	4006	5171	2979	2414	6446	3073	8325	4349	7087
11	5608	5973	5242	21450	5790	5329	4735	3785	4385	3842	6977	2158	5023	4200	6584
12	7405	5624	4248	10822	5561	4731	4618	6308	6097	3570	6274	2993	3279	4216	6453
Average	**14313**	**9986**	**9965**	**14401**	**7071**	**6466**	**5021**	**5248**	**4783**	**3300**	**5979**	**4096**	**5111**	**11377**	**6916**
std	**10066**	**2571**	**3885**	**5952**	**1332**	**1609**	**1315**	**1896**	**1486**	**1478**	**1609**	**1342**	**2269**	**12345**	**1366**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	3204	5691	14592	14573	4737	2936	2807	7021	10523	4338	3716	4702	5967	10828	3978
2	3768	5916	23582	14199	4121	6610	1715	6893	14827	2523	3062	6425	8116	9886	2865
3	4089	5233	16330	12096	6467	5265	537	10008	17896	5729	3187	8070	7705	9281	2582
4	4253	4664	14836	5528	13798	8429	9552	10985	11846	6764	2796	8626	5515	8706	6484
5	2765	5092	22078	11504	11270	9853	10148	12431	11023	3789	3883	7495	12083	10609	8624
6	8578	4950	6376	6368	5830	8993	9664	12993	20894	2579	3046	9663	11303	7161	8259
7	6444	6424	5594	6628	5339	8285	25446	12348	18652	5614	30528	8067	9747	10695	1662
8	6372	5921	5101	5780	5494	4464	21762	8415	13221	4284	12390	6146	12947	11535	1453
9	5472	5597	4789	8137	5083	2129	15940	5055	17906	5385	5216	8498	11895	7963	4549
10	5227	5413	12230	15528	5147	4582	10387	5342	16082	3818	3990	7008	8980	7402	5016
11	5036	2582	11955	21301	3636	2229	7960	7565	13470	3684	4784	6849	10310	8452	7234
12	5910	5090	8685	4256	3486	1963	7016	8448	4557	1924	3633	7559	13976	5483	7502
Average	**5093**	**5214**	**12179**	**10492**	**6201**	**5478**	**10245**	**8959**	**14241**	**4203**	**6686**	**7426**	**9879**	**9000**	**5017**
std	**1621**	**963**	**6411**	**5217**	**3128**	**2889**	**7598**	**2761**	**4468**	**1463**	**7942**	**1315**	**2703**	**1806**	**2569**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	10902	11088	7893	6479	5709	7512	12728	8388	8152	8686	6726	2211	14382	7882	17466
2	11286	5618	8947	4630	7718	6023	11019	6542	9328	7667	5960	2015	10310	8249	7029
3	12455	8738	14000	9265	6116	8849	6326	6844	6822	5566	3833	4646	10770	10242	4221
4	13055	12159	5876	17061	9076	14064	8373	5700	11251	5109	906	5511	9437	8483	3371
5	13563	12325	4755	23702	12077	12672	11068	7951	9527	6164	10441	5169	9725	9853	4833
6	7878	10632	4056	18352	16681	7008	8355	14534	5339	4463	8418	4306	13179	12201	4833
7	18351	11994	3981	17259	25677	6920	8260	8400	14754	3308	8465	4061	11799	11853	5750
8	18941	13781	5862	9857	21969	5140	7319	7382	12216	4902	13187	3380	6448	10113	6859
9	22956	9908	9222	9196	16478	5588	7619	6058	8100	4500	13312	10146	13647	13901	11617
10	20146	8015	12310	3330	8813	7625	7673	7002	7045	6100	2703	6406	11512	999	9818
11	15271	6707	13979	3513	5270	10278	6372	7585	7908	6571	3096	8146	11307	7957	9258
12	20390	6461	13596	11629	9011	15612	5401	7502	8376	6724	3387	8355	9416	7190	6470
Average	**15433**	**9786**	**8706**	**11189**	**####**	**8941**	**8376**	**7824**	**9068**	**5813**	**6703**	**5363**	**10994**	**9077**	**7627**
std	**4642**	**2654**	**3924**	**6569**	**6700**	**3468**	**2183**	**2275**	**2592**	**1493**	**4136**	**2507**	**2175**	**3252**	**3946**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	18126	5994	14506	11038	3938	4967	2185	18640	3684	4517	7263	7082	6014	4564	2486
2	20595	5848	17461	16461	4528	11117	3649	17331	1924	6811	8389	4084	5607	3296	16075
3	19476	7910	19708	5173	3565	8911	3434	12873	3716	4394	6072	7228	9871	7130	27866
4	9618	19334	10696	34219	4214	9141	4203	26179	3062	5288	5602	6828	14099	6300	10733
5	5995	15869	12141	25953	9192	10378	5137	23488	3187	6650	5128	7305	9770	2320	9709
6	5787	4656	11377	16344	11456	8529	5154	24020	2796	8391	4138	8326	11819	12470	24660
7	5057	6160	13226	17083	9998	11357	7533	16231	3883	14331	4111	9993	9225	6960	11137
8	4633	7848	9700	16178	4482	11254	5949	14232	3046	12278	3587	9579	5951	8410	13804
9	10142	8752	4707	12466	5101	14092	4432	7134	30528	9872	7361	5754	9658	5349	12753
10	17702	10614	6796	14814	5679	9550	5486	7579	13290	11128	9104	6216	6631	3995	10860
11	16776	11373	11537	11558	6468	3695	3051	5385	11083	10128	7375	6355	5912	6780	11862
12	11391	9124	11365	9953	10241	5309	10213	3818	7849	9847	7805	5775	3150	5112	7041
Average	**12108**	**9457**	**11935**	**15937**	**6572**	**9025**	**5036**	**14743**	**7337**	**8636**	**6328**	**7044**	**8142**	**6057**	**13249**
std	**6099**	**4351**	**4104**	**7655**	**2844**	**3038**	**2168**	**7612**	**8145**	**3171**	**1820**	**1655**	**3105**	**2674**	**6992**

Appendix I5: LIQUEFACTION COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

	Liquefaction														
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	477	88	263	102	102	149	64	315	150	56	20	95	74	1341	146
2	518	76	222	81	121	156	57	324	92	56	20	59	245	159	108
3	121	56	197	30	444	122	147	297	78	70	95	51	66	178	90
4	113	52	243	43	299	137	128	208	68	68	81	58	66	130	89
5	306	69	153	39	239	128	126	134	64	116	94	65	42	1953	86
6	179	50	94	32	152	96	115	350	59	69	101	47	142	1825	92
7	180	59	121	48	143	73	159	232	60	105	98	39	106	1543	91
8	477	60	211	90	135	72	475	97	64	39	102	42	107	223	94
9	256	102	371	93	188	88	176	87	131	30	104	58	77	142	104
10	1	104	236	74	950	129	107	132	181	30	156	54	72	17	96
11	7	170	292	90	133	185	151	121	196	29	148	877	766	1241	1246
12	11	161	41	35	128	177	104	131	3553	3029	2773	719	153	1044	193
Average	**221**	**87**	**204**	**63**	**253**	**126**	**151**	**202**	**391**	**308**	**316**	**180**	**160**	**816**	**203**
std	**189**	**41**	**91**	**28**	**240**	**38**	**108**	**98**	**997**	**857**	**775**	**291**	**199**	**745**	**330**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	233	2986	191	198	2902	930	313	215	184	146	587	3448	1444	217	2811
2	97	22	145	93	2582	3000	997	235	120	130	1447	1989	1618	151	1404
3	134	11	2089	116	2590	492	724	170	115	90	377	2237	519	181	769
4	260	512	145	152	307	84	140	211	133	105	520	1860	609	196	370
5	163	3560	151	130	559	85	137	170	115	110	1017	994	617	707	279
6	272	711	160	311	675	108	155	271	126	142	382	1654	446	231	260
7	121	169	151	143	119	99	1315	113	134	95	1560	1727	481	204	214
8	152	223	104	144	116	78	1312	308	335	81	2883	823	201	167	261
9	3722	115	108	134	138	89	1388	184	309	105	2722	637	182	266	452
10	2852	140	127	133	965	75	1307	132	190	104	1249	934	255	232	420
11	1226	281	138	129	309	180	1515	184	334	420	1866	663	342	830	557
12	1166	131	212	146	501	260	483	132	133	757	27474	2245	240	3733	238
Average	**867**	**738**	**310**	**152**	**980**	**457**	**816**	**194**	**186**	**190**	**3507**	**1601**	**580**	**593**	**670**
std	**1211**	**1207**	**561**	**56**	**1064**	**840**	**549**	**58**	**88**	**200**	**7595**	**836**	**471**	**1013**	**752**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	200	187	102	174	166	127	420	278	1733	340	115	18	12	131	36
2	185	197	311	1987	149	172	485	197	1944	134	80	43	12	102	41
3	193	195	223	1636	1184	145	357	219	1283	126	63	36	105	79	30
4	416	424	157	1045	600	156	86	115	103	105	213	12	118	94	28
5	474	250	179	899	103	164	103	108	96	120	181	50	107	104	47
6	335	311	133	1008	110	170	83	125	109	120	260	27	119	70	38
7	342	186	139	770	146	403	516	166	156	139	206	23	222	103	54
8	168	240	173	1169	137	35	76	209	147	125	184	52	334	109	37
9	169	202	163	782	124	33	103	171	234	99	24	54	36	81	66
10	203	214	145	771	114	37	107	135	445	58	28	22	12	1452	49
11	137	292	174	394	104	61	161	1315	181	96	48	39	20	366	36
12	326	336	181	183	253	261	187	973	245	67	27	25	58	47	41
Average	**262**	**253**	**173**	**902**	**266**	**147**	**224**	**334**	**556**	**127**	**119**	**33**	**96**	**228**	**42**
std	**111**	**74**	**53**	**537**	**320**	**107**	**170**	**388**	**683**	**72**	**85**	**14**	**98**	**394**	**11**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	280	158	132	189	46	75	550	191	179	447	239	1401	115	192	151
2	181	125	124	271	114	181	174	606	210	640	474	2970	373	197	119
3	231	63	172	255	5	166	190	698	123	347	557	1675	380	5662	113
4	30	53	118	168	2	118	479	765	134	575	559	765	562	540	121
5	72	31	72	190	189	26773	22348	823	587	341	147	690	211	306	119
6	41	17	61	191	119	28597	28827	10	132	597	505	109	152	179	114
7	90	89	30	229	150	22498	27250	11	240	335	291	87	174	143	112
8	45	62	32	209	149	21432	27071	14	183	1321	269	96	326	179	81
9	178	87	39	1919	1276	23373	24083	175	118	733	1295	104	266	157	311
10	33	61	40	132	74	20657	21676	133	158	223	1285	33	210	164	136
11	49	55	60	259	79	6559	276	140	127	298	882	130	197	87	235
12	63	207	185	34	90	1835	345	338	312	328	1331	99	184	129	100
Average	**108**	**84**	**89**	**337**	**191**	**12689**	**12772**	**325**	**209**	**515**	**653**	**680**	**263**	**661**	**143**
std	**86**	**55**	**55**	**502**	**346**	**12013**	**13139**	**311**	**132**	**299**	**437**	**913**	**127**	**1579**	**65**

Appendix I6: CONDENSATE COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Condensate															
	Number of day														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	8698	8236	6987	5661	7752	20770	8836	7372	6015	4047	20611	7313	12043	9242	8777
2	8432	8694	12632	5696	7418	20068	8557	7593	6301	4165	20372	8473	26174	12147	8693
3	7842	9530	11315	5092	6792	18799	7953	8361	5733	4849	20174	8459	10203	15464	8602
4	7383	8808	8810	7596	6855	21107	6714	9370	4671	4136	20555	8293	17204	32440	8381
5	5845	8735	9514	8144	7355	20922	7908	6570	3390	6914	8253	8351	9987	9086	8066
6	3651	9048	11254	8146	7677	20838	10373	5203	3355	6449	6359	8339	10346	9036	7936
7	9454	60765	4122	8056	7269	23029	9551	6530	32086	10097	8137	9516	9604	12531	9591
8	11256	52234	4256	7148	8055	22399	6431	6674	20213	10260	7728	10098	8689	10104	8946
9	4145	12885	12145	7902	6960	17671	6252	5941	16971	11018	24150	11278	7886	9315	7948
10	5421	12067	14323	6124	5214	16974	6190	6254	22849	9870	22542	10763	7501	9178	7678
11	9252	6459	125432	5243	14543	16938	7562	5455	4415	7515	21809	8312	7778	8634	7511
12	12254	8242	10555	6475	5356	15325	7664	4256	2199	5215	12541	5154	7729	7572	6988
Average	**7803**	**17142**	**19279**	**6774**	**7604**	**19570**	**7833**	**6632**	**10683**	**7045**	**16103**	**8696**	**11262**	**12062**	**8260**
std	**2664**	**18552**	**33580**	**1190**	**2355**	**2400**	**1330**	**1403**	**9799**	**2662**	**6853**	**1615**	**5405**	**6773**	**713**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	7960	7732	16302	7714	11257	13117	91056	11414	12961	16689	15959	19271	6660	16317	28701
2	7141	7582	166689	9878	10751	14800	14922	10868	15780	14577	6296	19059	17504	17348	21060
3	7586	6493	12756	6769	10608	31817	22965	10500	15341	12972	13427	18736	18346	18758	29108
4	8207	7133	13384	12235	10279	30501	22614	11042	18195	15038	9342	17639	27110	18591	18132
5	8957	7112	10704	10235	10970	12140	20886	11688	17576	15873	10289	17692	20919	19655	30713
6	9259	7909	11006	10909	10252	11131	21300	10447	11764	15016	1689	20636	9380	20246	33044
7	8717	7055	9608	8647	11030	12000	10493	12576	18380	14430	1935	19913	20770	16947	31137
8	9922	7017	4784	12115	10539	11131	12294	11686	14033	14702	1044	55146	18268	16198	25552
9	9902	7200	8416	10235	10142	12000	12294	45860	11966	14505	21693	5219	13115	16979	31081
10	9539	6942	7910	46112	12300	11968	11552	13077	5281	15323	21355	18786	20302	16106	31766
11	9492	6958	9332	17947	12360	10917	11812	12080	7634	15498	23276	16709	20053	14474	30482
12	7215	4245	6215	10124	9542	25874	10985	12812	16321	12728	21236	17276	18487	28713	29096
Average	**8658**	**6948**	**23092**	**13577**	**10836**	**16450**	**21931**	**14504**	**13769**	**14779**	**12295**	**20507**	**17576**	**18361**	**28323**
std	**1013**	**934**	**45331**	**10624**	**834**	**7987**	**22314**	**9912**	**4113**	**1108**	**8425**	**11618**	**5510**	**3651**	**4530**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	11660	28904	40372	13443	14993	17540	15232	18930	15372	64738	30892	31927	10412	15420	16012
2	31173	34268	34629	14772	19361	17133	10461	15667	31120	18110	34446	22816	22734	13929	11178
3	29983	21224	33057	20107	19082	20987	9416	11953	23042	19737	22985	23370	23267	20857	6064
4	26965	26446	29603	15040	19901	19431	6152	50123	19940	21694	22850	15789	22547	18575	6459
5	29409	29601	25899	17232	32622	18461	6046	17982	21803	20824	20479	23136	24439	14151	3991
6	31319	35444	23642	20954	20050	18785	5955	16440	10311	19234	74668	21541	2248	16451	11106
7	28921	30566	14742	17401	18196	18958	20736	19612	9146	19965	21107	29894	21105	16833	16724
8	18150	29865	14152	17298	17839	19265	20443	18950	14576	19370	23011	34640	23814	21524	16412
9	23179	37308	17064	16353	18732	18748	20674	17171	14422	21141	20305	20273	14969	21975	10411
10	32992	21807	18257	16514	17616	18923	22016	17104	21374	20755	21042	23275	37416	20283	17463
11	18915	23680	20939	16607	19229	20748	25750	10561	7706	30309	20828	20152	13859	19806	11551
12	19681	29211	17921	24995	18115	25169	24271	14154	25786	19794	22281	17587	13529	16051	12360
Average	**25196**	**29027**	**24190**	**17560**	**19645**	**19512**	**15596**	**19054**	**17883**	**24639**	**27908**	**23700**	**19195**	**17988**	**11644**
std	**6732**	**5125**	**8570**	**3134**	**4301**	**2093**	**7588**	**10176**	**7151**	**13002**	**15375**	**5689**	**8881**	**2873**	**4478**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	13570	16216	12296	12858	15002	13801	17014	18614	18995	18905	14677	13827	7356	17178	1937
2	12400	12899	14217	13836	15419	15801	16445	16116	18603	11158	11865	12933	7373	20123	3046
3	13965	25698	15530	12616	27182	15840	15651	9106	17734	18086	14350	13035	3731	3768	2732
4	12340	12004	14294	17961	25522	17140	17339	5946	17037	16086	14303	12495	13804	4046	2570
5	11614	30068	27243	15836	17189	24180	29773	6691	17264	15829	10350	12897	8652	4299	4206
6	11803	7302	25324	15914	14718	6681	13912	18265	16518	15696	17148	5047	4012	3564	5887
7	11019	6411	26204	17320	15322	4256	15844	16665	18882	14542	9987	7266	10958	2670	6031
8	10524	10472	26236	21798	15646	1538	15690	16273	19855	14343	73648	6653	7729	2262	22441
9	10855	9563	14426	16925	14833	1307	17496	14761	39180	15099	13710	6714	10015	3977	6335
10	9681	13491	13809	13887	15188	16417	19185	37337	36611	13277	14969	6310	10570	3522	2353
11	13460	13486	14999	13053	16119	14435	18146	21460	37689	13808	13755	8457	5418	4179	2658
12	16084	13219	16634	15961	15137	17406	19309	20874	36351	14777	13211	4380	8236	3047	2763
Average	**12276**	**14236**	**18434**	**15664**	**17273**	**12400**	**17984**	**16842**	**24560**	**15134**	**18498**	**9168**	**8155**	**6053**	**5247**
std	**1771**	**7008**	**5876**	**2661**	**4307**	**7211**	**4023**	**8255**	**9593**	**2062**	**17481**	**3572**	**2938**	**5949**	**5636**

Appendix I7: EVAPORATOR COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Evaporator cpv															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	381	505	114	43	303	106	144	82	49	79	4	58	56	2122	26
2	78	590	137	18	106	126	94	80	66	207	6	53	63	303	119
3	127	358	276	9	82	63	69	60	87	266	180	48	89	143	92
4	128	710	293	9	257	52	128	555	90	170	103	40	80	169	72
5	169	726	647	10	160	76	125	47	120	162	133	63	109	153	74
6	173	622	272	10	133	105	103	52	86	95	107	69	96	157	78
7	753	757	563	17	119	50	96	78	96	187	142	51	83	583	146
8	667	1122	884	368	70	50	103	103	75	166	144	37	77	620	138
9	617	626	240	323	87	42	125	161	75	65	66	33	76	177	85
10	399	648	181	64	45	44	88	129	51	78	109	40	78	167	86
11	439	67	166	174	78	41	107	73	94	99	4	5	47	159	6
12	334	325	131	191	78	38	112	48	12	132	6	59	46	3	4
Average	**355**	**588**	**325**	**103**	**127**	**66**	**108**	**122**	**75**	**142**	**84**	**46**	**75**	**396**	**77**
std	**230**	**262**	**243**	**130**	**79**	**30**	**20**	**140**	**28**	**61**	**64**	**17**	**19**	**573**	**46**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	4	379	219	117	142	160	74	192	134	55	299	131	58	8832	682
2	8	886	199	148	169	167	44	184	132	103	125	141	118	8968	482
3	6	797	955	189	281	159	178	2305	272	247	81	124	14	94	527
4	612	736	749	148	599	138	479	218	111	554	115	94	74	62	693
5	564	648	230	124	288	151	289	205	107	194	114	122	82	223	534
6	34	732	127	67	155	74	238	15	110	170	188	100	113	52	5138
7	29	746	529	74	228	201	307	157	121	875	196	84	167	178	540
8	624	178	113	50	105	156	242	91	69	285	191	318	185	246	502
9	677	120	180	63	153	132	220	178	40	222	164	91	276	288	370
10	927	154	206	119	92	410	207	192	46	185	224	87	224	905	170
11	841	206	150	159	136	105	422	111	43	432	161	113	143	764	251
12	705	717	128	165	71	129	410	23	36	145	154	120	163	184	1129
Average	**419**	**525**	**315**	**119**	**202**	**165**	**259**	**323**	**102**	**289**	**168**	**127**	**135**	**1733**	**918**
std	**369**	**292**	**277**	**46**	**143**	**83**	**132**	**628**	**66**	**230**	**58**	**63**	**74**	**3358**	**1351**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	177	955	175	95	62	188	150	179	309	473	787	2979	103	49	15
2	657	965	122	111	255	259	123	143	309	419	395	2310	437	52	16
3	808	453	181	82	50	304	77	141	26	592	412	2443	257	62	129
4	643	280	173	62	203	36	87	11	59	528	24	2144	260	64	24
5	562	350	259	347	84	85	163	47	181	190	124	68	319	77	40
6	572	237	111	249	434	124	191	52	885	227	96	54	800	126	31
7	446	208	158	74	349	95	213	22	33	108	99	85	204	41	33
8	453	105	299	44	340	24	311	107	35	774	308	112	138	19	36
9	663	823	148	56	271	321	204	298	473	615	56	79	2885	30	34
10	803	66	222	48	126	329	317	442	426	521	63	171	104	31	20
11	498	548	144	49	417	552	280	461	552	268	1073	41	62	20	25
12	487	398	142	50	392	613	146	226	309	659	2033	110	59	9	32
Average	**564**	**449**	**178**	**106**	**249**	**244**	**189**	**177**	**300**	**448**	**456**	**883**	**469**	**48**	**36**
std	**173**	**313**	**56**	**95**	**142**	**192**	**81**	**154**	**259**	**208**	**593**	**1187**	**789**	**32**	**30**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	18	71	30	141	18	28	8	403	17	19	69	23	31	121	43
2	39	147	41	24	19	32	16	318	113	9	16	15	10	334	49
3	40	61	67	36	61	92	27	110	150	28	32	18	35	142	20
4	27	62	129	39	67	15	15	87	45	78	51	172	88	163	16
5	49	75	118	40	73	27	29	23	52	14	44	51	102	135	25
6	48	96	62	23	185	47	59	218	17	25	22	171	51	109	51
7	34	129	38	31	169	27	26	12	21	17	46	168	27	150	8
8	41	35	43	35	408	19	25	20	52	13	21	34	266	113	123
9	2130	88	59	78	318	9	24	41	16	1	17	39	217	109	217
10	1799	105	62	37	72	14	44	23	21	17	16	11	223	95	135
11	897	120	96	29	17	18	14	43	22	93	190	9	119	119	68
12	51	152	43	59	50	15	39	12	20	7	22	11	104	634	66
Average	**431**	**95**	**66**	**48**	**121**	**29**	**27**	**109**	**46**	**27**	**46**	**60**	**106**	**185**	**68**
std	**761**	**37**	**32**	**33**	**126**	**22**	**14**	**132**	**43**	**29**	**49**	**68**	**86**	**155**	**61**

Appendix I8: CONCENTRATOR COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Concentrator															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	20	34	25	6	15	87	10	13	15	14	3	31	79	87	156
2	18	83	37	4	26	92	34	12	11	22	6	33	52	22	23
3	27	19	42	4	39	36	12	14	16	31	74	20	21	129	28
4	25	15	21	5	41	17	27	11	19	30	24	17	21	24	25
5	20	18	47	6	65	16	21	20	19	19	34	18	57	21	23
6	36	13	44	6	86	16	17	11	21	12	42	21	24	22	24
7	32	14	72	11	102	22	17	15	19	33	51	19	19	34	35
8	31	27	64	38	53	20	16	13	26	28	39	9	17	34	34
9	34	33	35	40	16	21	14	13	21	12	40	8	19	38	41
10	36	32	32	59	16	19	13	13	15	17	37	17	25	35	42
11	31	20	28	2	10	16	23	13	15	26	35	27	17	34	9
12	40	15	18	13	15	15	20	12	14	68	48	34	7	6	7
Average	**29**	**27**	**39**	**16**	**40**	**31**	**19**	**13**	**18**	**26**	**36**	**21**	**30**	**41**	**37**
std	**7**	**19**	**16**	**19**	**31**	**28**	**7**	**2**	**4**	**15**	**19**	**9**	**21**	**34**	**39**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	7	19	46	26	78	39	390	90	62	27	28	11	19	724	49
2	27	18	3031	52	91	24	252	62	125	31	24	13	19	35	37
3	24	241	521	27	67	17	24	67	25	101	26	25	25	25	23
4	14	159	87	22	69	67	14	126	46	295	15	51	26	29	13
5	31	57	40	24	68	74	9	82	43	193	18	38	35	40	20
6	16	55	34	29	133	79	38	84	49	180	13	34	43	54	24
7	17	50	333	35	244	80	33	8	69	174	32	33	22	67	68
8	34	41	116	41	74	19	39	16	73	312	98	47	21	43	40
9	39	40	0	41	31	19	79	17	19	137	154	50	33	25	38
10	42	30	29	28	45	33	71	20	21	53	152	46	20	23	76
11	29	34	27	30	84	22	68	56	14	29	12	62	28	24	78
12	19	57	22	41	143	44	94	63	21	25	14	59	1319	585	30
Average	**25**	**67**	**357**	**33**	**94**	**43**	**93**	**58**	**47**	**130**	**49**	**39**	**134**	**140**	**41**
std	**11**	**66**	**856**	**9**	**57**	**25**	**114**	**36**	**32**	**103**	**54**	**17**	**373**	**243**	**22**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	37	33	21	35	45	20	38	22	35	67	32	30	106	43	18
2	35	58	19	37	49	45	34	30	47	57	27	40	23	52	19
3	29	36	50	34	48	66	32	30	37	78	37	953	11	53	36
4	28	34	35	37	52	38	32	53	34	58	51	190	7	30	30
5	27	30	24	40	34	30	54	61	109	62	44	160	16	22	37
6	60	25	23	45	39	34	117	28	40	35	24	46	13	19	42
7	62	25	39	53	38	36	82	456	38	37	25	21	22	21	113
8	60	23	20	24	84	49	79	31	59	30	24	18	16	19	65
9	41	25	32	194	33	51	10	2199	60	35	36	19	15	26	40
10	25	28	26	256	30	57	68	29	115	22	36	20	160	36	15
11	22	23	37	162	23	66	32	124	65	27	45	45	36	41	36
12	108	25	48	33	21	66	26	37	111	20	86	37	45	21	55
Average	**45**	**30**	**31**	**79**	**41**	**47**	**50**	**258**	**63**	**44**	**39**	**132**	**39**	**32**	**42**
std	**25**	**10**	**11**	**78**	**17**	**15**	**31**	**623**	**31**	**19**	**17**	**265**	**47**	**13**	**27**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	19	21	13	5	8	3	10	5	3	28	59	2	31	22	91
2	13	49	27	1	3	24	10	9	5	154	28	3	104	25	33
3	15	52	4	104	8	5	7	10	8	5	5	119	94	35	25
4	23	25	34	3	14	8	7	1	7	9	7	1	64	26	24
5	39	11	6	5	4	20	276	2	47	4	6	3	6	34	41
6	110	15	17	16	4	140	77	5	7	4	8	4	1	479	11
7	21	18	3	16	6	17	22	8	11	3	13	26	2	28	17
8	16	31	9	9	17	21	11	4	10	12	9	18	16	25	10
9	89	41	8	9	12	3	65	6	14	8	9	1	6	26	127
10	89	52	1070	6	18	133	19	9	7	9	23	3	2	22	11
11	58	56	30	33	13	4	36	5	99	10	2	6	127	14	8
12	12	33	4	19	18	10	11	1	91	7	7	6	14	16	5
Average	**42**	**34**	**102**	**19**	**10**	**32**	**46**	**5**	**26**	**21**	**15**	**16**	**39**	**63**	**34**
std	**35**	**16**	**305**	**28**	**6**	**49**	**76**	**3**	**34**	**42**	**16**	**33**	**46**	**131**	**38**

Appendix I9: CPV TANK COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

CPV tank															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	117	165	144	45	296	75	70	117	82	55	3	80	109	52	145
2	131	167	156	17	333	76	74	78	80	77	135	129	65	493	112
3	114	128	155	16	216	124	65	73	75	90	129	61	94	479	106
4	82	160	183	21	175	75	122	85	55	99	128	62	61	473	86
5	89	163	155	18	107	72	121	73	63	107	114	66	72	208	90
6	90	146	155	29	105	64	94	70	68	97	93	69	56	321	104
7	91	182	171	672	88	67	96	84	70	327	102	63	52	328	86
8	95	302	289	521	79	74	89	76	75	331	97	56	56	299	85
9	114	370	410	331	258	55	110	81	55	40	92	34	84	316	82
10	95	329	476	231	265	56	94	70	67	56	95	70	84	306	82
11	97	350	479	181	165	45	75	101	1148	57	98	30	71	434	308
12	82	256	778	115	1136	46	75	79	361	65	75	44	68	250	171
Average	**100**	**227**	**296**	**183**	**269**	**69**	**90**	**82**	**183**	**117**	**97**	**64**	**73**	**330**	**121**
std	**16**	**89**	**200**	**220**	**286**	**21**	**20**	**14**	**315**	**101**	**35**	**25**	**17**	**128**	**65**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	189	961	5562	2538	87	77	178	78	269	4206	604	1368	204	1202	143
2	202	251	5575	1188	29	105	179	112	291	909	261	1046	187	1361	121
3	255	271	5407	1317	229	150	150	80	268	384	882	1142	242	1514	127
4	15	277	19501	1519	226	138	191	85	312	341	1121	77	187	162	125
5	13	295	18667	1251	145	25	124	80	264	348	52	155	753	151	91
6	11	239	1524	1497	107	28	117	17761	2752	266	43	134	830	95	146
7	13	248	3130	1892	288	181	92	8791	2929	333	42	144	758	87	266
8	7	1259	2758	1248	58	141	98	550	4538	77	0	134	71	94	130
9	6	1192	968	1010	75	136	80	98	1148	77	42	218	145	75	245
10	6	895	2509	733	84	669	296	212	5033	107	146	1252	1560	81	126
11	7	330	4225	668	71	146	112	266	4419	126	961	218	1776	84	448
12	1151	3677	3305	88	71	183	90	128	4213	777	740	180	1545	77	643
Average	**156**	**825**	**6094**	**1246**	**123**	**165**	**142**	**2353**	**2203**	**663**	**408**	**506**	**688**	**415**	**218**
std	**327**	**983**	**6253**	**620**	**82**	**167**	**62**	**5450**	**1973**	**1146**	**423**	**521**	**627**	**574**	**167**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	1122	89	346	394	643	135	153	465	1337	969	443	37	242	41	51
2	151	97	258	422	508	127	95	325	171	125	430	35	248	117	3680
3	122	90	255	350	495	103	84	265	188	155	286	35	277	530	3590
4	58	472	339	347	448	94	90	288	171	103	235	73	269	415	3976
5	55	284	361	292	308	581	96	212	159	363	239	259	275	381	120
6	96	297	409	368	233	717	108	191	105	77	220	253	245	398	130
7	433	324	528	234	183	566	127	191	113	263	280	901	246	372	117
8	264	287	469	430	163	622	120	184	115	71	115	918	199	1236	54
9	260	276	358	493	151	870	684	268	115	78	52	1092	1023	57	48
10	129	249	255	164	1456	112	519	248	114	69	30	1496	358	48	55
11	96	300	2201	159	967	167	549	480	136	44	39	547	245	50	46
12	97	261	355	168	1168	230	518	452	128	235	38	567	35	60	64
Average	**240**	**252**	**511**	**318**	**560**	**360**	**262**	**297**	**238**	**213**	**201**	**518**	**305**	**309**	**994**
std	**298**	**112**	**539**	**114**	**427**	**287**	**230**	**110**	**347**	**257**	**147**	**489**	**238**	**345**	**1663**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	43	283	35	47	37	32	169	13	120	8807	11	28	21	151	399
2	40	187	104	50	38	30	13	24	68	57	6	45	60	119	66
3	25	56	100	101	34	28	21	27	37	140	7	28	59	95	58
4	20	30	53	58	62	38	24	16	58	326	9	26	11	145	74
5	17	27	44	64	69	24	8	24	36	95	23	23	71	217	51
6	17	29	51	147	81	27	34	63	25	33	28	17	36	122	42
7	64	40	64	40	72	25	19	34	37	56	41	31	37	112	26
8	31	26	48	35	78	19	18	27	28	23	4	51	34	142	12
9	167	44	57	63	84	60	21	9	20	24	5	63	115	1830	151
10	90	94	71	80	54	25	15	7	45	55	26	23	66	3887	101
11	114	109	52	42	41	141	9	33	135	17	19	23	59	3261	1836
12	112	32	95	41	47	119	11	23	8	21	16	39	134	1683	1041
Average	**62**	**80**	**65**	**64**	**58**	**47**	**30**	**25**	**51**	**805**	**16**	**33**	**59**	**980**	**321**
std	**49**	**80**	**23**	**32**	**19**	**40**	**44**	**15**	**39**	**2522**	**11**	**14**	**36**	**1367**	**558**

Appendix I10: CONDENSATE RETURN COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Condensate return															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	10	28	8	8	8	15	6	12	8	18	3	8	8	19	25
2	5	8	6	5	11	19	6	10	10	10	3	1	11	13	16
3	15	12	11	6	8	56	19	10	27	14	14	19	13	12	15
4	9	12	20	3	28	86	19	7	30	11	13	6	7	19	25
5	18	399	10	2	18	14	5	26	11	38	22	8	14	15	15
6	14	6	20	3	9	9	11	13	14	26	14	10	13	14	15
7	12	4	88	7	10	12	12	11	13	24	18	22	6	13	13
8	26	7	25	9	8	11	11	12	10	15	19	10	10	13	12
9	53	10	8	10	7	12	82	8	78	5	20	5	6	17	13
10	69	8	10	20	10	9	16	11	11	5	13	7	7	13	15
11	11	10	10	6	45	13	16	9	20	7	12	7	6	14	13
12	18	9	22	53	22	75	23	8	197	8	13	4	40	15	12
Average	**22**	**43**	**20**	**11**	**15**	**28**	**19**	**11**	**36**	**15**	**14**	**9**	**12**	**15**	**16**
std	**19**	**112**	**22**	**14**	**11**	**28**	**21**	**5**	**54**	**10**	**6**	**6**	**9**	**2**	**5**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	16	6	10	20	26	17	37	6	7	91	41	27	22	16	10
2	24	8	16	17	734	20	19	16	10	11	13	17	16	23	12
3	34	9	18	14	81	29	16	6	8	18	14	13	19	43	11
4	42	3	15	14	24	22	48	8	12	9	15	9	86	204	44
5	9	3	20	9	37	159	16	8	8	9	20	12	38	23	12
6	15	3	15	11	41	46	90	6	9	11	14	21	30	38	125
7	42	2	15	9	35	36	20	51	13	9	18	22	18	24	101
8	34	1	23	456	21	28	10	18	12	11	63	24	16	9	686
9	14	2	14	27	20	21	16	105	24	33	208	20	14	13	26
10	14	15	13	45	29	28	5	9	642	100	31	19	12	9	232
11	11	16	14	43	27	39	16	10	244	106	12	21	10	11	19
12	9	14	16	41	23	37	70	10	33	19	18	18	11	10	20
Average	**22**	**7**	**16**	**59**	**92**	**40**	**30**	**21**	**85**	**36**	**39**	**19**	**24**	**35**	**108**
std	**13**	**6**	**3**	**126**	**203**	**38**	**26**	**29**	**188**	**39**	**55**	**5**	**21**	**54**	**194**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	16	54	19	14	16	19	18	20	100	42	26	19	24	29	24
2	30	20	24	17	15	25	797	21	17	21	248	18	22	37	33
3	28	17	22	15	25	40	177	23	21	20	23	65	22	71	34
4	31	17	21	35	15	38	80	30	44	27	25	20	34	46	23
5	63	10	18	27	50	37	22	21	20	23	19	23	49	32	19
6	73	11	24	14	26	67	19	22	14	22	18	26	19	37	31
7	41	8	16	21	30	22	31	16	119	15	19	28	22	27	52
8	43	9	7	78	34	283	23	20	278	42	19	34	36	28	62
9	20	12	12	47	25	18	20	14	34	18	26	30	33	30	29
10	21	21	12	23	21	20	24	18	23	20	32	23	77	39	23
11	15	22	11	18	19	14	20	19	32	29	21	23	32	32	29
12	14	19	12	16	18	13	19	15	25	19	22	26	14	24	40
Average	**33**	**18**	**17**	**27**	**25**	**50**	**104**	**20**	**61**	**25**	**42**	**28**	**32**	**36**	**33**
std	**19**	**12**	**6**	**19**	**10**	**75**	**223**	**4**	**76**	**9**	**65**	**13**	**17**	**13**	**13**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	19	107	21	73	26	35	19	11	20	35	9	11	4	2	6
2	18	97	26	25	25	16	45	17	6	10	4	14	7	8	5
3	25	82	22	30	23	35	17	7	13	12	30	2	17	8	12
4	20	39	19	24	23	23	23	9	29	14	27	2	3	5	5
5	18	54	18	26	24	31	26	21	26	13	6	10	7	13	5
6	18	44	11	44	22	38	15	24	7	149	8	2	6	27	10
7	14	46	22	24	27	25	6	9	8	22	4	1	4	4	15
8	36	25	19	21	19	19	6	25	9	23	6	420	26	2	23
9	14	100	27	46	18	25	5	8	8	13	4	4	43	10	25
10	20	63	27	22	30	17	5	10	13	13	129	18	7	4	3
11	38	27	32	14	18	19	6	7	9	9	20	4	5	6	22
12	46	44	79	12	114	14	39	8	25	5	14	6	7	5	5
Average	**24**	**61**	**27**	**30**	**31**	**25**	**18**	**13**	**14**	**27**	**22**	**41**	**11**	**8**	**11**
std	**10**	**29**	**17**	**17**	**26**	**8**	**14**	**7**	**8**	**39**	**35**	**119**	**12**	**7**	**8**

Appendix I11: HOT WATER COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

Hot water															
	Number of days														
Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	94	57	225	62	331	82	18	134	66	44	140	76	159	1075	163
2	52	62	149	31	254	81	19	92	76	207	120	74	74	920	226
3	60	63	213	24	182	97	21	94	41	266	110	220	99	850	120
4	49	894	178	16	123	74	42	93	61	170	152	227	70	528	99
5	50	69	193	192	105	70	64	110	101	162	113	76	79	554	105
6	49	230	310	160	72	50	57	130	115	95	118	65	64	495	105
7	53	216	274	141	59	44	57	115	57	187	134	52	54	405	102
8	69	336	481	186	65	53	62	104	51	166	122	50	68	409	85
9	60	384	534	219	4	52	91	76	94	65	91	77	75	406	87
10	46	408	742	137	19	53	97	70	6	78	92	83	75	421	25
11	67	459	540	307	13	48	77	67	10	99	126	46	64	347	58
12	49	452	141	303	11	50	93	8	6	132	9	46	3	420	565
Average	**58**	**303**	**332**	**148**	**103**	**63**	**58**	**91**	**57**	**139**	**111**	**91**	**74**	**569**	**145**
std	**14**	**245**	**195**	**101**	**104**	**17**	**29**	**34**	**37**	**65**	**37**	**63**	**35**	**241**	**141**
Sample	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	349	46	889	142	62	151	337	2415	288	67	484	75	37	89	36
2	717	6426	1004	122	64	160	4329	2384	241	75	414	739	40	144	287
3	375	2814	420	107	18	151	1852	2390	4	72	326	834	38	738	82
4	148	3031	1010	91	23	104	1553	1375	36	207	255	857	71	685	72
5	96	4074	238	65	64	108	1425	169	50	868	158	933	54	63	53
6	78	6477	181	60	33	42	936	213	97	346	138	873	52	49	57
7	81	6013	272	20	33	57	417	643	344	978	143	1349	108	46	48
8	98	2370	35	4	131	36	410	647	881	791	96	127	105	44	48
9	68	867	32	77	191	34	2902	1206	754	252	50	104	103	83	52
10	552	1316	31	72	145	75	2882	395	1055	1348	225	95	102	75	55
11	653	2516	24	70	2089	82	2590	376	81	571	345	101	117	84	56
12	358	3745	70	50	145	71	396	299	72	570	557	54	81	51	52
Average	**298**	**3308**	**351**	**73**	**250**	**89**	**1669**	**1043**	**325**	**512**	**266**	**512**	**76**	**179**	**75**
std	**239**	**2137**	**392**	**39**	**582**	**46**	**1283**	**895**	**366**	**415**	**161**	**461**	**31**	**250**	**68**
Sample	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**	**41**	**42**	**43**	**44**	**45**
1	303	369	131	88	3325	161	198	54	109	471	265	403	233	16	32
2	306	349	86	84	122	237	145	22	37	465	262	35	254	113	33
3	270	205	120	521	77	86	138	50	33	304	254	33	221	34	21
4	284	165	113	509	93	227	148	50	477	140	69	37	112	40	23
5	311	189	102	57	148	298	154	55	37	304	250	61	71	33	94
6	98	178	105	63	155	461	138	140	30	336	232	4808	52	188	17
7	84	354	107	45	148	478	138	227	27	51	10	4779	36	34	275
8	91	462	1132	35	144	658	143	219	33	50	32	156	20	31	27
9	622	120	1246	38	183	197	53	57	26	41	34	1468	15	23	24
10	1792	136	1071	38	206	198	276	43	285	45	48	2124	16	23	2
11	382	108	78	116	268	202	182	43	327	61	33	164	13	20	56
12	406	97	75	40	181	212	48	415	227	47	392	281	17	35	65
Average	**412**	**228**	**364**	**136**	**421**	**285**	**147**	**115**	**137**	**193**	**157**	**1196**	**88**	**49**	**56**
std	**460**	**123**	**476**	**179**	**916**	**164**	**60**	**118**	**154**	**171**	**131**	**1803**	**94**	**50**	**73**
Sample	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**60**
1	65	25	15	55	16	25	44	24	10	8	44	278	6	168	130
2	56	61	17	99	6	16	14	24	21	15	61	61	20	61	32
3	34	47	72	70	7	14	8	20	9	12	95	123	6	32	11
4	94	43	14	31	19	29	33	129	35	19	52	94	58	11	103
5	26	43	38	27	12	44	19	97	33	15	35	115	56	389	638
6	24	21	62	2	15	14	37	20	7	25	24	6831	88	511	89
7	38	20	29	9	24	8	50	14	1	33	55	1617	1020	304	200
8	89	99	26	144	23	16	59	32	12	25	53	1560	7976	297	80
9	92	49	21	30	41	25	97	14	11	9	558	5739	77	197	70
10	66	50	7	29	12	16	21	25	8	18	202	33	108	181	92
11	48	46	36	23	11	14	24	20	24	33	136	100	71	116	55
12	23	31	83	8	16	29	33	40	23	8	168	60	208	142	769
Average	**55**	**45**	**35**	**44**	**17**	**21**	**37**	**38**	**16**	**18**	**124**	**1384**	**808**	**201**	**189**
std	**27**	**21**	**25**	**42**	**9**	**10**	**24**	**36**	**11**	**9**	**148**	**2370**	**2274**	**150**	**247**

Appendix J: ONE-TAIL T-TEST CALCULATIONS OF THE COD CONCENTRATIONS DATA COLLECTED BY THE WCM LABORATORY FROM 6 MAY 2017 TO 30 JULY 2017

<u>One-tail t-test calculations for the WCM macro effluent channels:</u>

Number of samples (n) =6
t-critical at 95% confidence intervals at 59 (n-1) number of the degrees of freedom, the t-critical value from the t-table is 1.671.

<u>The tests claims are:</u>
Null hypothesis – H_0: µ COD concentrations ≤ 5000 ppm.
Alternate hypothesis - H1: µ COD concentrations > 5000 ppm.

The null hypothesis is accepted for the eight effluent channels, namely Anion, Cation, Liquefaction, Evaporator, Concentrator, CPV Tank, Condensate Return and Hot Water. The null hypothesis is rejected and the alternate is favoured for the three effluent channels namely, Condensate, Glucose and Effluent Tank.

Anion	**Cation**	**Glucose**
µ = 5000 ppm	µ = 5000 ppm	µ= 5000 ppm
Mean = 4130 ppm	Mean = 4417 ppm	Mean = 8928 ppm
S= 5101	S= 5495	s = 6067
$t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$	$t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$	$t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$
$t = \frac{4130 - 5000}{5101/\sqrt{60}}$	$t = \frac{4417 - 5000}{5495/\sqrt{60}}$	$t = \frac{8928 - 5000}{6067/\sqrt{60}}$
$t = \frac{-870}{658.5362683}$	$t = \frac{-583}{709.4014496}$	$t = \frac{3928}{783.246332}$
***t* = -1.3211**	***t* = -0.8218**	**t = 5.0150**
t-statistical < t-critical; therefore, accept the null hypothesis.	t-statistical < t-critical; therefore, accept the null hypothesis.	t-statistical falls outside the accepted criteria, therefore, reject the null hypothesis and accept the alternate hypothesis.

Effluent Tank μ = 5000 ppm Mean = 8536 ppm S = 3685 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{8536 - 5000}{3685/\sqrt{60}}$ $t = \frac{3536}{475.7314544}$ ***t* = 7.4328** t-statistical> t-critical; therefore, reject null hypothesis and accept the alternate hypothesis.	**Liquefaction** μ = 5000 ppm Mean = 817 ppm s = 912 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{817 - 5000}{912/\sqrt{60}}$ $t = \frac{-4183}{117.7386937}$ **t = -35.5278** t-statistical> t-critical; therefore, reject null hypothesis and accept the null hypothesis.	**Condensate** μ = 5000 ppm Mean = 14 757 ppm S = 4492 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{14757 - 5000}{4492/\sqrt{60}}$ $t = \frac{9757}{579.9147064}$ **t = 16.8249** t-statistical > t-critical; therefore, reject the null hypothesis and accept the alternate hypothesis.	**Evaporator** μ = 5000 ppm Mean = 817 ppm S = 251 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{817 - 5000}{251/\sqrt{60}}$ $t = \frac{-4183}{32.40396066}$ ***t* = -129.0892** t-statistical < t-critical; therefore, accept the null hypothesis.
Concentrator μ = 5000 ppm Mean = 54 ppm S = 73 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{54 - 5000}{73/\sqrt{60}}$ $t = \frac{-4946}{9.424259476}$ ***t* = -524.8158** t-statistical< t-critical; therefore, accept the null hypothesis.	**CPV Tank** μ = 5000 ppm Mean = 391 ppm S = 381 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{391 - 5000}{381/\sqrt{60}}$ $t = \frac{-4609}{49.1868885}$ ***t* = -93.7038** t-statistical < t-critical; therefore, accept the null hypothesis.	**Condensate Return** μ = 5000 ppm Mean = 3 1ppm s = 38 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{31 - 5000}{38/\sqrt{60}}$ $t = \frac{-4969}{4.905778905}$ ***t* = -1012.8871** t-statistical < t-critical; therefore, accept the null hypothesis.	**Hot Water** μ = 5000 ppm Mean = 297 ppm S = 286 $t = \frac{\bar{x} - \mu}{s/\sqrt{n}}$ $t = \frac{297 - 5000}{286/\sqrt{60}}$ $t = \frac{-4703}{36.92244123}$ ***t* = - 127.3751** t-statistical < t-critical; therefore, accept the null hypothesis.

Appendix K1: TRIAL ONE COD CONCENTRATIONS IMPROVEMENT DATA COLLECTED ON THE 28 AUGUST 2018 AND 10 SEPTEMBER 2018

TRIAL ONE – BATCH 1

Condensate

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9740			6090		
1	4,21	4,13	9720	6,65	5,02	5960	20	20
Average			9730	COD average		6025		
Std			14	Std		92		
COD Range (Raw v.s filtered)			3705					

Effluent tank

BEFORE				AFTER					
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Sample No**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			6800				4060		
1	5,94	6,6	6890	3	8,1	7,41	3996	20	20
Average			6845	COD average			4028		
Std			64	Std			45		
COD Range (Raw v.s filtered)			2817						

Glucose

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			5440		
1	4,68	2,75	8030	7,18	4,71	5510	20	20
Average			8145	COD average		5475		
Std			163	Std		49		
COD Range (Raw v.s filtered)			2670					

Stats	Raw Effluent	Filtered Effluent
Grand average	7801	4603
Grand Std	104	67
Grand Range		3198

Condensate

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9310			5510		
1	6,08	5,38	9930	8,88	6,4	5190	30	20
Average			9620	COD average		5350		
Std			438	Std		226		
COD Range (Raw v.s filtered)			4270					

Glucose

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			5030		
1	4,68	2,75	8030	7,84	4,87	4980	30	20
Average			8145	COD average		5005		
Std			163	Std		35		
COD Range (Raw v.s filtered)			3140					

Effluent tank

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7740			4950		
1	4,68	2,75	7430	7,81	4,89	4870	30	20
Average			7585	COD average		4910		
Std			219	Std		57		
COD Range (Raw v.s filtered)			2675					
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8785			4650		
1	4,21	6,26	8523	7,85	8,21	4540	60	20
Average			8654	COD average		4595		

Std			185	Std			78	
COD Range (Raw v.s filtered)			4059					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			4090		
1	4,68	2,75	8030	7,92	5,54	4020	60	20
Average			8145	COD average			4055	
Std			163	Std			49	
COD Range (Raw v.s filtered)			4090					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7930			4930		
1	6,30	2,75	7920	7,94	5,5	4960	60	20
Average			7925	COD average			4945	
Std			7	Std			21	
COD Range (Raw v.s filtered)			2980					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							8262	4606
Grand Std							98	83
Grand Range								3656

TRIAL ONE – BATCH 2								
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9740			5320		
2	4,21	4,13	9720	7,58	5,39	4990	20	30
Average			9730	COD average			5155	
Std			14	Std			233	
COD Range (Raw v.s filtered)			4575					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7740			4500		
2	6,61	9,91	7430	7,31	10,98	4390	20	30
Average			7585	COD average			4445	
Std			219	Std			78	
COD Range (Raw v.s filtered)			3140					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			4740		
2	4,68	2,75	8030	7,83	5,19	4691	20	30
Average			8145	COD average			4716	
Std			163	Std			35	
COD Range (Raw v.s filtered)			3430					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							7616	3994
Grand Std							66	108
Grand Range								3616
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9740			4720		
2	4,21	4,13	9720	8,03	5,72	4350	30	30
Average			9730	COD average			4535	
Std			14	Std			262	
COD Range (Raw v.s filtered)			5195					
Effluent tank								

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7920			4920		
2	5,94	6,6	7930	7,65	11,28	4920	30	30
Average			7925	COD average			4920	
Std			7	Std			0	
COD Range (Raw v.s filtered)			3005					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			4610		
2	4,68	2,75	8030	8,07	5,15	4590	30	30
Average			8145	COD average			4600	
Std			163	Std			14	
COD Range (Raw v.s filtered)			3545					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							8076	4281
Grand Std							115	87
Grand Range								3795

Condensate								
BEFORE				**AFTER**				
Raw effluent subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8300			3040		
2	4,67	5,65	8350	8,67	8,53	3090	60	30
Average			8325	COD average			3065	
Std			35	Std			35	
COD Range (Raw v.s filtered)			5260					
Effluent tank								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			5730			910		
2	3,89	4,42	5760	8,23	6,18	970	60	30
Average			5745	COD average			940	
Std			21	Std			42	
COD Range (Raw v.s filtered)			4805					
Glucose								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
Average	7925		7930			2070		
Std	7	2,75	7920	8,73	6,4	2010	60	30
COD Range (Raw v.s filtered)			5885	COD average			2040	
Stats			**Raw Effluent**	Std			42	
Grand average			7535					
Grand Std							57	**Filtered Effluent**
Grand Range							5331	2204

TRIAL ONE – BATCH 3								
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9740			4620		
3	4,21	4,13	9720	8,18	5,7	4593	20	40
Average			9730	COD average		4607		
Std			14	Std		19		
COD Range (Raw v.s filtered)			5124					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			6800			2860		
3	6,61	9,91	6890	8,3	11,48	2890	20	40
Average			6845	COD average		2875		
Std			64	Std		21		
COD Range (Raw v.s filtered)			3970					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8260			4170		
3	4,68	2,75	8030	8,26	5,44	4290	20	40
Average			8145	COD average		4230		
Std			163	Std		85		
COD Range (Raw v.s filtered)			3915					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	7613	3451
Grand Std	66	38
Grand Range		4162

Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9740			4180		
3	4,21	4,13	9720	8,4	5,96	3840	30	40
Average			9730	COD average		4010		
Std			14	Std		240		
COD Range (Raw v.s filtered)			5720					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7930			4110		
3	4,68	2,75	7920	8,55	5,6	4170	30	40
Average			7925	COD average		4140		
Std			7	Std		42		
COD Range (Raw v.s filtered)			3785					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8010			4040		
3	4,68	2,75	8070	8,63	5,7	4080	30	40
Average			8040	COD average		4060		
Std			42	Std		28		
COD Range (Raw v.s filtered)			3980					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	8460	4028
Grand Std	57	82
Grand Range		4432

Condensate

BEFORE				AFTER				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
3	4,2	6,26	8785	8,3	8,76	4240	60	40
			8523			4510		
Average			8654	COD average			4375	
Std			185	Std			191	
COD Range (Raw v.s filtered)			4279					
Glucose								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
3	4,68	2,75	8260	8,39	5,86	4000	60	40
			8030			3950		
Average			8145	COD average			3975	
Std			163	Std			35	
COD Range (Raw v.s filtered)			4170					
Effluent tank								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
3	4,68	2,75	7930	8,42	5,88	3390	60	40
			7920			3320		
Average			7925	COD average			3355	
Std			7	Std			49	
COD Range (Raw v.s filtered)			4570					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	8262	3858
Grand Std	98	81
Grand Range		4155

Appendix K2: TRIAL TWO COD CONCENTRATIONS IMPROVEMENT DATA COLLECTED ON THE 30 SEPTEMBER 2018 AND 29 OCTOBER 2018

TRIAL 2 – BATCH 1								
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
1	12,39	7,62	7570 7530	11,77	8,32	6280 6180	20	20
Average			7550	COD average			6230	
Std			28	Std			71	
COD Range (Raw v.s filtered)			1320					
Effluent tank								
BEFORE			**AFTER**					
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
1	12	7,62	7990 7980	11,83	8,28	4740 4720	20	20
Average			7985	COD average			4730	
Std			7	Std			14	
COD Range (Raw v.s filtered)			3255					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
1	13	7,62	7740 7793	11,81	8,3	4970 4930	20	20
Average			7767	COD average			4950	
Std			37	Std			28	
COD Range (Raw v.s filtered)			2817					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							7879	5353
Grand Std							48	46
Grand Range								2526

Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
1	13,1	7,62	8130 8300	11,8	8,38	4800 4840	30	20
Average			8215	COD average			4820	
Std			120	Std			28	
COD Range (Raw v.s filtered)			3395					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
1	12,39	7,62	7990 7980	11,73	8,35	4850 4840	30	20
Average			7985	COD average			4845	
Std			7	Std			7	
COD Range (Raw v.s filtered)			3140					
Glucose								
BEFORE				**AFTER**				

Batch No	pH	Conductivity (mS/cm)	COD (ppm)	pH	Conductivity (mS/cm)	COD (ppm)	Time (minutes)	GAC mass (g)
			7930			4790		
1	13,04	7,62	7740	11,82	8,41	4560	30	20
Average			7835	COD average		4675		
Std			134	Std		163		
COD Range (Raw v.s filtered)			3160					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							7896	5171
Grand Std							72	76
Grand Range								2725
Condensate								
BEFORE				**AFTER**				
Raw effluent subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8040			5100		
1	13,1	15,74	8400	11,87	12,77	5110	60	20
Average			8220	COD average		5105		
Std			255	Std		7		
COD Range (Raw v.s filtered)			3115					
Effluent tank								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8130			4940		
1	12,39	7,62	8300	11,68	8,43	5000	60	20
Average			8215	COD average		4970		
Std			120	Std		42		
COD Range (Raw v.s filtered)			3245					
Glucose								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7990			4890		
1	13,4	7,62	7980	11,7	8,38	4920	60	20
Average			7985	COD average		4905		
Std			7	Std		21		
COD Range (Raw v.s filtered)			3080					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							8050	4545
Grand Std							103	152
Grand Range								3505

TRIAL 2 – BATCH 2								
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8130			5219		
2	13,11	25,6	8300	12,68	20,2	5180	20	30
Average			8215	COD average			5199,5	
Std			120	Std			28	
COD Range (Raw v.s filtered)			3015,5					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8130			4630		
2	12,39	7,62	8300	11,64	8,53	4680	20	30
Average			8215	COD average			4655	
Std			120	Std			35	
COD Range (Raw v.s filtered)			3560					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7990			4740		
2	13,04	7,62	7980	11,6	8,5	4740	20	30
Average			7985	COD average			4740	
Std			7	Std			0	
COD Range (Raw v.s filtered)			3245					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							7879	4883
Grand Std							48	28
Grand Range								2996

Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8130			4330		
2	13,1	7,62	8300	11,45	8,65	4320	30	30
Average			8215	COD average			4325	
Std			120	Std			7	
COD Range (Raw v.s filtered)			3890					
Effluent tank								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7990			4310		
2	12,39	7,62	7980	11,5	8,7	4370	30	30
Average			7985	COD average			4340	
Std			7	Std			42	
COD Range (Raw v.s filtered)			3645					
Glucose								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7930			4270		
2	13,04	7,62	7740	11,4	8,67	4280	30	30

Average			7835	COD average			4275	
Std			134	Std			7	
COD Range (Raw v.s filtered)			3560					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							7896	4661
Grand Std							72	37
Grand Range								3235
Condensate								
BEFORE				**AFTER**				
Raw effluent subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9310			5180		
2	8,2	5,38	9930	9,09	9,43	5400	60	30
Average			9620	COD average			5290	
Std			438	Std			156	
COD Range (Raw v.s filtered)			4330					
Effluent tank								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8040			4520		
2	12,48	15,74	8400	11,63	12,73	4440	60	30
Average			8220	COD average			4480	
Std			255	Std			57	
COD Range (Raw v.s filtered)			3740					
Glucose								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7801			3500		
2	13,05	15,74	7760	11,36	13,41	4040	60	30
Average			7781	COD average			3770	
Std			29	Std			382	
COD Range (Raw v.s filtered)			4010,5					
Stats							**Raw Effluent**	**Filtered Effluent**
Grand average							8402	4596
Grand Std							182	156
Grand Range								3806

TRIAL 2 BATCH 3								
Condensate								
BEFORE				**AFTER**				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			9310			5900		
3	8,2	8,74	9930	9,26	7,6	5987	20	40
Average			9620	COD average			5944	

Std			438	Std			62	
COD Range (Raw v.s filtered)			3677					

Effluent tank

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8130			4000		
3	12,39	7,62	8300	11,49	8,71	4100	20	40
Average			8215	COD average			4050	
Std			120	Std			71	
COD Range (Raw v.s filtered)			4165					

Glucose

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7990			4340		
3	13,04	7,62	7980	11,49	8,69	4440	20	40
Average			7985	COD average			4390	
Std			7	Std			71	
COD Range (Raw v.s filtered)			3595					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	7879	4121
Grand Std	48	285
Grand Range		3758

Condensate

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			8040			3800		
3	13,1	15,74	8040	11,87	12,77	4220	30	40
Average			8040	COD average			4010	
Std			0	Std			297	
COD Range (Raw v.s filtered)			4030					

Effluent tank

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
	12,3		8130			4340		
3	9	7,62	8300	11,68	8,43	4300	30	40
Average			8215	COD average			4320	
Std			120	Std			28	
COD Range (Raw v.s filtered)			3895					

Glucose

BEFORE				AFTER				
Batch No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
	13,0		7930			4100		
3	4	7,62	7740	11,65	8,38	4130	30	40
Average			7835	COD average			4115	
Std			134	Std			21	
COD Range (Raw v.s filtered)			3720					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	8019	4294
Grand Std	65	92
Grand Range		3725

Condensate

BEFORE				AFTER				
Raw effluent subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
			7570			2850		
1	13,1	25,6	7530	12,64	18,25	2845	60	40
Average			7550	COD average			2848	
Std			28	Std			4	
COD Range (Raw v.s filtered)			4702,5					

Effluent tank

BEFORE				AFTER				
Raw effluent	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**

Subgroup No								
3	12,39	7,62	7740	11,21	8,5	3450	60	40
			7930			3450		
Average			7835	COD average		3450		
Std			134	Std		0		
COD Range (Raw v.s filtered)			4385					
Glucose								
BEFORE				**AFTER**				
Raw effluent Subgroup No	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**pH**	**Conductivity (mS/cm)**	**COD (ppm)**	**Time (minutes)**	**GAC mass (g)**
4	13,8	7,62	7990	11,2	8,54	3580	60	40
			7980			3510		
Average			7985	COD average		3545		
Std			7	Std		49		
COD Range (Raw v.s filtered)			4440					

Stats	**Raw Effluent**	**Filtered Effluent**
Grand average	7896	3542
Grand Std	72	57
Grand Range		4354

Appendix L1: PAIRED T-TEST TRIAL ONE CALCULATIONS, BEFORE AND AFTER TREATMENT

Paired t-test for the mean difference conducted on trial 1 batch 1: COD comparison before and after the treatment:

Sample No.	COD (ppm) Before Treatment - Acidic samples	COD (ppm) After Treatment - Acidic samples
1	9740	6090
2	9720	5960
3	6800	4060
4	6890	3996
5	8260	5440
6	8030	5510
7	9310	5510
8	9930	5190
9	8260	5030
10	8030	4980
11	7740	4950
12	7430	4870
13	8785	4650
14	8523	4540
15	8260	4090
16	8030	4020
17	7930	4930
18	7920	4960

Test claim:

H_0: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}} - \bar{X}_{\text{COD after treatment}}$)

H_1: $\bar{X}_{\text{Difference}} \neq 0$

		Trial One - Batch 1				
Sample No.	COD Before Treatment	COD After Treatment	Difference	Diff - Mean Diff	(Diff - Mean Diff) Power2	
1	9740	6090	3650	3650	13322500	
2	9720	5960	3760	3760	14137600	
3	6800	4060	2740	2740	7507600	
4	6890	3996	2894	2894	8375236	
5	8260	5440	2820	2820	7952400	
6	8030	5510	2520	2520	6350400	
7	9310	5510	3800	3800	14440000	
8	9930	5190	4740	4740	22467600	
9	8260	5030	3230	3230	10432900	
10	8030	4980	3050	3050	9302500	
11	7740	4950	2790	2790	7784100	
12	7430	4870	2560	2560	6553600	
13	8785	4650	4135	4135	17098225	
14	8523	4540	3983	3983	15864289	
15	8260	4090	4170	4170	17388900	
16	8030	4020	4010	4010	16080100	
17	7930	4930	3000	3000	9000000	
18	7920	4960	2960	2960	8761600	
		Mean Diff	3378		212819550	= Sum Variance
	Under root	12518797,06				
	SD =	3538,191213				
	sqrt3	4,242640687				

			Test stat = AveDiff/(SD/sqrtn		At 95% confidence in two tail (0.025) and df n-1 (17)		
		SD calculation	Test stat =	4,051089		Critical values 2.1098 and -2.1098	

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025(α/2) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.0511) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 1 batch 1 is rejected.

<u>Paired t-test for the mean difference conducted on trial 1 batch 2: COD comparison before and after the treatment:</u>

Sample No.	COD (ppm) Before Treatment - Acidic samples	COD (ppm) After Treatment - Acidic samples
1	9740	5320
2	9720	4990
3	7740	4500
4	7430	4390
5	8260	4740
6	8030	4691
7	9740	4720
8	9720	4350
9	7920	4920
10	7930	4920
11	8260	4610
12	8030	4590
13	8300	3040
14	8350	3090
15	5730	910
16	5760	970
17	7930	2070
18	7920	2010

Test claim:

H_0: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}}$ - $\bar{X}_{\text{COD after treatment}}$)

H_1: $\bar{X}_{\text{Difference}} \neq 0$

Trial One - Batch 2						
Sample No.	COD Before Treatment	COD After Treatment	Difference	Diff - Mean Diff	(Diff -Mean Diff)Power2	
1	9740	5320	4420	4420	19536400	
2	9720	4990	4730	4730	22372900	
3	7740	4500	3240	3240	10497600	
4	7430	4390	3040	3040	9241600	
5	8260	4740	3520	3520	12390400	
6	8030	4691	3339	3339	11148921	
7	9740	4720	5020	5020	25200400	
8	9720	4350	5370	5370	28836900	
9	7920	4920	3000	3000	9000000	
10	7930	4920	3010	3010	9060100	
11	8260	4610	3650	3650	13322500	
12	8030	4590	3440	3440	11833600	
13	8300	3040	5260	5260	27667600	
14	8350	3090	5260	5260	27667600	
15	5730	910	4820	4820	23232400	
16	5760	970	4790	4790	22944100	
17	7930	2070	5860	5860	34339600	
18	7920	2010	5910	5910	34928100	
		Mean Diff	4316		353220721	= Sum Variance
	Under root	20777689,47				
	SD =	4558,255091				
	sqrt3	4,242640687				

	Test stat = AveDiff/(SD/sqrtn			At 95% confidence in two tail (0.025) and df n-1 (17)
SD Calculation	Test stat =	4,016694		Critical values 2.1098 and -2.1098

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025(α/2) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.0167) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 1 batch 2 is rejected.

Paired t-test for the mean difference on trial 1 batch 3: COD comparison before and after the treatment:

Sample No.	COD (ppm) Before Treatment - Acidic samples	COD (ppm) After Treatment - Acidic samples
1	9740	4620
2	9720	4593
3	6800	2860
4	6890	2890
5	8260	4170
6	8030	4290
7	9740	4180
8	9720	3840
9	7930	4110
10	7920	4170
11	8010	4040
12	8070	4080
13	8785	4240
14	8523	4510
15	8260	4000
16	8030	3950
17	7930	3390

18	7925	3320

Test claim:

H_0: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}}$ - $\bar{X}_{\text{COD after treatment}}$)

H_1: $\bar{X}_{\text{Difference}} \neq 0$

			Test stat = AveDiff/(SD/sqrtn	At 95% confidence in two tail (0.025) and df n-1 (17)	
		SD Calculation	Test stat = 4,082418	Critical values 2.1098 and -2.1098	

Trial One - Batch 3						
Sample No.	**COD Before Treatment**	**COD After Treatment**	**Difference**	**Diff - Mean Diff**	**(Diff -Mean Diff)Power2**	
1	9740	4620	5120	5120	26214400	
2	9720	4593	5127	5127	26286129	
3	6800	2860	3940	3940	15523600	
4	6890	2890	4000	4000	16000000	
5	8260	4170	4090	4090	16728100	
6	8030	4290	3740	3740	13987600	
7	9740	4180	5560	5560	30913600	
8	9720	3840	5880	5880	34574400	
9	7930	4110	3820	3820	14592400	
10	7920	4170	3750	3750	14062500	
11	8010	4040	3970	3970	15760900	
12	8070	4080	3990	3990	15920100	
13	8785	4240	4545	4545	20657025	
14	8523	4510	4013	4013	16104169	
15	8260	4000	4260	4260	18147600	
16	8030	3950	4080	4080	16646400	
17	7930	3390	4540	4540	20611600	
18	7925	3320	4605	4605	21206025	
		Mean Diff	4391		353936548	**= Sum Variance**
	Under root	20819796,94				
	SD =	4562,871567				
	sqrt3	4,242640687				

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025($\alpha/2$) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.0824) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 1 batch 3 is rejected.

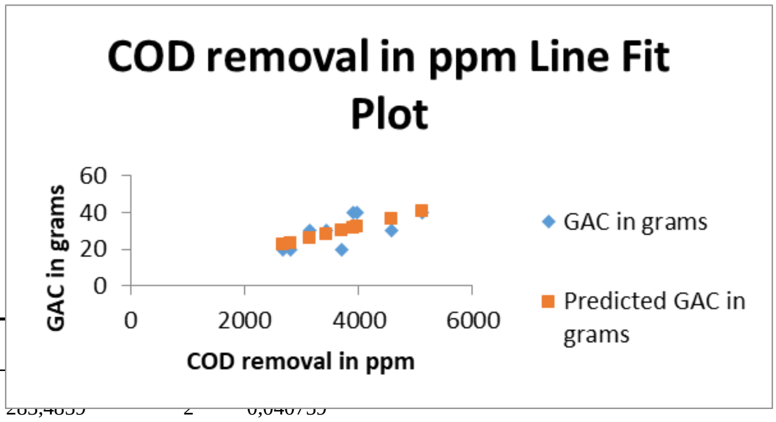
Appendix L2: TRIAL ONE CORRELATION STUDY ON IMPROVEMENT DATA – USING THE DATA COLLECTED ON THE 28 AUGUST 2018

Correlation of 20 minutes: 20g, 30g, and 40g:

CGAC in grams	COD removal in ppm
20	3705
20	2817
20	2670
30	4575
30	3140
30	3430
40	5124
40	3970
40	3915

SUMMARY OUTPUT

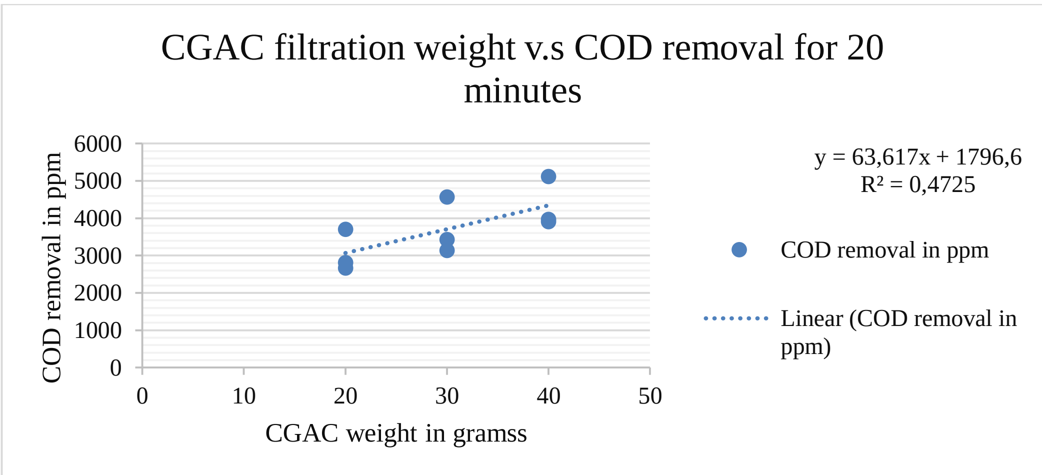
Regression Statistics	
Multiple R	0,687369
R Square	0,472477
Adjusted R Square	0,397116
Standard Error	6,724306
Observations	9



ANOVA

	df	*SS*	[illegible]	[illegible]	[illegible]
Regression	1	283,4859	[illegible]	[illegible]	[illegible]
Residual	7	316,5141	45,2163		
Total	8	600			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2,482399	11,21611	0,221324	0,831157	-24,0395	29,00429	-24,0395	29,00429
COD removal in ppm	0,007427	0,002966	2,503907	0,040759	0,000413	0,014441	0,000413	0,014441



r^2 of 0.4725 and r of 0.3971 indicates a weak positive correlation.

F- ANOVA of 6. 2696 indicates a small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.3971; which indicates that 39.71% of the total variation in the COD concentrations can be explained by the linear relationship. However, 60.29% of the variation cannot be explained because of other independent factors.

Correlation of 30 minutes: 20g, 30g, and 40g:

CGAC in grams	COD removal in ppm
20	4270
20	2675
20	3140
30	5195
30	3545
30	3005
40	5720
40	3980
40	3785

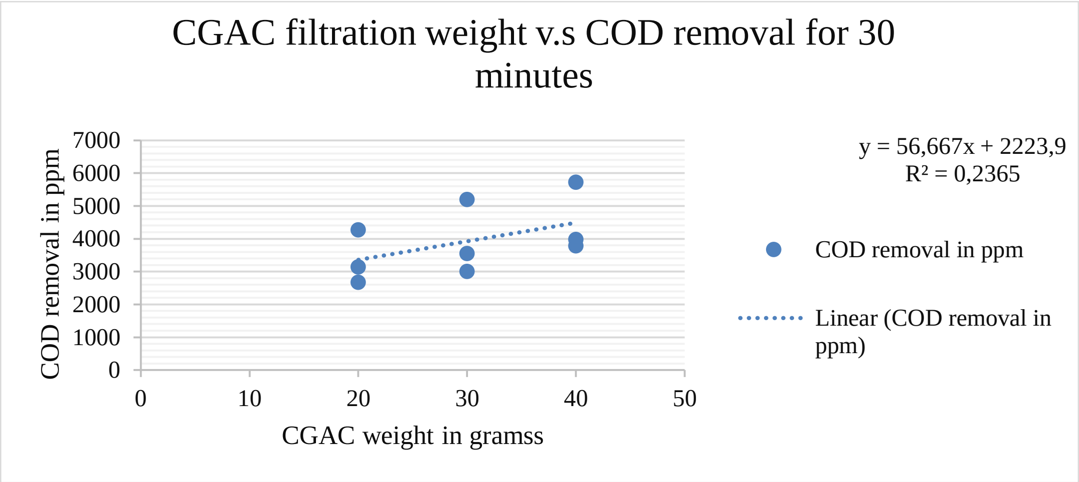
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,486327
R Square	0,236514
Adjusted R Square	0,127445
Standard Error	942,5969
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1926667	1926667	2,168476	0,184348
Residual	7	6219422	888488,9		
Total	8	8146089			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2223,889	1196,434	1,858764	0,105402	-605,228	5053,006	-605,228	5053,006
GAC in grams	56,66667	38,48136	1,472575	0,184348	-34,3273	147,6606	-34,3273	147,6606



r^2 of 0.2365 and r of 0.1275 indicates a very weak positive correlation.

F- ANOVA of 2.1685 indicates a small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.1275. This indicates that 12.75% of the total variation in the COD concentrations can be explained by the linear relationship, but 87.25% of the variation cannot be explained because of other independent factors.

Correlation of 60 minutes: 20g, 30g, and 40g:

CGAC in grams	COD removal in ppm
20	4059
20	4090
20	2980
30	4279
30	4570
30	3980
40	5260
40	5885
40	5745

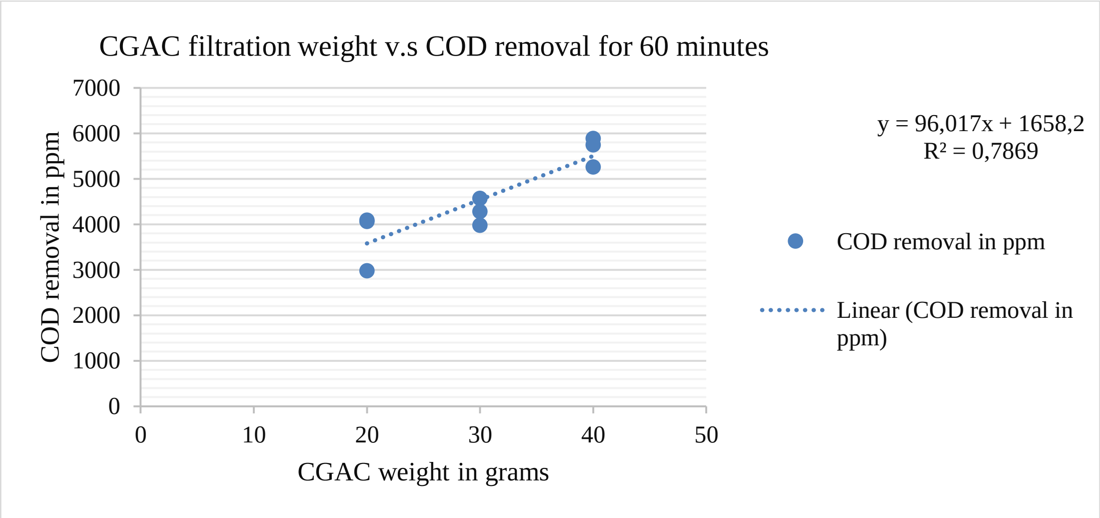
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,887073
R Square	0,786899
Adjusted R Square	0,756456
Standard Error	462,6007
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	5531520	5531520	25,8483	0,001424
Residual	7	1497996	213999,4		
Total	8	7029516			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	1658,167	587,177	2,823964	0,025627	269,7137	3046,62	269,7137	3046,62
GAC in grams	96,01667	18,88559	5,084122	0,001424	51,35933	140,674	51,35933	140,674



r^2 of 0,7869 and r of 0,7565 indicates a very weak positive correlation.

F- ANOVA of 25.8483 indicates a high number which implies significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.7565. This indicates that 75.65% of the total variation in the COD concentrations can be explained by the linear relationship; 24.35% of the variation cannot be explained because of other independent factors.

Correlation of 20 grams: 20 minutes, 30 minutes, and 40 minutes:

Contact time in minutes	COD removal in ppm
20	3705
20	2817
20	2670
30	4270
30	2675
30	3140
60	4059
60	4090
60	2980

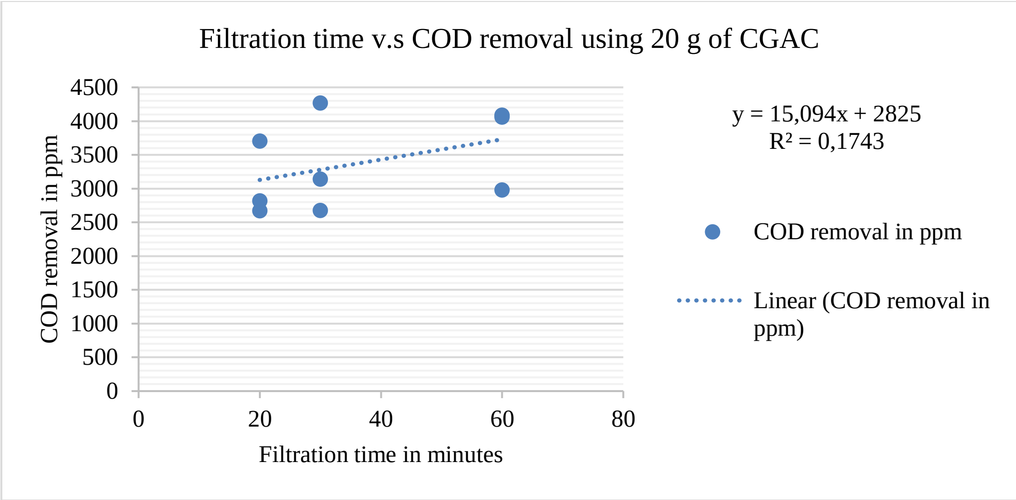
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,417478
R Square	0,174288
Adjusted R Square	0,056329
Standard Error	633,1571
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	592322,8	592322,8	1,477527	0,263562
Residual	7	2806215	400887,9		
Total	8	3398538			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2825,013	501,8365	5,629349	0,000791	1638,358	4011,668	1638,358	4011,668
Filtration in min	15,09359	12,41723	1,215536	0,263562	-14,2685	44,45568	-14,2685	44,45568



r^2 of 0.1743 and r of 0.05633 indicates a very weak positive correlation.

F- ANOVA of 1.4775 indicates a very small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.05633. This indicates that only 5.6330% of the total variation in the COD concentrations can be explained by the linear relationship, but 94.37% of the total variation cannot be explained because of other independent factors.

Correlation of 30 grams: 20 minutes, 30 minutes, and 40 minutes:

Contact time in minutes	COD removal in ppm
20	4575
20	3430
20	3140
30	5195
30	3545
30	3005
60	4059
60	4570
60	3980

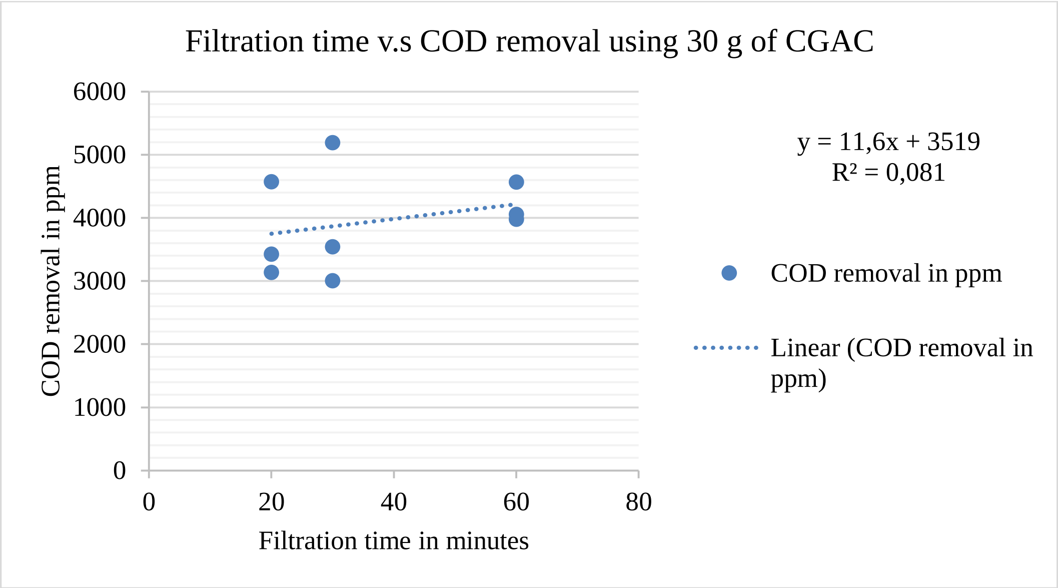
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,284543
R Square	0,0809647
Adjusted R Square	-0,050326
Standard Error	753,2061
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	349856	349856	0,6166826	0,4580331
Residual	7	3971236	567319,43		
Total	8	4321092			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	3519	596,98658	5,8946049	0,0006028	2107,3511	4930,6489	2107,3511	4930,6489
Filtration in min	11,6	14,771587	0,7852914	0,4580331	-23,329253	46,529253	- 23,329253	46,529253



r^2 of 0.08100 and r of 0,05033 indicates a very weak positive correlation.

F- ANOVA of 0.6168 indicates a very small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.05033. This indicates that only 5.0330% of the total variation in the COD concentrations can be explained by the linear relationship, but 94.97% of the total variation cannot be explained because of other independent factors.

Correlation of 40 gramss: 20minutes, 30minutes, and 40minutes.

Contact time in minutes	COD removal in ppm
20	5124
20	3970
20	3915
30	5720
30	3980
30	3785
60	5260
60	5885
60	5745

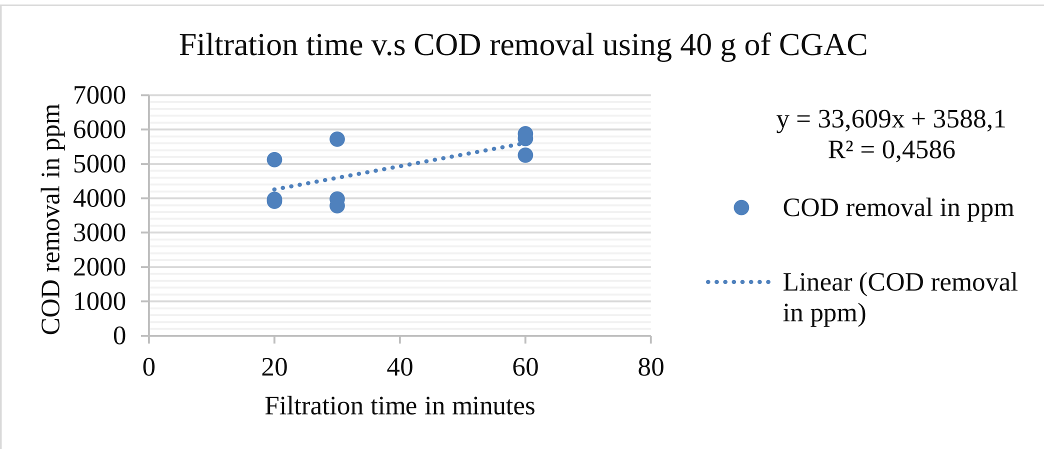
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,6771876
R Square	0,4585831
Adjusted R Square	0,3812378
Standard Error	703,80091
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	2936864,2	2936864,2	5,9290379	0,045091
Residual	7	3467350	495335,72		
Total	8	6404214,2			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	3588,1154	557,82832	6,4322933	0,0003562	2269,061	4907,1698	2269,061	4907,1698
Filtration in min	33,608974	13,802671	2,4349616	0,045091	0,9708429	66,247106	0,9708429	66,247106



r^2 of 0.4586 and r of 0.3812 indicates a weak positive correlation.

F- ANOVA of 5.9290 indicates a very small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.3812. This indicates that only 38.12% of the total variation in the COD concentrations can be explained by the linear relationship, but 61.88% of the total variation cannot be explained because of other independent factors.

Appendix M1: PAIRED T-TEST TRIAL TWO CALCULATIONS, BEFORE AND AFTER TREATMENT

Paired t-test for the mean difference conducted on trial 2 - batch 1: COD comparison before and after the treatment:

Sample No.	COD (ppm) Before Treatment - Alkaline samples	COD (ppm) After Treatment - Alkaline samples
1	7570	6280
2	7530	6180
3	7990	4740
4	7980	4720
5	7740	4970
6	7793	4930
7	8130	4800
8	8300	4840
9	7990	4850
10	7980	4840
11	7930	4790
12	7740	4560
13	8040	5100
14	8400	5110
15	8130	4940
16	8300	5000
17	7990	4890
18	7980	4920

Test claim:

H_0: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}}$ - $\bar{X}_{\text{COD after treatment}}$)

H_1: $\bar{X}_{\text{Difference}} \neq 0$

Trial Two - Batch 1						
Sample No.	COD Before Treatment	COD After Treatment	Difference	Diff - Mean Diff	(Diff -Mean Diff)Power2	
1	7570	6280	1290	1290	1664100	
2	7530	6180	1350	1350	1822500	
3	7990	4740	3250	3250	10562500	
4	7980	4720	3260	3260	10627600	
5	7740	4970	2770	2770	7672900	
6	7793	4930	2863	2863	8196769	
7	8130	4800	3330	3330	11088900	
8	8300	4840	3460	3460	11971600	
9	7990	4850	3140	3140	9859600	
10	7980	4840	3140	3140	9859600	
11	7930	4790	3140	3140	9859600	
12	7740	4560	3180	3180	10112400	
13	8040	5100	2940	2940	8643600	
14	8400	5110	3290	3290	10824100	
15	8130	4940	3190	3190	10176100	
16	8300	5000	3300	3300	10890000	
17	7990	4890	3100	3100	9610000	
18	7980	4920	3060	3060	9363600	
		Mean Diff	2947		162805469	= Sum Variance
	Under root	9576792,294				
	SD =	3094,639283				
	sqrt3	4,242640687				

			Test stat = AveDiff/(SD/sqrtn		At 95% confidence in two tail (0.025) and df n-1 (17)	
		SD Calculation	Test stat =	4,040766	Critical values 2.1098 and -2.1098	

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025(α/2) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.0408) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 2 batch 1 is rejected.

<u>Paired t-test for the mean difference conducted on trial 2 - batch 2: COD comparison before and after the treatment:</u>

Sample No.	**COD (ppm) Before Treatment - Alkaline samples**	**COD (ppm) After Treatment - Alkaline samples**
1	8130	5219
2	8300	5180
3	8130	4630
4	8300	4680
5	7990	4740
6	7980	4740
7	8130	4330
8	8300	4320
9	7990	4310
10	7980	4370
11	7930	4270
12	7740	4280
13	9310	5180
14	9930	5400
15	8040	4520
16	8400	4440
17	7801	3500
18	7760	4040

Test claim:

H_0: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}}$ - $\bar{X}_{\text{COD after treatment}}$)

H_1: $\bar{X}_{\text{Difference}} \neq 0$

Trial Two - Batch 2						
Sample No.	COD Before Treatment	COD After Treatment	Difference	Diff - Mean Diff	(Diff -Mean Diff)Power2	
1	8130	5219	2911	2911	8473921	
2	8300	5180	3120	3120	9734400	
3	8130	4630	3500	3500	12250000	
4	8300	4680	3620	3620	13104400	
5	7990	4740	3250	3250	10562500	
6	7980	4740	3240	3240	10497600	
7	8130	4330	3800	3800	14440000	
8	8300	4320	3980	3980	15840400	
9	7990	4310	3680	3680	13542400	
10	7980	4370	3610	3610	13032100	
11	7930	4270	3660	3660	13395600	
12	7740	4280	3460	3460	11971600	
13	9310	5180	4130	4130	17056900	
14	9930	5400	4530	4530	20520900	
15	8040	4520	3520	3520	12390400	
16	8400	4440	3960	3960	15681600	
17	7801	3500	4301	4301	18498601	
18	7760	4040	3720	3720	13838400	
		Mean Diff	3666		244831722	= Sum Variance
	Under root	14401866				
	SD =	3794,979051				
	sqrt3	4,242640687				

			Test stat = AveDiff/(SD/sqrtn	At 95% confidence in two tail (0.025) and df n-1 (17)	
SD Calculations			Test stat = 4,098696	Critical values 2.1098 and -2.1098	

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025(α/2) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.0987) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 2 batch 2 is rejected.

Paired t-test for the mean difference conducted on Trial 2 - batch 3: COD comparison before and after the treatment:

Sample No.	COD (ppm) Before Treatment - Alkaline samples	COD (ppm) After Treatment - Alkaline samples
1	9310	5900
2	9930	5987
3	8130	4000
4	8300	4100
5	7990	4340
6	7980	4440
7	8040	3800
8	8040	4220
9	8130	4340
10	8300	4300
11	7930	4100
12	7740	4130
13	7570	2850
14	7530	2845
15	7740	3450
16	7930	3450
17	7990	3580
18	7980	3510

Test claim:

H$_0$: $\bar{X}_{\text{Difference}} = 0$; Where: $\bar{X}_{\text{Difference}}$ is ($\bar{X}_{\text{COD before treatment}}$ - $\bar{X}_{\text{COD after treatment}}$)

H$_1$: $\bar{X}_{\text{Difference}} \neq 0$

Trial Two - Batch 3						
Sample No.	**COD Before Treatment**	**COD After Treatment**	**Difference**	**Diff - Mean Diff**	**(Diff -Mean Diff)Power2**	
1	9310	5900	3410	3410	11628100	
2	9930	5987	3943	3943	15547249	
3	8130	4000	4130	4130	17056900	
4	8300	4100	4200	4200	17640000	
5	7990	4340	3650	3650	13322500	
6	7980	4440	3540	3540	12531600	
7	8040	3800	4240	4240	17977600	
8	8040	4220	3820	3820	14592400	
9	8130	4340	3790	3790	14364100	
10	8300	4300	4000	4000	16000000	
11	7930	4100	3830	3830	14668900	
12	7740	4130	3610	3610	13032100	
13	7570	2850	4720	4720	22278400	
14	7530	2845	4685	4685	21949225	
15	7740	3450	4290	4290	18404100	
16	7930	3450	4480	4480	20070400	
17	7990	3580	4410	4410	19448100	
18	7980	3510	4470	4470	19980900	
		Mean Diff	4068		300492574	**= Sum Variance**
	Under root	17676033,76				
	SD =	4204,287545				
	sqrt3	4,242640687				

			Test stat = AveDiff/(SD/sqrtn		At 95% confidence in two tail (0.025) and df n-1 (17)	
		SD Calculation	Test stat =	4,104773	Critical values 2.1098 and -2.1098	

The critical values were found to be 2.1098 and -2.1098 at 95% 0.025(α/2) confidence limits, and 17(n-1) degrees of freedom. Since the calculated t_{stat} (4.1048) falls within a rejection area, there is significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. The alternate hypothesis states that there is a difference in the COD concentrations means, between the COD concentrations before and after treatment. Therefore, the null hypothesis which states that there is no difference between the two COD concentrations means for trial 2 batch 3 is rejected.

Appendix M2: TRIAL TWO CORRELATION STUDY USING THE DATA COLLECTED ON THE 30 SEPTEMBER 2018 AND 29 OCTOBER 2018

Correlation of 20 minutes: 20g, 30g, and 40g:

CGAC in grams	**COD removal in ppm**
20	1320
20	3255
20	2817
30	3016
30	3560
30	3245
40	3677
40	4165
40	3595

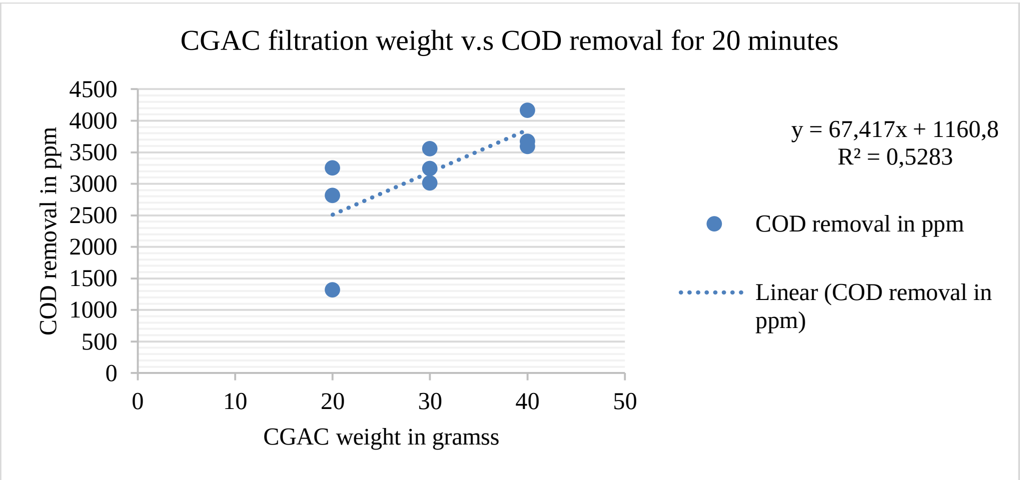
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,7268406
R Square	0,5282973
Adjusted R Square	0,4609112
Standard Error	589,77839
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	2727004,2	2727004,2	7,8398561	0,0265252
Residual	7	2434869,8	347838,55		
Total	8	5161874			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	1160,8333	748,60307	1,550666	0,1649177	-609,33163	2930,9983	-609,33163	2930,9983
GAC in grams	67,416667	24,077602	2,7999743	0,0265252	10,482185	124,35115	10,482185	124,35115



r^2 of 0.5283 and r of 0.4609 indicates a weak positive correlation.

F- ANOVA of 5.9290 indicates a small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.4609. This indicates that only 46.09% of the total variation in the COD concentrations can be explained by the linear relationship, but 53.91% of the total variation cannot be explained because of other independent factors.

Correlation of 30 minutes: 20g, 30g, and 40g:

CGAC in grams	COD removal in ppm
20	3395
20	3140
20	3160
30	3890
30	3645
30	3560
40	4030
40	3895
40	3720

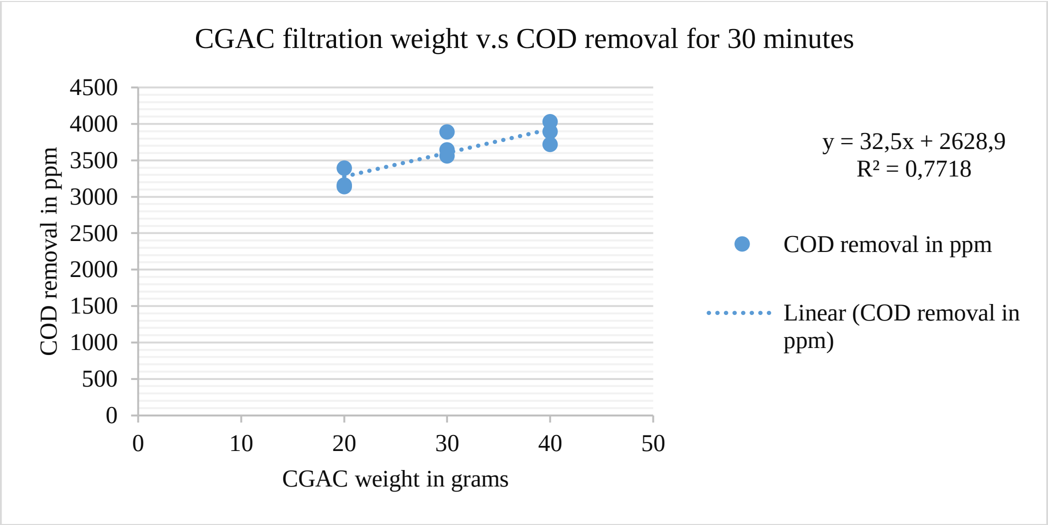
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,878518
R Square	0,7717939
Adjusted R Square	0,739193
Standard Error	163,61492
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	633750	633750	23,674029	0,0018234
Residual	7	187388,89	26769,841		
Total	8	821138,89			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2628,8889	207,67568	12,658627	4,439E-06	2137,8139	3119,9638	2137,8139	3119,9638
GAC in grams	32,5	6,679551	4,8655965	0,0018234	16,705372	48,294628	16,705372	48,294628



r^2 of 0.7718 and r of 0.7392 indicates a good positive correlation.

F- ANOVA of 23.6740 indicates a fair number which implies significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.7392. This indicates that 73.92% of the total variation in the COD concentrations can be explained by the linear relationship. The variation of 26.08% cannot be explained because of other independent factors.

<u>Correlation of 60 minutes: 20 g, 30g, and 40 g:</u>

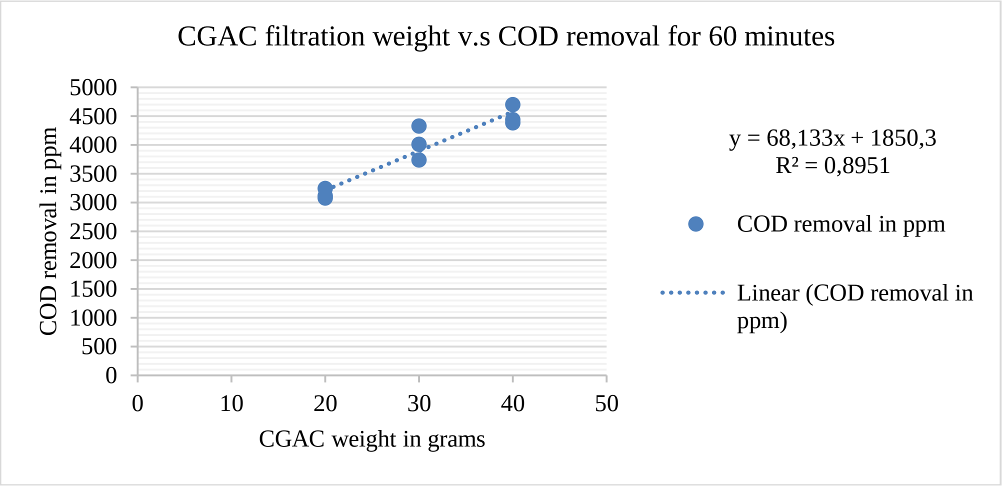
CGAC in grams	COD removal in ppm
20	3115
20	3245
20	3080
30	4330
30	3740
30	4011
40	4703
40	4385
40	4440

Regression Statistics	
Multiple R	0,946081
R Square	0,8950692
Adjusted R Square	0,8800791
Standard Error	215,97795
Observations	9

ANOVA

	df	SS	MS	F	Significance F
Regression	1	2785290,7	2785290,7	59,710634	0,0001137
Residual	7	326525,33	46646,476		
Total	8	3111816			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95,0%	Upper 95,0%
Intercept	1850,3333	274,13985	6,7495964	0,0002651	1202,0956	2498,5711	1202,0956	2498,5711
GAC in grams	68,133333	8,817263	7,7272656	0,0001137	47,283819	88,982847	47,283819	88,982847



r^2 of 0.8951 and r of 0.8801 indicates a good positive correlation.

F- ANOVA of 59.7106 indicates a high number which implies significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.8801. This indicates that 88.01% of the total variation in the COD concentrations can be explained by the linear relationship. Only the variation of 11.99% cannot be explained because of other independent factors.

Correlation of 20 grams: 20 minutes, 30 minutes, and 40 minutes:

Contact time in minutes	COD removal in ppm
20	1320
20	3255
20	2817
30	3395
30	3140
30	3160
60	3110
60	3245
60	3080

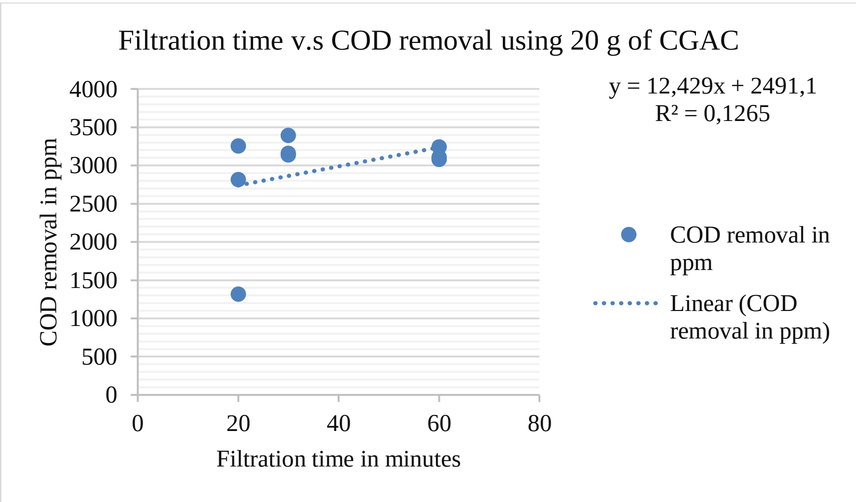
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,3556715
R Square	0,1265022
Adjusted R Square	0,0017168
Standard Error	629,46659
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	401679,59	401679,59	1,0137583	0,347533
Residual	7	2773597,3	396228,18		
Total	8	3175276,9			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	2491,141	498,91139	4,9931532	0,0015774	1311,403	3670,879	1311,403	3670,879
Filtration in min	12,429487	12,344855	1,0068556	0,347533	-16,761457	41,620432	- 16,761457	41,620432



r^2 of 0.1265 and r of 0.001717 indicates a very weak positive correlation.

F- ANOVA of 1.01376 indicates a very small number which implies non-significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.001717. This indicates that 0.1717% of the total variation in the COD concentrations can be explained by the linear relationship; however, 99.83% of the total variation cannot be explained because of other independent factors.

Correlation of 30 grams: 20 minutes, 30 minutes, and 40 minutes:

Contact time in minutes	**COD removal in ppm**
20	3245
20	3560
20	3245
30	3890
30	3645
30	3560
60	4330
60	3740
60	4011

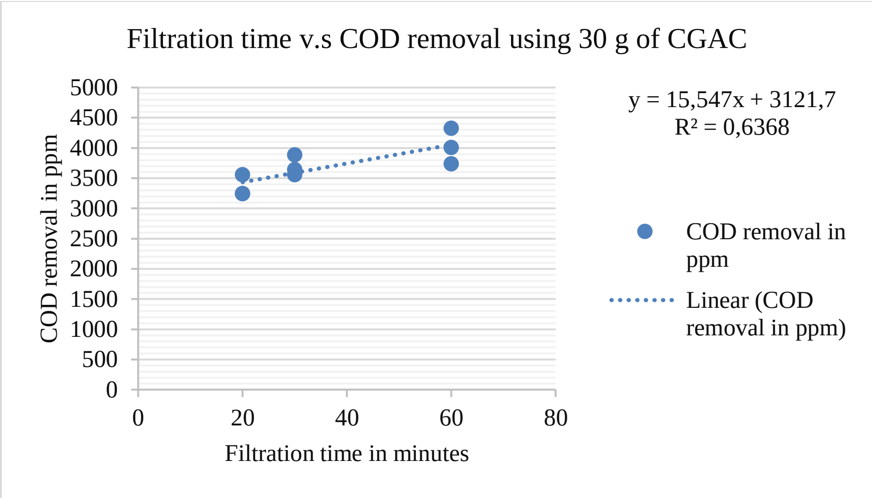
SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,7979756
R Square	0,6367651
Adjusted R Square	0,5848743
Standard Error	226,30838
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	628479,18	628479,18	12,271274	0,0099522
Residual	7	358508,37	51215,482		
Total	8	986987,56			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	3121,7051	179,37064	17,403657	5,086E-07	2697,561	3545,8493	2697,561	3545,8493
Filtration in min	15,547436	4,4382724	3,5030378	0,0099522	5,0525893	26,042283	5,0525893	26,042283



r^2 of 0.6368 and r of 0.5849 indicates a good positive correlation.

F- ANOVA of 12.2713 indicates a number which implies significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.5849. This indicates that 58.49% of the total variation in the COD concentrations can be explained by the linear relationship. The variation of 41.51% cannot be explained because of other independent factors.

Correlation of 40 grams: 20 minutes, 30 minutes, and 40 minutes:

Contact time in minutes	COD removal in ppm
20	3560
20	4125
20	3560
30	4030
30	3895
30	3720
60	4703
60	4385
60	4440

SUMMARY OUTPUT

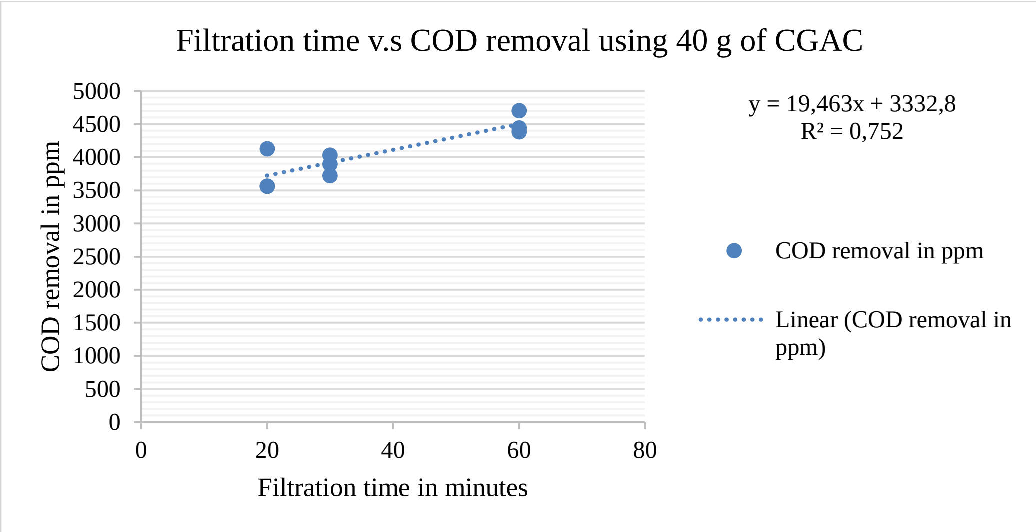
Regression Statistics	
Multiple R	0,8671513
R Square	0,7519514
Adjusted R Square	0,7165159
Standard Error	215,43532
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	984883,59	984883,59	21,22028	0,002465

Residual	7	324886,63	46412,375
Total	8	1309770,2	

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Intercept	3332,8077	170,75272	19,518328	2,312E-07	2929,0417	3736,5737	2929,0417	3736,5737
Filtration in min	19,462821	4,2250342	4,6065475	0,002465	9,4722023	29,453439	9,4722023	29,453439



r^2 of 0.7520 and r of 0.7166 indicates a good positive correlation.

F- ANOVA of 21.2203 indicates a big number which implies significant correlation.

The coefficient of determination of the increased CGAC weight versus the COD concentrations removal is found to be 0.7166. This indicates that 71.66% of the total variation in the COD concentrations can be explained by the linear relationship. The variation of 28.34% cannot be explained because of other independent factors.

Appendix N: T-TEST CALCULATIONS FOR EQUAL VARIANCE MEANS CONDUCTED ON TRIAL ONE AND TRIAL TWO COD REMOVAL DATA

Filtration condition: 20 grams & 20 minutes:

COD removal in ppm	
Acidic – 20 grams: 20 minutes	Alkaline 20 grams: 20 minutes
3705 ppm	3016 ppm
2870 ppm	3255 ppm
2670 ppm	2817 ppm

Test claim:

H$_0$: $\bar{X}_A = \bar{X}_B$

H$_1$: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 20 g &20 minutes	*COD removal: Alkaline 20 g &20 minutes*
Mean	3081,666667	3029,333333
Variance	301408,3333	48094,33333
Observations	3	3
Pooled Variance	174751,3333	
Hypothesized Mean Difference	0	
df	4	
t Stat	0,153325284	
P(T<=t) two-tail	0,885565781	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 0.1533 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 20 grams & 30 minutes:

COD removal in ppm	
Acidic – 20 grams: 30 minutes	Alkaline – 20 grams: 30 minutes
4270 ppm	3395 ppm
2675 ppm	3140 ppm
3140 ppm	3160 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 20 g & 30 minutes	*COD removal: Alkaline 20 g & 30 minutes*
Mean	3361,666667	3231,666667
Variance	672858,3333	20108,33333
Observations	3	3
Pooled Variance	346483,3333	
Hypothesized Mean Difference	0	
df	4	
t Stat	0,270487878	
P(T<=t) two-tail	0,800168017	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 0.2705 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 20 grams & 60 minutes:

COD removal in ppm	
Acidic – 20 grams: 60 minutes	Alkaline – 20 grams: 60 minutes
4059 ppm	3115 ppm
4090 ppm	3245 ppm
2980 ppm	3080 ppm

Test claim:

H$_0$: $\bar{X}_A = \bar{X}_B$

H$_1$: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	Acidic – 20 g: 60 minutes	*Alkaline – 20 g: 60 minutes*
Mean	3709,666667	3146,666667
Variance	399550,3333	7558,333333
Observations	3	3
Pooled Variance	203554,3333	
Hypothesized Mean Difference	0	
df	4	
t Stat	1,528318423	
P(T<=t) two-tail	0,201157585	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 1.5283 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 30 grams & 20 minutes:

COD removal in ppm	
Acidic – 30 grams: 20 minutes	Alkaline – 30 grams: 20 minutes
4575 ppm	1320 ppm
3140 ppm	3560 ppm
3430 ppm	4165 ppm

Test claim:

H$_0$: $\bar{X}_A = \bar{X}_B$

H$_1$: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 3 0g & 20 minutes	*COD removal: Alkaline 30 g & 20 minutes*
Mean	3715	3015
Variance	575725	2246275
Observations	3	3
Pooled Variance	1411000	
Hypothesized Mean Difference	0	
df	4	
t Stat	0,721738982	
P(T<=t) two-tail	0,510390544	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 0.7517 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 30 grams & 30 minutes:

COD removal in ppm	
Acidic – 30 grams: 30 minutes	Alkaline – 30 grams: 30 minutes
5195 ppm	3890 ppm
3005 ppm	3645 ppm
3545 ppm	3560 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 30 g & 30 minutes	*COD removal: Alkaline 30 g & 30 minutes*
Mean	3915	3698,333333
Variance	1301700	29358,33333
Observations	3	3
Pooled Variance	665529,1667	
Hypothesized Mean Difference	0	
df	4	
t Stat	0,325277621	
P(T<=t) two-tail	0,761274072	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 0.3253 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 30 grams & 60 minutes.

COD removal in ppm	
Acidic – 30 grams: 60 minutes	Alkaline – 30 grams: 60 minutes
4279 ppm	4330 ppm
4170 ppm	3740 ppm
4570 ppm	4011 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 30 g & 60 minutes	***COD removal: Alkaline 30 g & 60 minutes***
Mean	4339,666667	4027
Variance	42760,33333	87217
Observations	3	3
Pooled Variance	64988,66667	
Hypothesized Mean Difference	0	
df	4	
t Stat	1,502133044	
P(T<=t) two-tail	0,207476454	
t Critical two-tail	3,4954 to 3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 1.5021 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, since the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 40 grams & 20 minutes.

COD removal in ppm	
Acidic – 40 grams: 20 minutes	Alkaline – 40 grams: 20 minutes
5124 ppm	3677 ppm
3970 ppm	4165 ppm
3915 ppm	3595 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples**.**

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 40 g & 20 minutes	*COD removal: Alkaline 40 g & 20 minutes*
Mean	4336,333333	3812,333333
Variance	466070,3333	94961,33333
Observations	3	3
Pooled Variance	280515,8333	
Hypothesized Mean Difference	0	
df	4	
t Stat	1,211708702	
P(T<=t) one-tail	0,146153203	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 1.2117 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, as the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 40 grams & 30 minutes.

COD removal in ppm	
Acidic – 40 grams: 30 minutes	Alkaline – 40 grams: 30 minutes
5720 ppm	4030 ppm
3785 ppm	3895 ppm
3980 ppm	3720 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 40 g & 30 minutes	*COD removal: Alkaline 40 g & 30 minutes*
Mean	4495	3881,666667
Variance	1134975	24158,33333
Observations	3	3
Pooled Variance	579566,6667	
Hypothesized Mean Difference	0	
df	4	
t Stat	0,9867122	
P(T<=t) two-tail	0,37964372	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits; since the calculated t_{stat} of 0.9867 falls within a non-rejection area, there is enough significant statistical evidence to accept the null hypothesis and reject the alternate hypothesis. Therefore, as the null hypothesis is accepted, it can be concluded that mean of the reduced COD concentrations obtained when using acidic and alkaline samples are the same for this treatment.

Filtration condition: 40 grams & 60 minutes.

COD removal in ppm	
Acidic – 40 grams: 60 minutes	Alkaline 40 grams: 60 minutes
5260 ppm	4703 ppm
5745 ppm	4385 ppm
5885 ppm	4440 ppm

Test claim:

H_0: $\bar{X}_A = \bar{X}_B$

H_1: $\bar{X}_A \neq \bar{X}_B$

Where A is reduced COD concentrations from acidic samples and B is reduced concentrations from alkaline samples.

T-test: Two-sample assuming equal variances.

	COD removal: Acidic 40 g & 60 minutes	*COD removal: Alkaline 40 g & 60 minutes*
Mean	5630	4509,333333
Variance	107575	28886,33333
Observations	3	3
Pooled Variance	68230,66667	
Hypothesized Mean Difference	0	
df	4	
t Stat	5,254510649	
P(T<=t) two-tail	0,006277894	
t Critical two-tail	3,4954 to -3,4954	

The critical values were found to be 3.4954 and -3.4954 at 95% 0.025(α/2) confidence limits since the calculated t_{stat} of 5.2545 falls within a rejection area. Therefore, there is enough significant statistical evidence to reject the null hypothesis and accept the alternate hypothesis. Therefore, as an alternate hypothesis is accepted, it can be concluded that means of the reduced COD concentrations obtained when using acidic and alkaline samples are not the same for this treatment. Since there was a significant difference in the reduced COD concentrations means, the t-test for unequal variances was conducted.

Since the t-statistic falls within the rejection area, we can therefore conclude that there is no significant statistical evidence to reject the null hypothesis which states that the reduced means obtained from the acidic samples are the same as the alkaline samples. Thus, the alternate hypothesis which states that there is a statistical difference in the reduced COD concentrations means obtained when the two pH content samples were used.

Appendix O1: SEVERITY RATING SCALE FOR THE LIKELIHOOD OF THE IMPACT

Bad	Rating	Criteria: A failure could cause
	10	Kill aquatic animals and plants
	9	Be illegal
	8	Render the effluent unfit for discharge
	7	Cause extreme customer dissatisfaction
	6	Result in partial malfunction
	5	Cause a loss of performance likely to result in a complaint
	4	Cause minor performance loss
	3	Cause a minor nuisance; can be overcome with no loss
	2	Be unnoticed; minor effect on performance
	1	Can be noticed and no effect on organization
Good		

Appendix O2: OCCURRENCE RATING SCALE: FREQUENCY OF OCCURRENCE

	Rating	Time or period	Probability
Bad	10	More than once per day	>30%
	9	Once every 3-4 days	≤30%
	8	Once per week	≤5%
	7	Once per month	≤1%
	6	Once every 3 months	≤0.03%
	5	Once every six months	≤1 per 10 000
	4	Once per year	≤6 per 100 000
	3	Once every 1-to-3 years	≤6 per 1000 000
	2	Once every 3-to-6 years	≤3 per 1000 000
	1	Once every 6-to-100 years	≤2 per 1000 000
Good			

Appendix O3: DETECTION RATING SCALE: HOW EASILY THE FAILURE CAN BE DETECTED

Bad → Good	Rating	Definition
Bad	10	Failure is not detectable
	9	Occasional units are checked for non-compliance
	8	Units are systematically sampled and inspected
	7	All the units are manually inspected
	6	Manual inspection with mistake-proofing modifications
	5	Process is monitored using SPC and manually inspected
	4	SPC used with an immediate reaction to out of control conditions
	3	SPC and 100% inspection surrounding out of control conditions
	2	All the units are automatically inspected
Good	1	Defect is obvious and can be kept from affecting the effluent quality(COD).

Appendix P: TROUBLESHOOTING PROCEDURE FOR IMPROVING NON-CONFORMING COD CONCENTRATIONS IN THE WCM EFFLUENT

Purpose: To prevent the COD concentrations in the WCM effluent from exceeding COD regulatory.
Frequency: Every time the COD siren or alarm goes off when the effluent is more than 4800ppm.
Responsibility: Operator/Supervisor/Engineer/Production Manager,