Enhancing adhesion strength of a solvent-free lamination unit for flexible packaging applications using Taguchi based optimization

By

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Declaration

I, **Sandisile Mgobo**, declare that the contents of this thesis represent my own unaided work, and the thesis has not been previously submitted for any academic exemption towards any qualification. Furthermore, it represents my own opinions and not necessarily those of the Cape Peninsula University of Technology.

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Signed **Date**

Abstract

A solvent-free (SF) laminating unit was modelled with the objective to demonstrate the influence of the various process parameters to improve adhesion between polyamide (PA) and polyethylene (PE) films. The effects of the process parameters were investigated using an experimental design based on a fractional factorial method. Taguchi's L_{18} orthogonal array (OA) was considered to study eight process parameters. Taguchi's L_{18} OA is capable of studying seven factors at three levels, namely: rewind tension (RT), taper tension (TT), surface energy (SE), coating weight (CW), machine speed (MS), application temperature (AV), mix ratio (MR), plus one factor at two levels – curing temperature (CT).

In theory, assumptions are made when optimising the manufacturing process; however, in practice, these assumptions can distort the output response, thereby affecting precision and accuracy of the DOE method utilised. To limit the system and part-to-part variation in this study, a Gage repeatability and reproducibility (R&R) was performed to eliminate variability within the system. Furthermore, the Gage R&R study was performed to maximise the output adhesion strength (AS) with a higher degree of accuracy and precision. In this study, the multivariate optimisation technique by Taguchi method (TM) was utilised to reduce the effect of the noise factors (NF) on the response while quantifying the contribution of each design parameter through signal-to-noise (S/N) ratio.

A model was derived to predict adhesion bond strength between two flexible films using the solvent-free laminating unit. The two process outputs that were used to predict the model were AS and tensile strength (TS). Based on the result, the highest AS achieved was 640 N; at the same parameter condition, a TS of 21.02 seconds was achieved. The optimum operating conditions for optimising the solvent-free (SF) lamination process and achieving a high output response for AS and TS were: SE = 44 dyne/cm, MS = 150 m/min, AV at 45 ^{o}C, MR = 85 %, TT = 25%, RT = 100 N, CW = 1.5 gsm, and CT = 32 ^{o}C.

The optimum parameters were employed to generate a model and predict the behaviour of the system at different parameter conditions. Using the optimum operating condition, an output of 613.05 N was achieved with an experimental error of 4.2%. The output on the S/N ratio estimated using the model was 56.12, and the predicted value was 55.57 with 0.97% error. The analysis of variance (ANOVA) was also conducted to illustrate the contribution of controllable variables on the output response. Based on the ANOVA results, the contribution of design parameters to the output response was calculated. The result demonstrated that SE was the most statistically significant variable with a contrition of 70.2%. According to the response table, MS is the second most significant parameter ranked at number 2 with a Delta

of 46.4. The AV is the third most important variable with a Delta of 60 and ranked at number 3. The estimated constant for the model using the ANOVA was 444.16 with S:57.39, R-Sq of 96.5%, and R-Sq (adj) of 70.5%. The regression equation was also developed, and analysis done with the assumption that there is limited interaction between variables. The two polymeric films used in this study were polyamide $(C_{12}H_{22}N_2O_2)_n$ and polyethylene $(C_2H_4)_n$ materials. A two-component polyurethane (PU) adhesive which consists of resin and hardener were used as SF laminating adhesive heated at 40 ^oC dosed at 35 ^oC. The hardener in this case was used as a catalyst to drive the curing process.

The relationship and the interaction between the variables were also studied. Based on the result obtained, an increase in surface energy (SE) of the PE causes an increase in the AS and TS. Therefore, it was recommended that the SE of the PE should be always kept at level 3 which is 44 dyne/cm. Under such conditions, the molecules of the film are activated and rearranged in order to prepare them for adhesion. When process parameters were set according to the achieved operating condition, waste was reduced and more material was recovered since adhesive and cohesive forces between the PE and polyamide (PA) were improved.

Chapter 6 quantified the value of all resources utilised in this study. The profitability and cost of waste generated were highlighted using the current value stream map (CVSM). A new process is mapped using one of the lean tools called the future value stream map (FVSM). Upon achieving the best operating level, the Taguchi quality loss function (QLF) was used to validate the effectiveness of the generated model. The QLF was examined to reduce process variation and improve consistence. A new experiment was conducted to monitor deviation, and a normal distribution curve was plotted to assess the distribution of the actual output response with respect to the desired new target (640 N).

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- I would also like to acknowledge AFSA for their financial assistance, as well as thank their executive team for allowing me to utilise their equipment to make my dream a reality.

Dedication

To My Loving Family:

My mother, Nontle Mgobo,

My late uncle, Mpendulo Wiseman Mgobo,

Xatyiswa Mgobo

and

Momelezi Goodman Mgobo.

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Glossary

List of Abbreviations

AFSA	Amcor Flexible South Africa
ANOVA	Analysis of Variance
AS	Adhesion Strength
AV	Application Temperature
BBD	Box-Behnken Design
CCC	Central Composite Circumscribed
CCD	Central Composite Design
CCF	Central Composite Face-Centred
CCI	Central Composite Inscribe
COF	Coefficient of Friction
Cpk	Process Capability
CT	Curing Temperature
C/T	Cycle of Time
CVSM	Current Value Stream Map
CW	Coating Weight
DF	Degree of Freedom
DMF	Dimethylformamide
DOE	Design of Experiment
DOF	Degree of Freedom
EA	Ethyl Acetate
EL	Extrusion Lamination
EVA	Ethylene Vinyl Acetate
EVOH	Ethylene Vinyl Alcohol
FFD	Full Factorial Design
FVSM	Future Value Stream Map
HDPE	High Density Polyethylene
LB	Light Barrier
LDPE	Low Density Polyethylene
LLDPE	Linear Low-Density Polyethylene
MEK	Methyl Ethyl Ketone
MFI	Melt Flow Index
MR	Mix Ratio
MS	Machine Speed

MW	Molecular Weight
MWD	Molecular Weight Distribution
NF	Noise Factors
OA	Orthogonal Array
OPR	Oxygen Permeation Rate
OTR	Oxygen Transmission Rate
PA	Polyamide
PBT	Polyester Polybutylene Terephthalate
PE	Polyethylene
PEN	Polyethylene Naphthalate
PET	Polyesters
PP	Polypropylene
PU	Polyurethane
QLF	Quality Loss Function
RM	Raw Materials
R&R	Repeatability and Reproducibility
RSM	Response Surface Methodology
RT	Rewind Tension
SB	Solvent-Based
SE	Surface Energy
SF	Solvent-Free
SL	Solventless
S/N	Signal-to-Noise
SOC	Standard Operating Condition
SS	Sum of The Squares
SST	Total Sum of The Squares
TM	Taguchi Method
TPM	Total Preventative Maintenance
TS	Tensile Speed
TT	Taper Tension
VS	Value Stream
WB	Water-Based
WPR	Water Permeation Rate
WVP	Water Vapour Permeation

List of Symbols

System	Explanation	Unit
T	Time	S
N	Force	$Kg.m.s^{-2}$
L	Length	mm
CW	Coating weight	Gsm
N	Quantity of replicates	-
Y	Output response	-
SE	Surface energy	Dyne/cm
TT	Taper tension	%
MS	Machine speed	m/min
RT	Rewind tension	N
K	Design parameters	-
T_g	Glass transition temperature	^oC

Chemical Formulae

Element / Compound	Description
$C_{18}H_{35}N_3O_3$	Polyamide
$(C_2H_2)N$	Polyethylene
CO_2NH	Polyether Urethane
COH	Polyether
NCO	Isocyanate
CO_2	Carbon dioxide
N_2	Nitrogen

Greek Symbols

Symbol	Explanation	Units
μ	Viscosity	mPa.s
β's	Coefficient of a linear function, Quadratic and interaction	-
ε	Process response	-
β_0	Constant term	-

Key Terms and Concepts

- **Amorphous polymers**: refers to polymers with molecular structures arranged in a randomly ordered manner with a maximum degree of disorder.
- **ANOVA:** is a statistical tool used to quantify the contribution of each parameter on the output response and investigate the significance between the process mean of three or more independent variable groups.
- **Corona treatment:** A surface modification method performed to alter the chemical structure of substrates to improve their wettability and surface energy since polymers are chemical inert.
- **Crystalline:** A solid material made up of crystals with microscopic structures containing interlocking particles arranged in a highly ordered manner and definite pattern.
- **Glass transition temperature:** can be described as the specific temperature by which amorphous polymers are transformed from a glassy state to rubber state.
- **Lamination:** A process by which two or more web substrates are bonded together using adhesive. Heat and pressure are supplied by nip rollers to improve the rate of reaction. During the process, web is corona treated to improve wettability and surface energy.
- **Modelling:** is a technique that uses engineering principles to derive and develop linear and nonlinear equations to predict a static/dynamic behaviour of the system at certain operating conditions.
- **Noise factors**: These are parameters that are outside the designers' control but affect the performance of a product or process.
- **Process optimisation:** is a branch of the engineering discipline that is based on manipulating process parameters to maximise the output response without compromising the quality and the design of the system.
- **Quality loss function:** A statistical technique for evaluating losses as the result of process variation causing the target value to deviate from the process mean.
- **Taguchi method**: A statistical design approach based on using orthogonal array to equally assess all process parameters through design of experiment (DOE).
- **Taper tension:** Simply means the rate at which the film tension decreases while the radius on the winding roll increases.

- **Water vapour permeation:** The ability of a vapour mass to penetrate through a thin layer of web material at a specified temperature condition.
- **Winding:** A process by which web is transformed into a form of centred-wound roll by controlling the strain of the film.

CHAPTER 1

INTRODUCTION

Chapter 1: Introduction

1.1 Introduction and Background

The growing demand for high barrier polymer structures in the flexible packaging industry has compelled organisations in this sector to operate at optimum processing conditions in order to improve the strength of polymers and their rheological behaviour during processing (Siracusa, 2012; Mohsin et al., 2012; Zhong et al., 2019). In this day and age, polymers are required to possess certain characteristics such as oxygen barrier, light barrier, heat saleability, and water vapour permeation, which a single polymer component cannot offer; thus lamination is utilised (Wang et al., 2017; Michiels et al., 2017). These attributes are critical to prolong the shelf life of a packaging material and prevent product loss after lamination (Tamarindo & Pastore, 2016). The lamination process in the flexible packaging industry involves the continuous coating and bonding of multilayer web substrates to accomplish good mechanical integrity of the films (Anjan & Annu, 2015). During this process, two or more web substrates with different polarity and molecular weight distribution (MWD) are bonded together using adhesive. There are three common types of adhesive lamination utilised during the production of flexible packaging material, namely: Solvent-based (SB), water-based (WB), and solvent-free (SF) lamination. For the purpose of this thesis, the focus will be on SF laminations.

The SF lamination process has become more attractive in recent years since there is no solvent emission during processing, thus low energy is used to drive the process. In addition, the SF lamination process is driven by means of heat and pressure to accelerate the bonding rate between two or more films. The process protects and improves the appearance of the packaging films. SF Lamination enhances the strength of the packaging material and roll geometry. Given the increase in demand for high strength polymers, it is essential to optimise process parameters during the lamination of packaging material to meet the requirement (Onyeagoro, 2012).

The study combines optimisation and process modelling of SF lamination in the flexible packaging industry using Taguchi Orthogonal Array (OA). There has been limited focus on optimising process parameters in the flexible packaging industry using the Taguchi method (TM). Particular attention will be given to the process parameters used during the lamination of polyethylene $(C_2H_2)_n$ and biaxially oriented polyamide $(C_{18}H_{35}N_3O_3)$ films in this study. These parameters include raw material surface energy (dyne/cm); coating weight (CW) which is measured in grams per square meter (gsm); machine speed (MS) with a unit of measure metres per minute (m/min); taper tension (TT) and mix ratio (MR) which are both

measured in percentage (%); rewind tension (RT) which is measured in Newtown (N); and application temperature (AW) and curing temperature (CT) which are both measured in degrees Celsius (^{o}C). These factors are critical in maintaining film bond strength and laminate elongation with effective barrier properties.

Without modelling and process optimisation, the demand for high barrier polymeric films would not be achieved or met. As a result, many design of experiments (DOE) approaches have been utilised to model systems and maximise the process output responses in a number of manufacturing industries. These models are critical to predict the behaviour of processes in order to minimise the risks of existing systems and increase the reliability of new processes. Such models can be generated using the TM OA to reduce variation and quantify the contribution of each input variable (Davis & John, 2017; Hamzacebi, 2016; Kondapalli et al., 2015; Karna et al., 2012).

There have been limited studies focusing on using the TM approach in the flexible packaging industry. In fact, a search yielded no results on reducing process variation and quantifying the contribution of each process parameter in a SF laminating unit employing TM OA, indicating a paucity in the literature. TM is utilised to quantify the amount of controllable factors with a limited number of experiments run (Hamzacebi, 2016). This research aimed at using TM OA to optimise the SF laminating system to improve the adhesion strength (AS) between polyethylene (PE) and polyamide (PA) web substrates. In recent years, SF lamination is preferred over other types of lamination processes since it has a low operating cost with no solvent component retained during processing.

1.2 Problem Statement

The SF lamination process has been shown to be significant in enhancing barrier properties and sustainability of packaging films. For food packaging films, such attributes are achieved through a two-component polyurethane (PU) adhesive system which facilitates adhesion. However, the problem of poor adhesion and inconsistent strength during SF lamination of PE and PA have been reported. After the SF lamination process, 35% of 2.3 tonnes of PE and PA film that was laminated exhibited poor adhesion. Moreover, these deviations have resulted in the reduction of production of PE and PA laminates. If SF lamination design parameters are not controlled to improve process efficiencies, there will be a loss in the quality of the product, and profitability will be pretentious. Thus, this research investigates the effect of each process parameter on AS of PE and PA films, and hence proposes the development of a Taguchi based model which can be used to optimise SF lamination to maximise the output.

1.3 Research Rationale

Low adhesion between two contacting surfaces of film would result in poor material of construction for multilayer composite structures.

1.4 Research Questions

1) How do lamination process parameters such as application weight, temperature, and film tension affect adhesion strength?
2) What effect does the Taguchi method possess in reducing process variability in a solvent free laminating system?
3) What contribution does the Taguchi quality loss function (QLF) have in maintaining the adhesion strength target value?
4) What polynomial regression satisfies optimum operating conditions for a solvent-free laminating system?
5) How will the combination of the Taguchi method and process modelling reduce the plastic carbon footprint?

1.5 Hypothesis

Lower corona treatment (dyne/cm) and inconsistent adhesive distribution during solventless (SL) lamination of PE and PA materials will result in poor AS, thus applying the TM to quantify the contribution of each design parameter during SF lamination is crucial. In addition, a poor TT and RT will result in poor cleavage and shear stress between two contacting surfaces.

1.6 Aim and Objectives

The aim of this study was to model and optimise a SF laminating system using TM OA to enhance adhesion between PE and PA web films. This includes application of signal-to-noise (S/N) ratio to quantify the most significant design parameters within the system.

The objectives of the study were to:

1) Study the effect of application weight, temperature, and film tension on the bond strength of PA and PE material.
2) Recover PE and PA laminate in order to maximise productivity of a solvent-free laminating unit.
3) Optimise solvent-free laminating using the Taguchi method and identify controllable parameters that minimise the effect of noise factors (NF).

4) Evaluate the significance of data in driving process improvement while increasing process profitability.

1.7 Significance of the Study

Although there are studies performed focusing on the capabilities of different lamination technologies within the flexible packaging industry, there is a dearth of research focusing on modelling and optimisation of a SF lamination system using the TM OA. This research project will provide a reference data set and a strategic foundation baseline study in quantifying the most significant design parameters during SF lamination of PE and PA.

1.8 Delineation of the Study

In outlining the scope of this study, this project did not focus on the following aspects:

1) Rheological behaviour of the adhesive system at molten state;
2) Optimisation using response surface methodology (RSM) techniques, such as: Box–Behnken design (BBD), central composite design (CCD); full factorial (FFD), and D-optimal designs;
3) Hydrophilic and hydrophobic nature of polymers during the curing process.

Therefore, in relation to the study's objectives, the research focused on modelling and optimisation of a SF laminating unit using TM OA to improve AS. Experiments are only conducted on an industrial scale in a batch production process. The PE and PA film are the only two web substrates used as model systems bonded together by a PU adhesive.

1.9 Layout of Chapters

This thesis consists of the following seven chapters:

- **Chapter 1: Introduction**

Chapter 1 introduces the topic under investigation and provides contextual information on the different types of adhesive systems that can be used to facilitate bonding in the flexible packaging industry. The significance of this research project and its rationale are explained, along with the research questions, aim, and objectives. The scope of the study is delineated, and the forthcoming chapters are outlined to provide the reader with a 'road map' of what is to come.

- **Chapter 2: Literature Review**

Chapter 2 focuses on the background of process optimisation of a SF laminating unit utilising the TM OA. In addition, the multivariate and univariate process optimisation approaches are both discussed in more detail. Some of the mathematical models and S/N ratio developed by the TM are illustrated in this chapter. Moreover, PU and epoxy adhesives are illustrated in the chapter with their chemical and physical nature during processing. The background and previous literature on the subject matter have been described in order to provide an overview of the research topic and establish the knowledge gap that needs to be filled by this project. Different methods of designing experiments have been discussed in detail. Chemical properties of thermoplastics and thermoset polymers are shared in this chapter. The application of different adhesive technologies within the flexible packaging industry are also demonstrated, along with a review of their pros and cons. Morphology and rheological behaviour of PE and PA films have been examined since they are used as the model system in this study.

- **Chapter 3: Experimental methods, optimisation and lamination design parameters for bonding PE and PA materials**

Chapter 3 details the method and procedures utilised during the thesis. This chapter consists of four sections. The first phase of this chapter focuses on the process parameters within the SF laminating system. Some process optimisation techniques are considered in this section. In addition, the second phase stipulates the method and procedures for modelling and optimising the SF laminating unit. Strategic planning of this experiment is covered in the third phase. The experiments were carried out in a batch industrial production scale. Prior Gage repeatability and reproducibility was performed to minimise process variation. Different testing methods and equipment utilised for measurement are detailed in phase four of this chapter.

- **Chapter 4: Results and Discussion**

Chapter 4 describes the methodology of the experiment conducted as well as the material utilised during experimentation. The results of this project are presented and discussed in detail.

- **Chapter 5: Quality loss function, selection of lamination adhesive, and effectiveness of the model**

The accuracy and precision of the result developed during experimentation are tested and verified in this chapter. The optimum operating condition and process modelling using the experimental data are discussed. The distribution curve is presented to understand the behaviour of the system.

- **Chapter 6: Solvent-free economic feasibility and value stream mapping**

Chapter 6 describes the quantity and cost of resources utilised in this study. The current and future value stream mapping are demonstrated, and the profitability and loss analysis are conducted using a lean methodology.

- **Chapter 7: Conclusions, recommendations, and contributions**

This final chapter describes the critical analysis of the experimental result. Based on the insights of the study, recommendations are made for future research. The contribution of the study is also discussed.

Figure 1.1 below provides a flow diagram of the overall steps followed in this thesis. These are critical steps crafted to achieve the objectives of this study.

Critical Steps of the Thesis

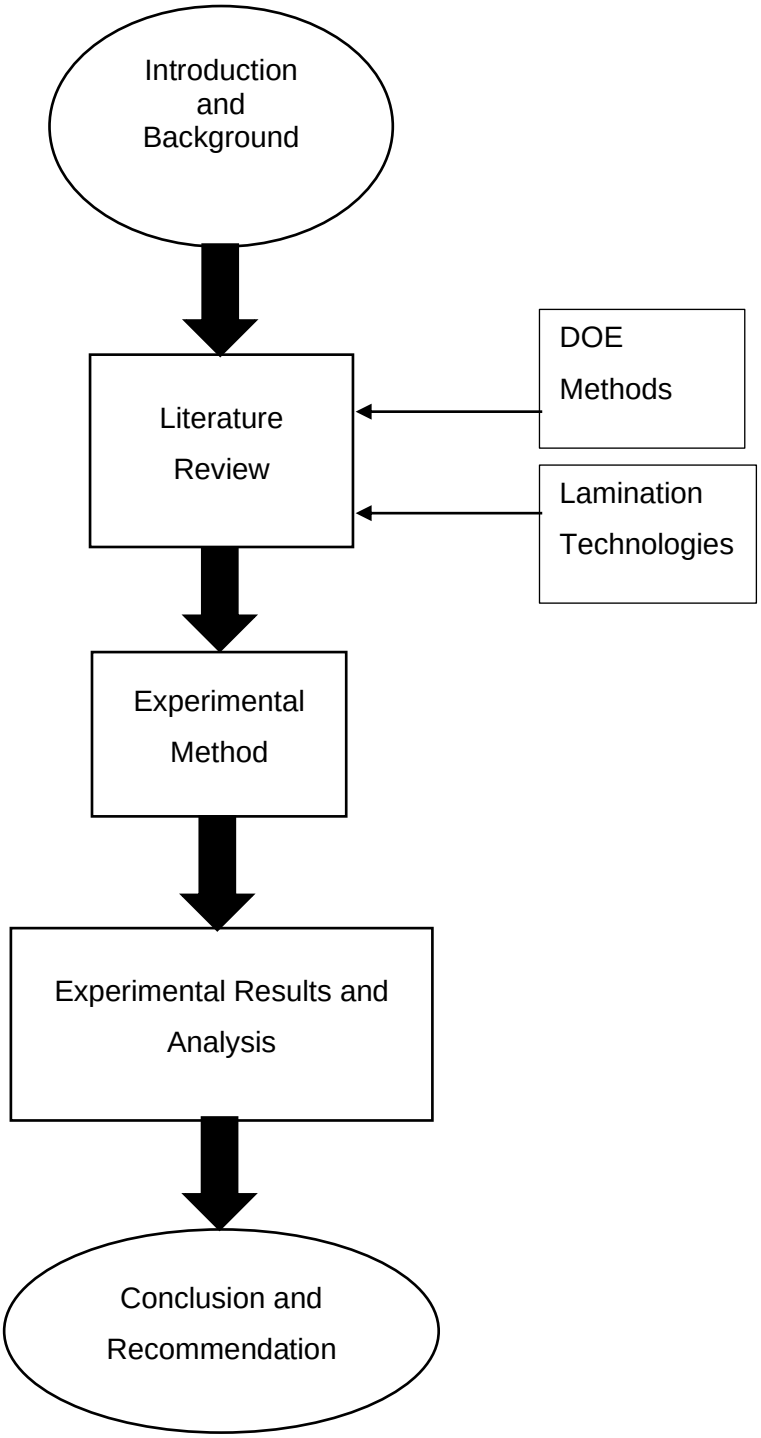


Figure 1.1: Map and flow diagram of the overall thesis

CHAPTER 2

LITERATURE REVIEW

Chapter 2: Literature Review

2.1 Introduction

The previous chapter introduced this study and all the main elements of the research process pertaining to modelling and optimisation of a SF laminating unit. The current chapter presents the literature review, focusing on several pertinent topics to shed light on the research questions and achieve the aim and objectives of the study. The structure of the chapter is as follows. The first section after the introduction focuses on the QLF. Thereafter, two common designs developed by TM are discussed. A number of other designs are also explored, including the CCD, fractional factorial method, full factorial approach, BBD, and D-optimal design. Attention then shifts to lamination technologies and adhesive systems for bonding substrates; extrusion coating; the lamination material model system; and sustainability and demand of high barrier packaging material. Also discussed is effective barrier polymers and structures, and PE used as a barrier material. The chapter closes with a brief summary that recaps the main points of the chapter.

The flexible packaging industry has been experiencing rapid technological changes due to constant transformation in material design and construction to improve machine efficiency while minimising waste. Many organisations have managed to accomplish high productivity while reducing waste through the implementation of process modelling and process optimisation. The process optimisation procedure is based on improving the efficiency of systems by manipulating the controlled variables to achieve the best combination of input that maximise the output response (Hamzacebi, 2016; Gaitone et al., 2006). According to Asghar et al., (2014), process optimisation techniques can be classified into two categories, namely: multivariate and univariate. *Multivariate* optimisation is based on testing different design parameters simultaneously, while *univariate* optimisation focuses on testing each parameter at a time within the system (Asghar et al., 2014). The multivariate approach of optimisation has many advantages over the univariate approach, which includes, but is not limited to, investigating interaction and linearity of equation (Witek-Krowiaka et al., 2014). One of the disadvantages of the univariate approach is that the technique requires a large number of experiments which are costly to conduct. Due to time and cost constraints, the DOE has become an efficient tool in driving multivariate optimisation of design parameters (Jiju, 2015).

The use of DOE to optimise processes can be traced back to the 1920s when FFD was established by Sir R.A. Fisher (Davis & John, 2017). In 1951, Box and Wilson laid a solid foundation for RSM with CCD (Witek-Krowiaka et al., 2014). Over the past decade, DOE has

been effective in modelling and optimisation of processes to reduce variation and improve process capabilities. DOE focuses on rearranging process parameters to achieve optimum operating conditions based on the desired response. The DOE approach facilitates the use of three or more levels of process parameters to predict a non-linear equation with integration and interest in the interaction of variables at different levels. Due to the result achieved in the past decades with the old traditional FFD, the DOE methodology has gained significant recognition in optimising production processes (Asghar et al., 2014; Giunta et al., 2003; Yolmeh & Jafari, 2017). Modern DOE techniques that have been utilised for decades include CCD, D-optimal, and TM (Giunta et al., 2003).

TM can be described as a statistical approach to studying variation of design parameters within the industrial process (Davis & John, 2017). The TM was established in 1949 as an important tool for driving robust design in industrial experimentation. In TM, variation is reduced through a quadratic loss function technique known as signal-to-noise ratio (S/N). In this approach, the quality of the product is built on the product as in the design stage of the product. This method is still regarded as the best technique for driving process improvement of quality and reduction in operating cost. The method combines both the engineering approach and statistics. This technique facilitates the reduction in production time and cost through testing combination and interaction between different process parameters to reduce variation (Kondapalli et al., 2015). The use of OA by TM during industrial experimentation has become more attractive in the last four decades than any other process optimisation technique due to low resources required to perform the experiment. Thus, the TM application has become popular in many industries, including the petroleum, chemical manufacturing, as well as textile and packaging industry. However, the electronic and automotive industry is leading in terms of applying TM techniques and the DOE methodology (Kondapalli et al., 2015). In the packaging industry, the blow moulding side has been utilising the TM approach more regularly compared to the flexible sector.

In the TM approach, mathematical models are developed to measure the effect of input variables to output variables to create a less sensitive production process to variation. TM uses OA to quantify the number of experiments to be run as trials, and the number of experiments required depends on the number of design parameters involved in a system and their levels (Aamir et al., 2020). The DOE by TM ensures that at all stages the number of experiments performed provides sufficient information for both a quantitative and qualitative approach for efficient production and in cost effective ways (Davis & John, 2017). To study the effect of the independent variable to the responses and their combination, TM applies OA (Mikovic et al., 2014). With TM OA, linear equation and graphs are utilised to assign

design variables and their interactions accordingly on the OA. However, using linear equation alone is insufficient when investigation is based on the interaction of the variables, as linear graphs cannot simulate the exact structure of the design. Thus, the use of OA by TM becomes ideal for studying interaction.

Firstly, the TM approach in DOEs is facilitated by the S/N ratio which quantifies the contribution of design parameters and optimum operating conditions using OA (Jiju, 2015). The use of the S/N ratio by TM combines both the output variable and mean response in one performance measure while this is not the case in traditional DOEs. The analysis of output response and mean response are conducted separately on the old traditional DOE. The traditional DOE approach uses regression models with confidence levels to estimate the behaviour of a process at a given operating condition.

Secondly, traditional DOE investigations are not interested in the effect of NF on the output response, while this is the case in the TM approach which desires to optimise the process and improve the quality of the product. Hence, each DOE approach has its own requirement, thus understanding each DOE method is crucial prior to applying these tools. Considering a system with six parameters, the BBD would require 54 runs to be conducted, while the CCD would need 80 experiments to be conducted. Thus, in a large manufacturing unit, the DOE technique through these methods becomes very costly. TM, on the other hand, requires a minimum number of experiments to be studied at low cost with minimum effort, thus TM has delivered satisfying results in designing and optimisation of the production process in a number of industries (Gaitone et al., 2006).

The selection and application of DOEs depends on the following: magnitude and size of the problem, resource availability, cost and time constraints, and validity of statistics. The TM approach to DOE is advocated by the S/N ratio to quantify the contribution of each design parameter to the output variables and achieve optimum operating conditions (Mikovic et al., 2014). S/N ratio facilitates the performance of a system to minimise the impact of NFs on the output response. NFs are process variables which are uncontrollable within a system. DOEs have been an effective and efficient method in many manufacturing industries for experimentation and for solving complex problems. Figure 2.1 below demonstrates some of the most used DOE approaches within manufacturing industries:

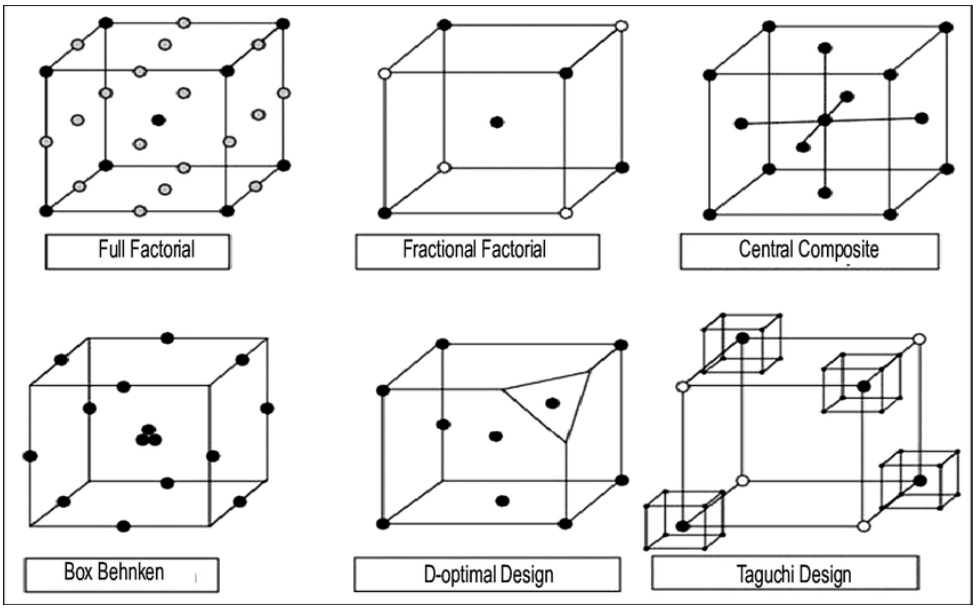


Figure 2.1: Variate DOEs found in practice (Veldhuis et al., 2016)

Although each structure in Figure 2.1 depends on the quantity of design parameters involved in a system and their level, coordinate points can be different for each design. The interaction and combination of variables also affect the shape of the DOE applied in studying variation.

According to the TM approach, process optimisation is based on three phases, namely:

- System Design

The system design is based on identification of the most significant process parameters and their operating levels.

- Parameter Design

Generally, the nominal settings of the design parameters are determined by parameter design. In this approach, experiments are performed to determine the optimal settings points of the design factors while reducing sensitivity caused by NF.

- Tolerance Design

This type of design is aimed at identifying the tighter tolerance limits of each parameter design. The tolerance design focuses on reducing the gap between the lower specification and upper specification so that the quality of the product can be enhanced while improving process capabilities. The tolerance design method provides a very robust design with limited variability on the output response.

2.2 Quality Loss Function (QLF)

QLF can be defined as the total degree at which the output response varies from the desired process mean. According to the TM, when the actual process mean deviates from the target, there will always be a loss that will result in a defect that will lead to deficiency in the performance of the product (Sharma et al., 2007). In many cases, the TM utilises the QLF to quantify the deviations between the experimental error and the desired output response target. With TM, the QLF characteristics are altered by the S/N ratio. The QLF by TM has been used in many manufacturing industries to optimise a multi-component quality characteristic. The method has proven to be proficient in reducing process variability (Gaitone et al., 2006). The robustness of the design in TM is officially accomplished when the process mean is exactly as the response, as demonstrated in Figure 2.2. The output response L(y) should be the same as the process mean (m).

Figure 2.3 demonstrates a traditional approach in monitoring process variation known as "step function", where lower limit ($m-\Delta_o$), upper limits ($m+\Delta_o$), and process mean (m) are illustrated. The step function suggests that as long as the output response within a system is still between the lower limit and upper limits, there will be no losses on the cost and product performance.

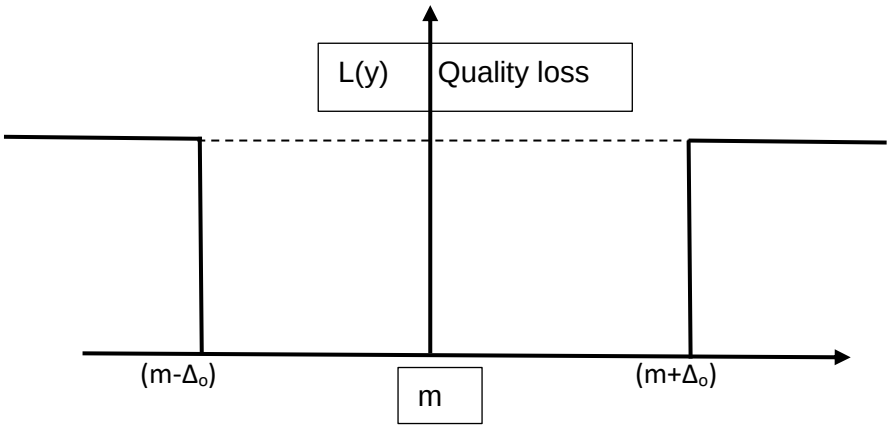


Figure 2.2: Step function

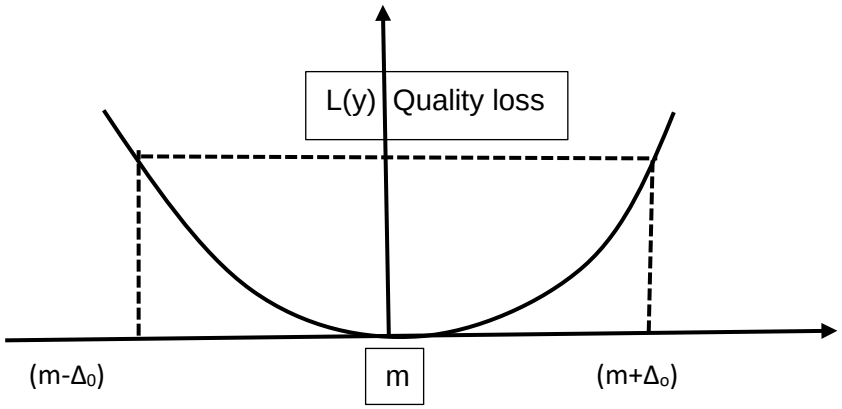


Figure 2.3: Quality loss function

In a quadratic relationship, the QLF by the TM can be expressed according to the equation below:

$$L_{(y)} = K(y - m)^2 \tag{2.1}$$

The quality loss represented by equation 2.1 above demonstrates *the nominal the better* according to the TM category of quality function. The L on the equation represents a quality loss at a certain parameter (y) condition; k is the constant which relates to the number of defects generated as a result of the y deviation to the process mean(m). In cases where a large group of population and experimental data have to be considered for the QLF, the equation below can be utilised.

$$L = k[\sigma^2 + (\mu - m)^2] \tag{2.2}$$

L represents the total average quality loss for a number of samples in an experiment conducted with $(\mu - m)^2$ to signify the deviations between the expected output response (μ) and the desired target (m). The total average variance of the parameter (y) is represented by σ^2 with a constant k.

2.2.1 Taguchi signal-to-noise ratio to quantify the output response

TM uses the S/N ratio to study variability on the response variable. The S/N ratio reduces variation in a production process by determining control factors which are sensitive to the noise and minimising the effect of uncontrollable factors (Philiphall et al., 2014). The TM developed three ways of categorising quality: *the lower the better, the nominal the better*, and *the high the better* (Sharma et al., 2007). Each quality category is expressed below:

The S/N ratio with *the lower the better* characteristics is evaluated using the equation below:

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n} y_i^2\right) \quad (2.3)$$

The following equation demonstrates *the nominal is better* characteristics:

$$S/N = -10\log_{10}\left(\frac{1}{ns}\sum_{i=1}^{n} y_i^2\right) \quad (2.4)$$

The equation (2.5) below represents the S/N ratio with *the higher the better* characteristics:

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n} \frac{1}{y_i^2}\right) \quad (2.5)$$

Where y_i is the experimental response variable, s is the variance of y, and n is the number of tests in the trial. For the purpose of this study, equation (2.5) was used because high bond strength is required between the two films. To improve the quality standard in any organisation, the TM approach uses a performance indicator, namely: S/N ratio, analysis of variance (ANOVA), and OA. The S on the S/N ratio stands for standard deviation, while the N stands for the total number of experiments on the ortho. However, for the purpose of this study, the focus is on the application of OA in flexible packaging.

Figure 2.4 below demonstrates process variables that need to be considered during the application of TM.

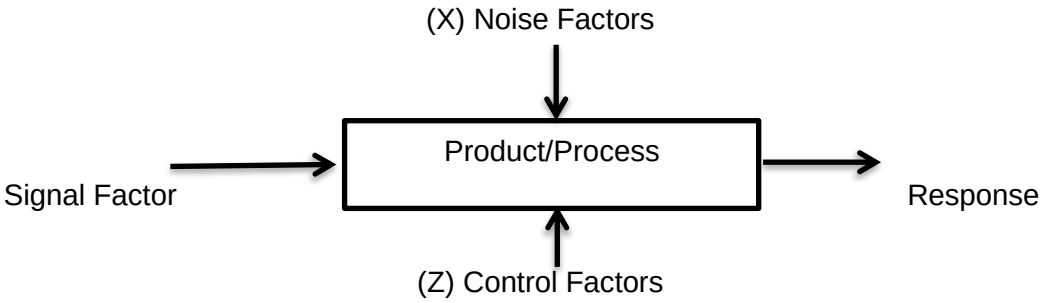


Figure 2.4: Robust design process

The NF are variables that cannot be controlled within the system; this includes factors such as environmental moisture and ambient temperature. The values of NF can be calculated by using the input S/N ratio to the output S/N ratio. A simplified approach when applying the TM can be summarised according to the steps in the flow chart shown in Figure 2.5:

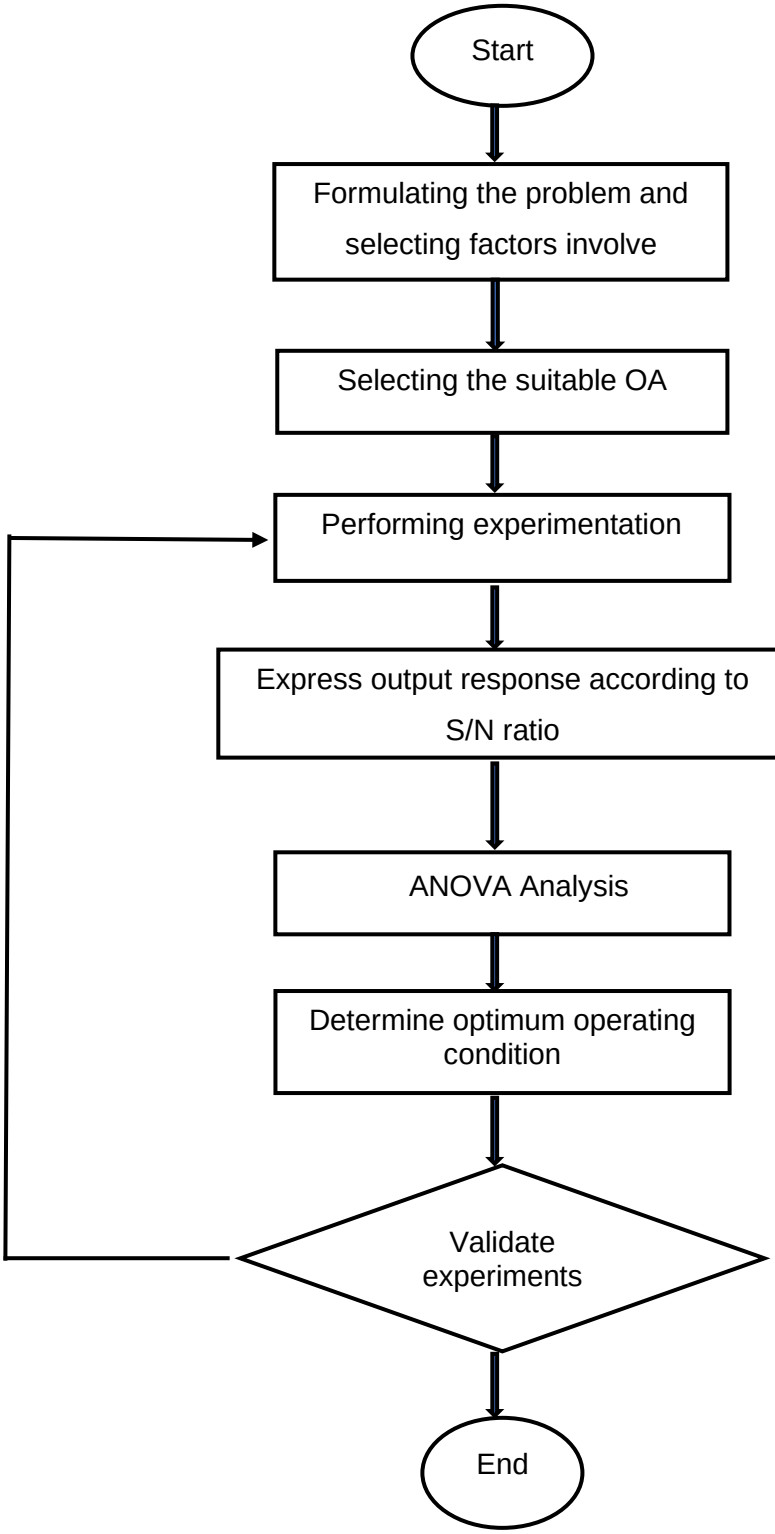


Figure 2.5: Simplified Taguchi method process flow (Yosoff & Ramasamy, 2010)

Controllable factors are variables that can be manipulated in the system by turning and adjusting some components of the machine/system. The response output can be defined as the process out of the system which depends on the interactions between input variables.

2.3 Two Common Designs Developed by TM

- Static response design

A *static response design* incorporates distributing consistent output responses during operation. This design approach aims at accomplishing fixed process parameters with no degree on the QLF (Wu, 2015).

- Dynamic response design

The *dynamic response design* is based on process optimisation with the aim to achieve the best operating levels by manipulating the controllable variables to find a relationship between the input and the output response.

2.4 Central Composite Design (CCD)

The RSM with CCD can be described as a systematic approach based on mathematical models and statistics to optimise processes (Asghar et al., 2014). In RSM, CCD quantifies the number of experiments required for process optimisation purposes. This method is ideal for second order models (Ahmadi et al., 2005). The technique requires a large number of experiments. The CCD approach is applicable at a later stage of RSM, after all critical parameters are known in the process and parameter screening has been done. In engineering, RSM is used to optimise systems and design new processes within the manufacturing industry. The RSM can also be utilised to centralise unknown variables prior to running experiments (Asghar et al., 2014).

The RSM was established in 1951 by Box and Wilson. This approach has been widely used globally in manufacturing for optimising design parameters to improve the output response and DOE (Prasad et al., 2012). The RSM and CCD requires that the region of interest and the region of operability must be identified prior to carrying out optimisation. The identified disadvantage of RSM is that the data derived from the experiments are fitted on the polynomial second order model, while not all systems and processes follow the quadratic equation model. In CCD, it is crucial to quantify the distance from the star points to the centre. This distance is recognised as alpha, and so quadratic equations are developed.

When the experiment is conducted under CCD, the following are considered

- Direct effect of parameters.
- Interaction of process parameters.

- Curvilinear.

CCD, also known as the Box-Wilson design, is a statistical method based on using an empirical model and mathematical equation to design experiments (Prasad et al., 2012). The multivariate CCD and RSM optimisation approaches have been used in many industries to fit second order or quadratic models with low order polynomial, as represented in the equation below:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_{ii}^2 + \sum_{i=1}^{k} \sum_{i \neq j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (2.6)$$

The factors in the above equation are presented below in the following manner:

- Y is the output responses.
- β's is the coefficient of linear(i), quadratic (ii) and interaction (ij).
- K quantity of independent variables.
- X_i and X_j demonstrates the level of each independent variable.
- β_0 is the constant term.
- ε is the output response.

Generally, there are groups that are considered when designing experiments with CCD through RSM. Their categories are described below:

- Fractional factorial (2^k) at two level factor.
- Replication through centre points and prediction of experimental error.
- Establishment of quadratic equation with 2^k axially points.

2.5 Fractional Factorial Method in DOEs

FFD was developed in the 1920s by Sir Fisher. The technique has been used in many manufacturing facilities for screening and optimisation of production processes (Chollom et al., 2018). The screening technique is performed to identify the most critical variables within the system. In this case, the design parameters are screened at two level conditions. These levels incorporate the low (-1) condition and upper (+1) limits. The number of experiments to be conducted on fractional factorial are less when compared to full factorial (Mee, 2009). However, when applying the FFD, process parameters must be re-arranged first in order to understand the purpose of each parameter within the system to screen critical factors properly and avoid incorrect assumptions (Miryam, 2014). When considering the fractional

factorial approach in designing experiments, it is important to note that not all interaction between variables are tested, since not all factors are significant (Miryam, 2014).

The general formula in estimating the quantity of experiments to be conducted in a CCD is as follows:

$$N = k^2 + 2k + n \tag{2.7}$$

k = represent the number of design parameters within the system.

N = Total number of experiments.

n = represents the quantity of replicates.

There are two experimental designs under RSM:

- CCD, and
- BBD.

There are three types of CCD:

- Central composite face (CCF) centred.
- Central composite circumscribed (CCC).
- Central composite inscribed (CCI).

The designed model under DOEs can fit a linear, cubic faction, and quadratic equation. When conducting the CCI design, the upper (+1) and lower (-1) limits become the axial points, while a factor factorial approach is created within the system (Prasad et al., 2012). The area of interest for the CCF methodology is on all the points that are located within the specified boundaries. In the CCC design, the upper and the lower limit of each process parameter are far apart to form axial points, and the star points have the capability to trigger the specification of all parameters involved in the design in terms of establishing the lower and upper limits (Dutka et al., 2015). The obtainability of curvature within the system does not justify that all systems with the phenomenon are second order polynomial models. The structure and the shape of the CCD discussed above is illustrated in Figure 2.6 below:

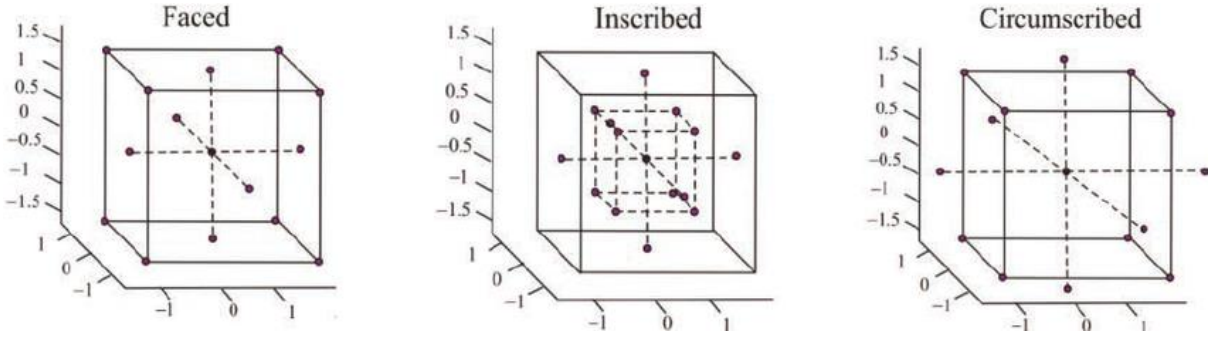


Figure 2.6: Cube plot of (a) faced-centred, (b) inscribed, and (c) circumscribed design (Prasad et al., 2012)

2.6 Full Factorial Approach in Designing Experiments

FFD is another statistical approach where a combination of controllable variables are tested with their relationship against the output response (Prasad et al., 2012). One of the advantages of the FFD is that all possible combinations are verified; however, this design technique not only requires a lot of effort, but it also becomes costly for most manufacturing organisations to perform as it is a very time-consuming experiment. In FFD, the number of experiments required for each design depends on the quantity of factors available within the system and the level of each parameter to be studied. Generally, a FFD technique is normally used to study design parameters at two levels; however, in a case of investing parameters above two levels, this approach becomes extensive, thus the BBD is applied. In FFD, the quantity of experiments to be conducted can be calculated using 2^K, where k is the levels of parameters to be studied during experimentation (Yasemin, 2013). The FFD approach is based on practical models or mathematical equations produced by fundamental principles of the experimental design. The FFD system is a statistical method which is key in planning industrial experiments with dependent and independent variables at multiple levels. Although the FFD technique can be complex, it is ideal for optimising systems with multivariable systems (Kordkandi & Forouzesh, 2014).

2.7 Box-Behnken Factorial Design

BBDs have been found to be more efficient compared to other types of designs, especially because they require less experiments than other designs, such as the CCD and TM.

However, it cannot be utilised when performing a multi-level parameter design, hence it is mostly used to study parameters at a level that is less or equal to 3 (Hsu & Su, 2019). With only three levels of each design parameter, the BBD can derive a polynomial equation, where in the case of CCD, up to five levels of each parameter would be required (Chollom et al., 2018).

In BBD, DOE involves the planning of experiments to find the cause and effect between design parameters in relation to the output response variables. Through this approach, the DOE has been used in many operations since it reduces the number of experiments to be conducted for the study. Under the response surface methodology, there are other techniques such as the FFD, BBD, D-optimal, and CCD, which are utilised to drive modelling and optimisation of the production process.

2.8 D-Optimal Design

The D-optimal approach in DOEs is based on the application of a computer aided design to select the best optimal combination of designs with variate experimental matrix (Nguyen et al., 2015). These matrices include dispersion and informative design, design matrix, and the candidate set matrix. The method utilises different criteria to predict different variances. The D-optimal design technique requires less experiments than classical designs, such as full factorial and fractional factorial (Tazliqoh et al., 2018). One of the advantages of this method is that higher order polynomials and linear functions can be generated; however, in cases where certain design parameters are not significant during experimentation, they can be easily eliminated in this approach that reduces the number of experiments to be performed.

Table 2.1: Previous scholars who have used Taguchi orthogonal array

Reference	DOE tool used	No. of parameters	Parameter Level	Topic
(Borges Silva et al., 2014)	Taguchi: L_{16}	7	2	An Application of the Taguchi Method (robust Design) to Environmental Engineering: Evaluating Advanced Oxidative processes in Polyester-Resin Wastewater Treatment.
(Yosoff & Ramasamy, 2010)	Taguchi: L_{27}	9	3	Selection of RGP Optimization Variables Using Taguchi Method.
(Wu, 2015)	Taguchi: L_{18}	8	3	Robust Design of Mixing Static and Dynamic Multiple Quality Characteristics
(Gaitone et al., 2006)	Taguchi: L_9	4	2	Multi-Response Optimization in Drilling Using Taguchi's Quality Loss Function`
(Mikovic et al., 2014)	Taguchi: L_9	4	3	Application of Taguchi Methods in Testing Tensile Strength of Polyethylene.
(Asghar et al., 2014)	CCD face-centred Taguchi: L_{16}	4	2 3	A Comparison of Central Composite Design and Taguchi Method for Optimizing Fenton Process
(Aamir et al., 2020)	Taguchi: L18	3	3	Optimization and Modelling of Process Parameters in Multi-Hole Simultaneous Drilling Using Taguchi Method and Fuzzy Logic Approach

2.9 Lamination Technologies and Adhesive System for Bonding Substrates

The different packaging structures manufactured by the flexible packaging industry would not be possible without the modern adhesive system (Petrie, 2011). These systems, which consist of PU adhesives, are critical in ensuring that certain mechanical properties are achieved within the film, especially when a single layer of a film cannot achieve the required performance, that's when a composite multilayer of films is achieved by applying PU adhesive between two or more polymers. Lamination of multilayer substrates has proven to be the key in achieving properties such as high bond strength, high tensile strength, and high gas permeability, which results in a very complex lamination. Different lamination technologies are available for various applications. Lamination adhesive technologies are categorised according to the following types:

- Solvent-based.
- SF lamination.
- Waterborne.
- Hot melt.

Adhesive lamination involves application of adhesive systems to achieve multilayer structures with good mechanical properties on the laminate. The importance of process optimisation and process modelling by DOE is significant, as was discussed in the above section. However, in the current decade, thermoset and thermoplastic polymers are also required to meet certain processing conditions and environmental requirements. In that case, polymers are required to be recyclable, compostable, and biodegradable, hence it is critical to choose an adhesive system that meets these demands.

2.9.1 Polyurethane characteristics and adhesive properties

PU is classified according to performance and capability, which includes SB adhesives, SF adhesives, and WB adhesives (Davis & John, 2017).

The equations below Figure 2.7 demonstrate how the reaction between two-component (resin and hardener) PU adhesives occur to create adhesion.

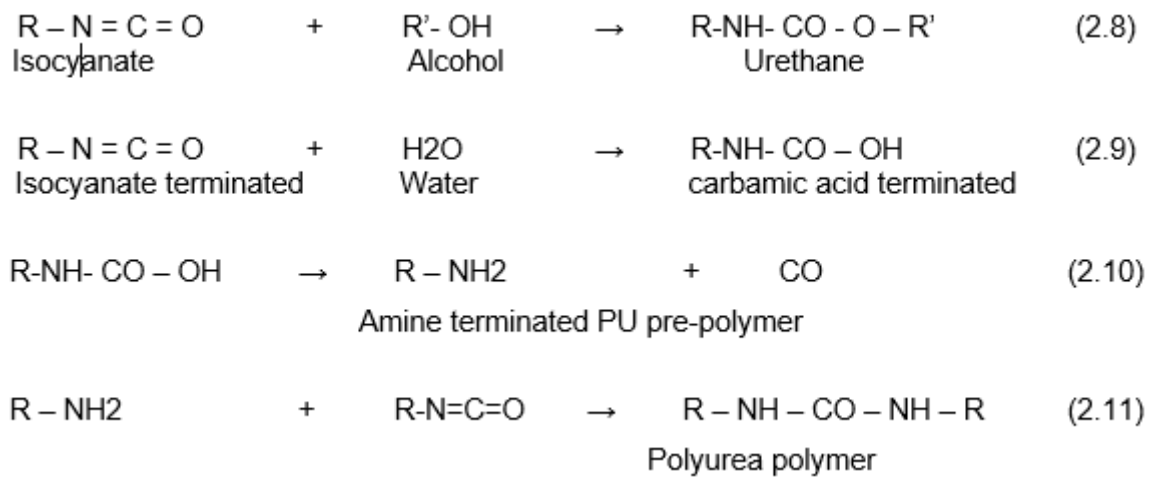


Figure 2.7: Polyurethane adhesive curing mechanisms during lamination (Toenniessen, 2018).

Table 2.2 below demonstrates the capabilities of common PU adhesive systems used in the flexible packaging industry to bond multilayer polymer composites during lamination. These are critical characteristics which justify the use of each adhesive technology when bonding different corona treated films. The use of each adhesive system depends on the type of product to be packaged inside, thus the below parameters need to be considered before lamination. For food packaging, SF lamination is always considered since there is no solvent retained during its application. The quantities of each adhesive required to initiate proper bonding are also detailed in Table 2.2.

Table 2.2: Different polyurethane adhesives with their capabilities during flexible web lamination

Adhesive Parameters	Polyurethane Adhesive		
	Water-Based	Solvent-Based	Solvent-Free
Initial Bond (g)	300	300	20
Port Life (hr)	8	8	0.5
Coating Weight (gsm)	(1.0 - 2.0)	(1.5 - 3.5)	(1.0 - 2.0)
Time to slit (hr)	1	2	12
Retained Solvent	Amine Possible	Possible	No
Emission	No	Yes	No
Fire Hazard	No	Yes	No
Cost (adhesive + Energy)			
Lamination Speed	Oven limit	Oven Limit	1500
Energy used	High	High	No

Coating weight 5(CW) is another important property which is critical during adhesive applications as it depicts the amount of adhesive applied. The distribution of adhesive is required to be uniform during SL lamination as this ensures consistent wettability on the surface of the material. If the CW is distributed unevenly, the adhesion and cohesion will be affected because bond strength depends on the amount of adhesive supplied (Ling et al., 2010). The spec of CW during SL lamination is set between 1.5 – 2 gsm. CW lower than detailed on the specification will result in PE/PA substrate peeling off, while high distribution of adhesives will cause blocking. The impact of this will result in poor product quality.

2.9.2 Solvent-based lamination process

The dry bond lamination, also known as solvent-based (SB) lamination, involves the bonding of two or more multilayer construction of films with a PU adhesive which consists of the solvent content during processing. The solvent content is mixed with the adhesive at a ratio which allows easy application of adhesive to the film. The mixture of adhesive and solvent content is transferred through the mandrel roller by rotation. The mandrel roller is employed for application of the adhesive. This is one of the very hazardous and expensive processing approaches since more heat is required in the machine oven to evaporate the applied solvent content. The SB lamination process is undesirable as it releases a lot of harmful gases into the atmosphere.

2.9.3 Solvent-free lamination

Wet bonding or SF lamination is a process by which multiple roll configurations are utilised to apply two-component PU adhesives between two dissimilar polymers during lamination (Wolf, 2010). During this process, isocyanate-based PU group laminate is processed at a controlled temperature to increase the rate of the reaction. At atmospheric conditions, the isocyanate will react with the moisture to yield the desired bond strength. After coating the substrate with application weight, the two bonded films are nipped at a pressure between 2 bar and 4 bar to improve adhesion and surface wettability. The process is more convenient when compared to other laminating technology since minimum energy is required during the process. Another advantage of this type of adhesive is its ability to enhance the machine to coat at high speed while extrusion lamination (EL) runs at a much lower speed (Singha, 2012). The bonding of different polymers using adhesive lamination depicts consistence on the gauge which is not the case on the EL (Wolf, 2010). Although the pot life of SF lamination is limited, this type of lamination is still considered to be environmentally friendly. There is no heat required to evaporate the solvent since this type does not possess solvent.

Figure 2.8 demonstrates a SL laminating unit with multiple rollers used to transfer the required PU adhesive into the film. The diagram also demonstrates the section on the unit at which the treater is applied for easy application of corona discharge to modify the substrate molecular structure.

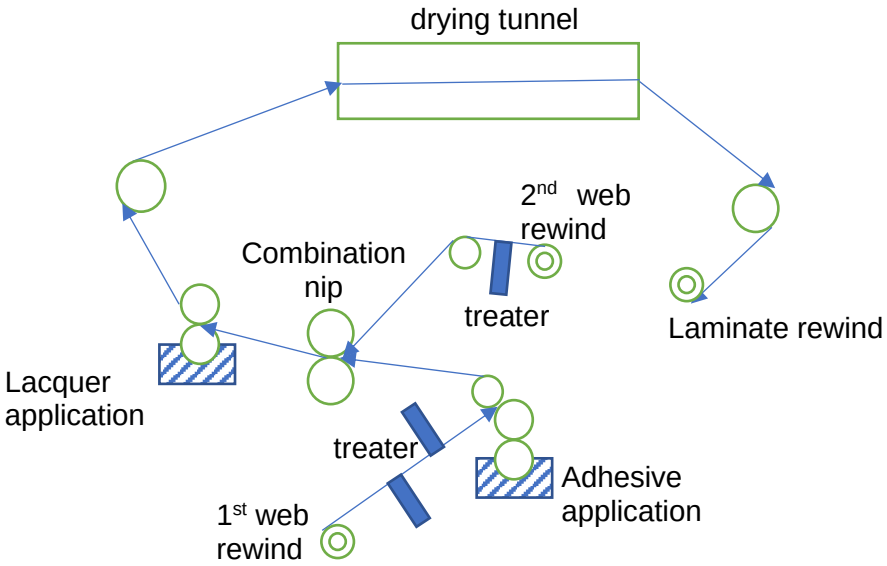


Figure 2.8: Solvent-free lamination process (Singha, 2012)

Figure 2.9 below describes a dry lamination process carried out when a SB adhesive system is applied during lamination. The film is allowed to pass through an oven/dry tunnel at certain temperature conditions in order to remove the amount of solvent trapped in the film during adhesive application. The solvent is removed because it does not take part in the reaction and bonding, but rather acts as a carrier property of the adhesive and improves viscosity. This ensures uniform CW distribution.

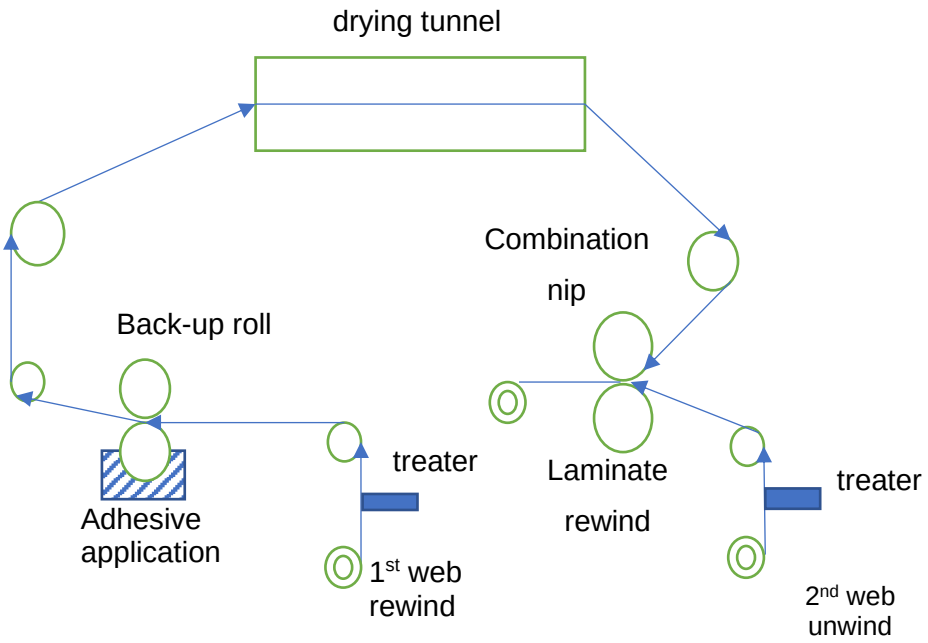


Figure 2.9: Solvent-based lamination process (Singha, 2012)

The PU adhesive type can be categorised into two forms, namely: aromatic and aliphatic isocyanate-based adhesive. The *aromatic* adhesive type is utilised when there is a need for low migration on the film. It also provides a very good work of adhesion and cohesive forces which delivers high bond strength. The aromatic SL adhesive has played a key role to substantiate its need for high heat seal strength, good AS, and product resistance. At the initial stage, the aromatic PU adhesive has proven to provide zero green strength. *Green strength* is the amount of force demonstrated by substrate which keeps the two polymers together. Some of the properties of the *aliphatic isocyanate-based* adhesive are similar to the aromatic adhesive type, but the former costs more and possesses a very low migration compared to the aromatic type (Markwart et al., 2019). Additionally, the aliphatic adhesive cannot provide the same intermolecular forces as aromatic adhesive.

2.9.4 Two-component epoxies

This section discusses the two most important laminating adhesives in the bonding of polymers that incorporate PU adhesives and two-component epoxies. The PU adhesive was discussed above. Epoxy adhesives possess good mechanical properties, and some of their advantages include the following (Zarybnicka et al., 2015):

- Excellent chemical resistance to vibration and shock.
- Good bond strength.

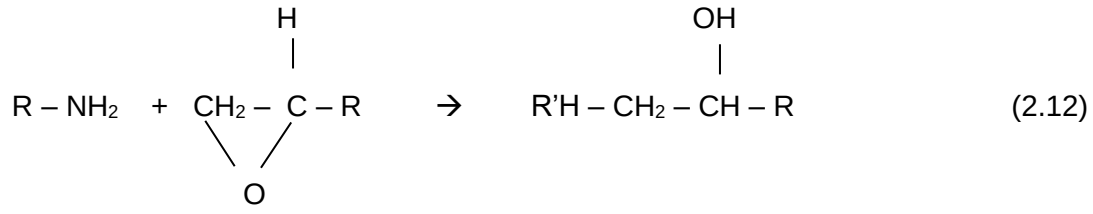
- Uniform stress distribution
- Good stability in mechanical properties.

The above properties distinguish the epoxies from the other forms of adhesives. Epoxies belong to a certain group of plastic called “thermoset”, which depicts their recyclability since their chemical structure is modified after heating. Generally, plastics are categorised into two groups, namely: thermoplastic and thermoset. *Thermoplastics* are those polymers which chemical structure does not change after they have been exposed to heat, thus thermoplastic can be reversable, remoulded, reshaped, and or reformed back into their original state (Saba et al., 2015). High density cross-links and slippages in thermoset plastic restricts their mechanical properties to weak strength than thermoplastic.

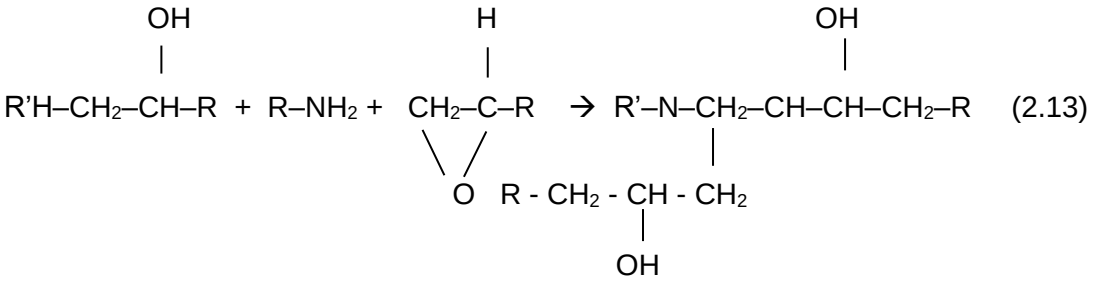
2.9.5 Application of epoxies

Epoxy resin is organic pre-polymer utilised globally in many different industries. Some of the industries that utilise epoxies includes aircraft formulation structures. Epoxies are best described by the chemical formula of COC which is broken by a hardener during a chemical reaction to form a very strong bonding between the films. To achieve this, the molecules of both components are cross-linked and rearranged. After the rearrangement of molecules, a very high strength bond is formed. There are various types of epoxies with different strengths and curing at different temperature. During processing, the room temperature should be kept between 25 ^{o}C and 35 ^{o}C. Anything below these temperatures will result in poor adhesion and cohesive forces between the laminated films. The excellent durability and low volatile properties of epoxies justify their use in the aircraft industry and automotive industry. Thus, epoxies have gained access in many different industries. Low shrinkage and chemical resistance are some epoxy properties.

2.9.5.1 Curing process of the epoxies



(2.12)



(2.13)

2.10 Extrusion Coating

The co-extrusion method involves joining two or more polymers through a feed-block of extruded molten PE with multilayer composites to advance the mechanical properties of the material. Multilayer co-extrusion provides a combination of different properties of multi-layer polymer structures that impact resistance and material toughness. Polymer structures produced by co-extrusion are based on processability and barrier requirement. During processing, a molten PE substrate is applied between two webs through a flat die section to form a single layer. The feed-block/die is pressured with molten material to release the material at a controlled viscosity rate and elastic properties. To ensure consistent flow of output from the feed-block, the gear pump is assembled on the line with thermocouples which measure the temperature distribution of the component. Pressure transducers are installed in the co-extruder design to ensure constant pressure and material flowrates. Different feed-blocks are designed every day to meet the current need of co-extruded polymers. These substrates include polyester film, polypropylene film, aluminium, paper board, and aluminium foil. Unlike injection moulding, the parameters influencing the processability of co-extrusion molecular structures include:

- Layer thickness gauge inconstancies.

- Pressure drop.
- Instabilities on the interlayer.

The material layer thickness inconsistences are more visible during rearrangement of molecules in the elastic layer and viscous encapsulation, while the influence of pressure drop is based on layer placement. The interlayer inconsistences are more evident at higher flowrates. In the flexible packaging industry, there have been limited studies focussing on the rheological behaviour and morphology of co-extruded molecular structures, and this has posed a risk in recent designed feed-blocks since degradation of produced polymers cannot be analysed. Such analysis has been identified to be critical for all material produced during extrusion to understand their strength, especially when these materials are exposed under oxygen and nitrogen atmosphere so that their oxidative properties after co-extrusion can be scrutinised.

2.11 Lamination Material Model System

Lamination of PE and PA are used as the model system in this study to optimise the lamination process using TM. PE is a semi-crystalline polymer with a chemical structure which consists of repeating units such as (- Ch_2 – Ch_2-), with the amorphous phase providing the elastic properties, and crystalline phase providing flexibility integrity. With such properties, PE has become a widely used polymer in the flexible packaging industry. The PE utilised consists of different grades which include *linear low-density polyethylene* (LLDPE) and *low-density polyethylene* (LDPE) at 50:50 ratio. Another grade of PE which is not employed in this blend is called *high density polyethylene* (HDPE). This type of PE grade is only used when a high strength material is required (Siengchin & Abraham, 2012). All different grades of PE exhibit the same identical repeating chemical structure (- Ch_2 – Ch_2 -), however parameters such as MWD, percentage crystallinity, molecular weight (MW), and the number of branches in a chain are critical in differentiating these polymers. These parameters influence the mechanical strength and processability of PE films, with material having high MWD demonstrating excellent rheological behaviour (Al-Attar et al., 2018). This can be due to shorter chains which act as lubrication. Generally, PE is employed globally to protect food during packaging as containers and or as an insulator of electrical cables. The PE material exhibits both liquid and solid like viscoelastic properties. Therefore, its density can be used to categorise different grades and types of PE utilised during blending (Sadiku-Agboola et al., 2010). However, the melting point of each grade is another critical parameter that distinguishes the type of polymer to be used. There is a good relationship between the density of every PE polymer and its nature of crystallinity. Both LLDPE and HDPE have high

elasticity and excellent impact resistance, demonstrating a lamellar and spherulitic morphology structure (Mohammed, 2007). Material with high density continues to depict increases in crystallinity with greater tensile strength and stiffness on the material. High strength provided by HDPE explains why many industries have decided to utilise such PE polymer during processing. Although PE has different types which are categorised by their density, they all show identical behaviour at a temperature below the glass transition (T_g), a more rigid solid like structure is demonstrated, however, at above T_g, the PE demonstrates a liquid state form with an increase in viscosity and reduction in molecular structural integrity. This can be due to the rheological behaviour of these polymers when exposed to heat. Most polymers demonstrate a significant change in viscosity to molecular weight. The movement of molecules on each PE material depends on the molecular structures and branches involved (Hussein, 2003).

In polymer science, the movement of these molecules is known as *reputation*. Double path reputation occurs when a primitive path and a single reputation occur in a polymer. The molecular chain of each and every polymer increases with a rise in MW, while the relaxation time decreases. In this case, the rheological behaviour of each polymer property is critical since reputation of long chains may affect the relaxation time; hence, polymer morphology is key in understanding the materials of construction of every polymer during blending.

PE is one of the highest produced polymers around the globe. Different techniques which utilise diverse manufacturing operations with varying catalysts and monomers are employed to produce ethylene copolymers and other PE resins. Various grades of PE are available with different density and melt flow index's (MFI).

Table 2.3 below demonstrates different grades of PE material with their properties.

Table 2.3: Density and melt flow index of different PE grades (Khanam & AlMaadeed, 2015)

PE Category	Density (g/m^3)	Melt Flow Index (g/min)
LLDPE	0.91o – 0.925	0.2 – 3.0
LDPE	0.926 – 0.94o	0.3 – 2.6
HDPE	0.94o – 0.959	0.1 – 10
HD copolymer	0.96 and above	0.3

Figure 2.10 below demonstrates different grades of PE material. The density of PE composites indicates its crystallinity, while MFI depicts processability and MWD. It is critical for these parameters to be understood before application of any PE materials to avoid degradation during melt processing. With properties such as low COF, chemical inertness, low moisture absorption, and non-conduct of electricity, PE has been utilised in many industries, including automotive, pharmaceutical, space application, and thermal energy storage for the packaging of objects and other substances. The strength of PE depends on the strain, temperature, and the load exerted on the object.

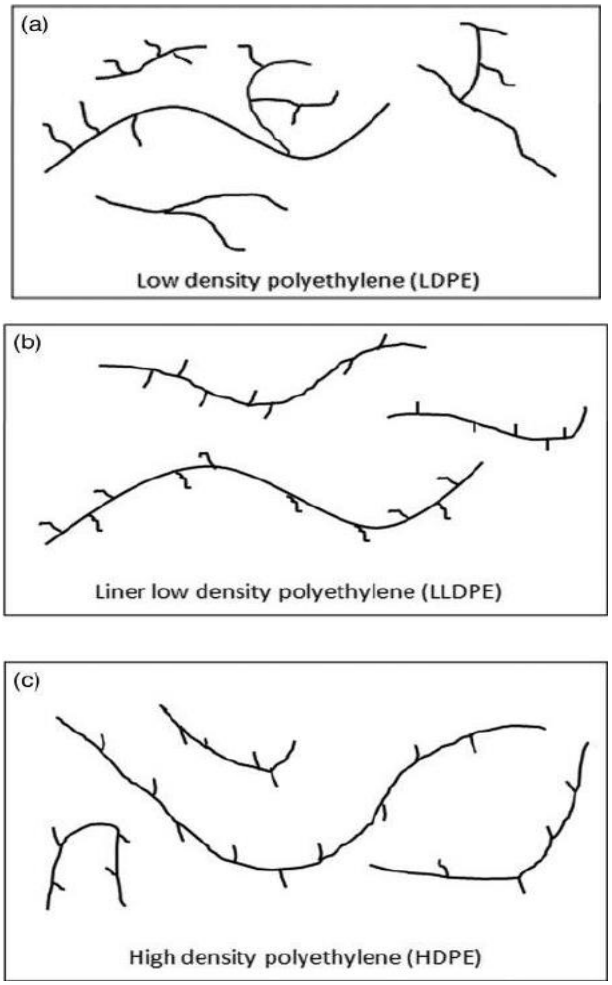


Figure 2.10: Different PE molecular structures (Khanam & AlMaadeed, 2015)

2.11.1 Polyamide mechanical properties and processing

Polymerisation of PA is driven by combining two monomers known as 1,6-Diaminohexane with adipic acid, and with additives added to improve the mechanical and physical strength of the material (Gaymans et al., 1977). PA or nylon matrix consists of five different

components, and these different grades include: Nylon 6, Nylon 66, Nylon 11, Nylon 12, and Nylon 46 (Ali & Kaneko, 2017). The presence of amide in nylon depicts that the film belongs to the amide group and is regarded to be aliphatic with a linear surface. With semi-crystalline structures, PA atoms can form electrostatic forces between molecules of CO and NH bonding, causing high melting points with good chemical resistance, exceptional barrier properties, and stiffness (Samanta et al., 2012). PAs are hydrophilic since they absorb excessive moisture, leading to a hydrolysis reaction. The moisture content absorbed by PA can lead to hydrolysis and molecular weight degradation, hence it is crucial that processing conditions are controlled. The percentage moisture composition absorbed will migrate outside the thin layer of PA and act as a film plasticiser which lowers the glass transition temperature (T_g). Mostly, PA is utilised in a multilayer construction to optimise the material of construction (Jo et al., 1993; Walha et al., 2016; Yeh & Fan-chiang, 1997). It is crucial that PA films are prepared under certain conditions before processing to improve their processing ability. With PA being conditioned at temperatures above glass transition, the film post-crystalises and shrinkages occur (Ali & Kaneko, 2017). The accepted T_g of PA material is 55 ^oC. The properties of these PA materials are derived from the amide group with enhanced intermolecular forces through hydrogen bonding. These forces of attraction provide high barrier properties to carbon dioxide, oxygen, and existing solvents. The PA material also poses high mechanical properties (stiffness and strength) with high toughness and puncture resistance. PA also exhibits exceptional thermoformability and therefore can only be utilised when a complex polymer structure with oxygen barrier properties is necessary. Most PA materials are processed with other multi-complex polymers during SF lamination or co-extrusion. These materials are used with less oxygen demand or sensitive to oxygen food products such as fish, cereals, semi-finished food, smoked fish, and meat products. Table 2.4 lists some characteristics of different PA polymers.

Table 2.4: Different PA polymer characteristics

Parameters	Unit	Nylon 6	Nylon 66	Nylon 11	Nylon 12	Nylon 46
Tensile Strength (T_s)	MPA	83	80	48	60	100
Density (ρ)	g/cm^3	1.13	1.14	1.04	1.02	1.18
Melting Temperature (T_m)	^{o}C	220	255	19o	184	295
Glass Transition temperature (Tg)	^{o}C	47	70	42	97	80
Shore hardness	D	85	88	71	75	85
Coefficient of Friction (COF)	-	1.4	0.55	0.36	0.38	0.45
Tensile Strain @ break	%	100	-	49	51	40
Water absorption	%	1.2	1.6	1.9	1.7	3.7

2.11.2 Polyamide/Nylon 6 barrier properties

The PA6 is one of the most used grades of nylon. It contains six atoms with a structure that is in cyclic form. Some of its advantages include excellent impact strength, good tensile strength, chemical resistance, and abrasion resistance with superior toughness. At high humidity composition, PA has a high intermediate oxygen transmission rate (OTR). High polar chemicals, such as water and methanol PA, demonstrate inferior barrier properties, while low polar substance nylon continues to demonstrate superior barrier properties. The barrier properties of different polymer films are presented in Table 2.5 below.

Table 2.5: Comparison of various barrier properties for different resins

Substrates	Component						
	CO_2 0% Cm^3 /m2 d bar	H_2O 85-> 0% Cm^3/m^2 d bar	C_2H_6 Cm^3 /m^2 d bar	N_2 0% Cm^3/m^2 d bar	Methol 50%rh g/m^2 d	Limonene 50%rh g/m^2 d	Isooctane 50%rh g/m^2 d
LDPE		5	-	-	10	60	35o
EVOH 44%		-	-	-	< 0.01	0.12	0.1
BOPA6	150	30	0.4	12			
PA6	200	35	0.5	14	< 0.01	0.12	0.08

Firstly, when choosing a barrier property for a packaging material, it is crucial to identify the type of product to be packaged. Secondly, it is significant to analyse whether the film used is thermoset or thermoplastics. Thermoset polymers are insoluble and infusible since they are irreversible and can only be bonded through heat or chemical reaction. Whereas

thermoplastics are reversable. Thermoplastics are categorised into crystalline and amorphous thermoplastics. In amorphous thermoplastic, molecular chains are randomly arranged, while in crystalline thermoplastic, molecules are arranged regular. Another third set of polymers, called semi-crystalline, is a combination of both amorphous and crystalline polymers, a very good example of this type of polymer is the polyamide (nylon) and polyester polybutylene terephthalate (PBT). During flexible packaging, each polymer demonstrates very unique properties physically, chemically and electrically, which makes these polymers ideal for the intended use.

2.11.3 The extrusion laminating process

Extrusion lamination includes bonding one or two polymers together to improve mechanical properties (Morikawa et al., 2010). In this case, the bonding is achieved through a molten PE which is pressurised and fed on the flat die. The molten PE applied between different construction becomes a tie layer between the films. During EL, it is crucial to control the neck-in phenomena to ensure that the required molten PE-LD that is extruded is distributed on the film evenly. The length of the neck-in must be controlled to prevent deviation and variability. The elastic properties of the molten PE affect the neck-in length of the PE. One of the disadvantages of the EL is that the process exhibits the edge beads which relate to the edges of the die of the molten PE to be thicker than the central point within the die section (Morikawa et al., 2010). Higher neck in value means high edge beads. To control the EL process, parameters such as processing temperature, take-up velocity, and air gap are critical. When the resin is flattered on both sides, only then the edges beads can be removed. Table 2.6 shows barrier properties of common polymers and their areas of use during processing.

Table 2.6: Common polymers and their barrier properties

Substrates	Light Barrier	Oxygen Barrier	Moisture Barrier	Sealant Layer	Tie Layer
PS	-	polyesters, OPET	ionomers	ionomers	-
PP, OPP	TiO_2 filled polymers	PVDC	PP, OPP	Acid/anhydride grafted polyolefins	LDPE
OPET	-	polyamides (nylon, BOPA)	EVA	EVA	-
HDPE	Aluminium	EVOH	PE (LD, LLD, HD)	LLDPE	Polyurethane
Paper	-	coatings (SiOx, Al2O3, PVOH, nano particles)	PVDC	PP, OPP	-
		Aluminium	-	-	PA, OPA

2.12 Sustainability and the Demand of High Barrier Packaging Material

In the contemporary era, polymers are required to possess certain characteristics, such as high barrier properties and heat saleability. At the same time, these polymeric materials must be biodegradable, compostable, or even recyclable in order to reduce the carbon footprint (Siracusa, 2012). Furthermore, significant focus has been placed on developing efficient packaging that suits the needs of consumers while providing cost effective material. Barrier performance of flexible polymers can be described as the ability of a film to avert diffusion of moisture, gas, and light through the wall of the laminated material (Coltro & Borghetti, 2017).

Barrier polymers are classified into two categories, namely: transparent or opaque. Under *opaque*, metallised polyester and aluminium foil are used, while for *transparency*, material such as ethylene vinyl alcohol (EVOH), PA and HDPE exhibits moisture barrier. Oxygen and moisture transmission have a significant impact on shelf life and packaging material, thus monitoring such parameters is key.

In recent years, plastic material is required to meet a very complex requirement. Polymers that are used to handle food material must be designed in such a way that they can withstand external factors from the environment and molecule diffusion (Feng et al., 2018). Twenty-five percent of plastic waste material is collected in the environment every year, and the plastic carbon footprint has become dangerous for the ecosystem. With packaging film contributing to aquatic and terrestrial pollution in the environment, there is a high demand for a packaging polymeric structure that protects the food organoleptic properties and prolongs the shelf life of the materials. Figure 2.11 further below compares the water permeation rate (WPR) and

oxygen permeation rate (OPR) of different polymers at 25 ^{o}C and at relative humidity between (50% – 100%). The WPR and OPR are the most crucial properties considered when designing new polymer structures.

If the barrier properties are not constructed within the composite structures, then the atmospheric conditions at which the packaging material are produced need to be controlled to avoid degradation and diffusion of particles from the environment to the material walls. Polymer structures are required to be environmental eco-friendly. Subject to the desired properties in films, the most used polymeric films include PE, polypropylene (PP), polyesters (PET), polystyrene, PA, EVOH, and ethylene vinyl acetate (EVA). But PA and EVOH are the most common gas-barrier properties (Mangaraj et al., 2009). Both PE and PP possess good saleability and chemical resistance with water vapour barrier properties, and are therefore used as a sealant layer in many applications. Furthermore, PP polymers are considered to be more transparent and harder compared to PE materials.

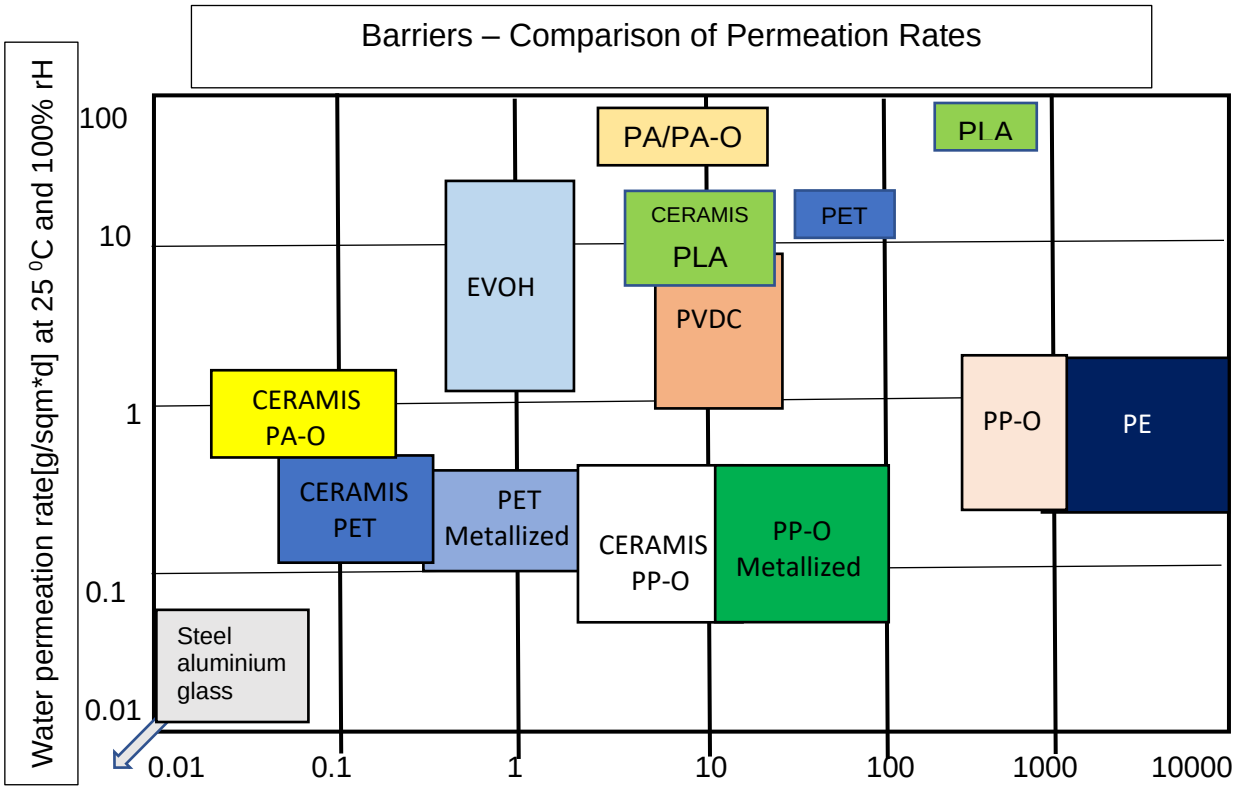


Figure 2.11: Water and oxygen permeation (Rosato, 2011)

2.12.1 Light barrier structures in protecting food packaging

Radiation energy possesses photo-oxidation effects which may cause polymers to undergo oxidative degradation, thus light barrier properties are critical in food composite structures (Coltro & Borghetti, 2017). There are three methods of protecting food and stabilising radiation found in the flexible packaging industry, namely: *free-radical scavengers*, *UV absorbers*, and *excited state quenchers*. These are introduced during processing. However, the nature of each polymer plays a big role in terms of the type of method to be used for each. These attributes are significant in photo-degradation. With some food being very sensitive to light and producing bacteria and unfavourable side reactions when they are exposed to light, light barrier polymeric structures become crucial to prevent losses and improve the safety of the package. Most of the HDPE polymers contain materials of construction that are made from semi-crystalline, however the material still provides high mold shrinkage during processing.

2.12.2 Significance of reducing water vapour permeation

Moisture and water vapour are some of the parameters that have a negative effect on the mechanical and physical properties of the product. Therefore, it is imperative that transmission of water through a packaging material is reduced by addition of a moisture barrier polymer that has low water vapour permeation.

According to Figure 2.12 below, various polymers were subjected to diverse environmental conditions in order to understand how permeability will occur under different parameters.

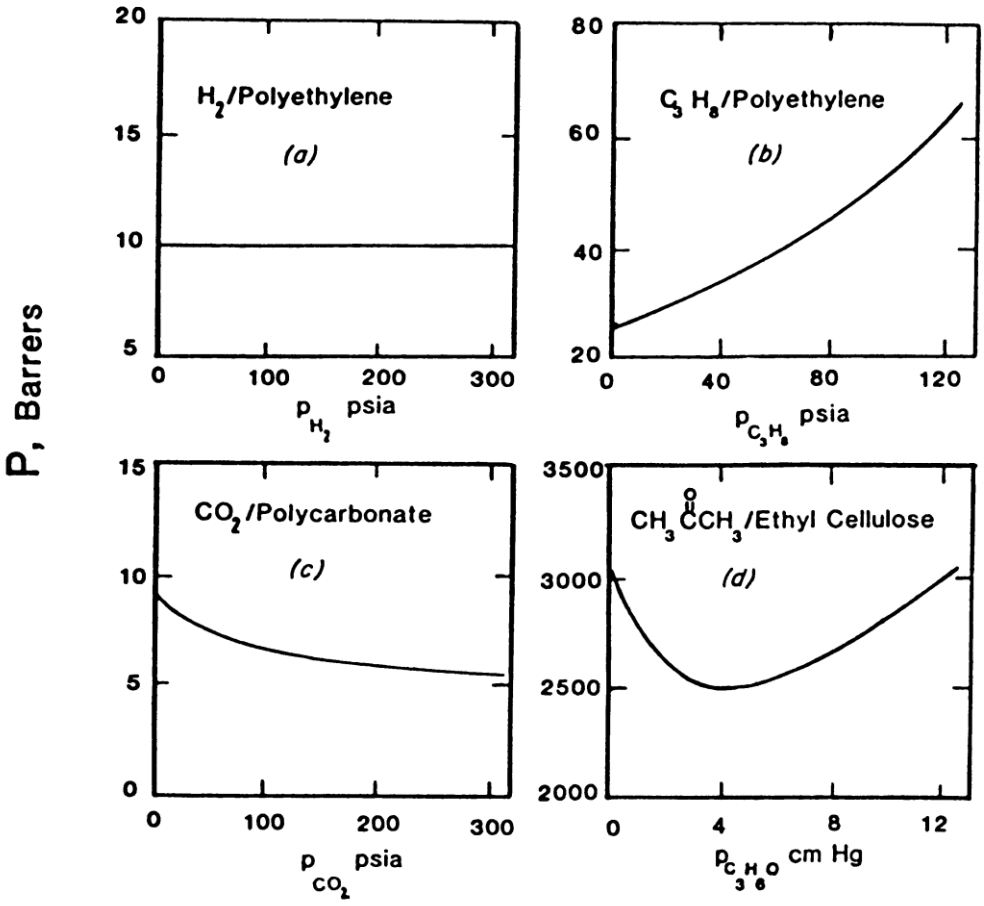


Figure 12.12: Polymer behaviour under different pressure conditions (Koros, 1990)

According to Koros (1990), the composition of different polymer structures can have a greater effect on the ability of the material to allow gases to pass through the membrane layer of polymers. In Figure 2.12, different polymers are subjected to diverse atmospheric conditions to measure gas permeation. The first graph in the figure above demonstrates a PE material subjected under hydrogen atmospheric conditions. Under the hydrogen atmosphere, there is low permeation in the PE material. The second graph demonstrates a variation in the gas permeation when PE is subjected to C_3H_8. The polycarbonate in the third graph is subjected under carbon dioxide and depicts that permeation rate decrease with the decrease in the amount of pressure subjected. The fourth graph demonstrates inconsistent permeation in ethyl cellulose.

2.13 Effective Barrier Polymers and Structures

The discovery of EVOH has played a critical role in meeting the demand of oxygen barrier properties (Feng et al., 2018). This polymer has been utilised in many applications, either as a copolymer or single layer for wrapping. While these polymers have demonstrated great barrier strength at dry state condition, the results have been poor when subjected to high

relative humidity. Because of its crystalline nature, EVOH polymers bond well and form a very strong hydrogen bonding with other polymers. EVOH also offers a crystal clear and glossy surface structure. Since the 1970s when EVOH was discovered, numerous studies have focused on the value of EVOH barrier properties. Because of its semi-crystal structure and alcohol monomer, EVOH has expanded its application as a barrier of gases such as carbon dioxide (CO_2) and nitrogen (N_2) (Maes et al., 2017). Due to the hydrophilic nature of EVOH materials, the polymer plasticised when used as a single component. As a result, inter and intermolecular becomes weak, thus the polymer can only be used as a co-polymer in multilayer structures.

Again, Figure 2.13 demonstrates that EVOH has been utilised as the co-polymer in many applications. In certain cases, EVOH is blended with PA to improve processing characteristics and the performance of the laminate. However, because of their physical and mechanical properties, blending these different resins can be a challenge. The EVOH melting point ranges between 162 ^{o}C – 178 ^{o}C, while the PA melting point ranges between 190 – 350 ^{o}C.

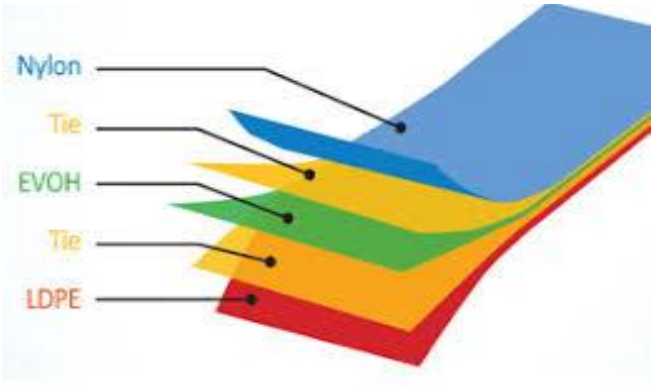


Figure 2.13: Co-layer EVOH simple multilayer construction

2.14 Polyethylene Used as Barrier Material

LDPE is an excellent barrier material for water vapour permeation; however, it has been reported to be a poor barrier material against oxygen, nitrogen, and carbon dioxide (Jordan et al., 2016; Shebani et al., 2018). LDPE has been used in many industrial applications because it possesses hot tack which resists forces that pull the seal strength apart during application. LDPE is more transparent than the translucent LLDPE material. However, when there is a demand for material that has high elongation, impact strength, and puncture

resistance, LLDPE is the best material available at low cost. Similar to LDPE, LLDPE has good barrier properties for water vapour permeation (WVP), but demonstrates weak barrier properties against oxygen, nitrogen, and carbon dioxide (Jagannath et al., 2005). Although HDPE is produced at the same pressure and temperature as LLDPE and LDPE, HDPE is much stiffer than both PE materials. HDPE also possesses good barrier properties against WVP and gases, but is a poor barrier against oxygen (Hamad et al., 2012). Another general polymer that is suitable for WVP is PP material. The PP material is more transparent and harder than other PE material but possesses similar characteristics that the HDPE has.

There are two categories of polyesters(PET), namely: polyethylene naphthalate (PEN) and polyethylene terephthalate (PETE). These are produced by ester monomers of alcohol and carboxylic acid. Polyesters provide excellent barrier properties for moisture permeation, carbon dioxide and oxygen. To improve the seal properties of the material, coating application is utilised. Table 2.7 summarises the list of references used in Chapter 1 and Chapter 2.

Table 2.7: Lamination technologies and properties of different plastics

Reference	Focus of the study	Comment	Topic
(Petrie, 2011)	SF, SB and EL with advantages of each technology.	Majored on properties of each adhesive technology	Laminating adhesive for flexible packaging
(Zarybnicka et al., 2015)	Hardener and resin curing properties – adhesive lamination	Epoxy adhesive lamination tested.	Synthesis of curing agent for epoxy resin based on halogenophosphazene
(Toenniessen, 2018)	SF and SB adhesive lamination - PU/isocyanate based.	Significance of adhesion and cohesive forces.	Packaging materials 10. Adhesive for food packaging application
(Wolf, 2010)	SF, SB and EL with advantages of each technology.	Majored on advantages of each adhesive technology.	A technology decision – adhesive lamination or Extrusion lamination
(Al-Attar et al., 2018)	Focuses on different PE blend and material properties.	Explain the rheology PE grades and elongation.	Thermal, mechanical, and rheological properties of low density/linear low density polyethene.
(Sadiku-Agboola et al., 2010)	Morphology, strain and stress of different polymer structures.	Studied the miscible and immiscible polymer blends.	Rheological properties of polymers: structure and morphology of molten polymer blends
(Khanam & AlMaadeed, 2015)	PE blends and different polymer multi-composites.		Processing and characterization of polyethylene-based composites
(Gaymans et al., 1977)	Polymerisation of PA films.	Studied the preparation of PA webs and their properties	Preparation and some properties of nylon 46
(Ali & Kaneko, 2017)	Studied the polymerisation of bio-based structures and their attributes.	Majored different approach of PA polymerisation	Polyamide syntheses

(Samanta et al., 2012)	Explained thermal properties and crystal nature of PA webs.		Polyamides based on the renewable monomer, 1,13-tridecane diamine II: Synthesis and characterization of nylon 13,6
(Jo et al., 1993)	Morphology, rheology of PA film and copolymers.	Advantages of controlling the morphology of PA film and its properties.	Morphological, rheological, and mechanical properties of polyamide 6/styrene-acrylic acid copolymer blends
(Walha et al., 2016)	Physical and mechanical properties of PA film under setting condition.	Detailed the PA compatible blends and morphology.	Rheological, morphological and mechanical studies of sustainably sourced polymer blends based on poly (lactic acid) and polyamide 11
(Yeh & Fan-chiang, 1997)	Studied the impact and barrier properties of PA, CP and PE films.	Test the efficiency of different films and their crystallinity.	The barrier, impact, morphology, and rheological properties of modified polyamides and their corresponding polyethylene–modified polyamide blends
(Coltro & Borghetti, 2017)	Studied different films which provide barrier properties.		Plastic packages for personal care products – evaluation of light barrier properties

2.15 Summary of Chapter

Chapter 2 detailed the different DOE approaches that are available to conduct modelling and optimisation of the production process. These DOE techniques include D-optimal, full factorial, response surface, and TM OA. Based on the properties and capabilities of each DOE technique, the TM based OA was found to be suitable for this study since it requires less experiments in reducing process variation. The TM based DOE utilises fewer resources to maximise the response output compared to other types of DOE. Apart from evaluating the QLF of any system, the TM also quantifies the contribution of each process variable on the output response using the ANOVA. The TM approach in studying variation also characterises quality using the S/N ratio. Since the study focuses on SF lamination of PA and PE film, different lamination techniques were covered to highlight the significance of SF adhesives. Furthermore, the use of barrier and sealant film, such as PA and PE, were also described. Moreover, the use of different grades of PE film with their advantages and disadvantages were explained.

The focus of the next chapter is on experimental methods, optimisation, and lamination design.

CHAPTER 3

EXPERIMENTAL METHODS, OPTIMISATION AND LAMINATION DESIGN

Chapter 3: Experimental Methods, Optimisation and Lamination Design

3.1 Introduction

The previous chapter presented the literature review of the study. Building on these insights, the current chapter focuses on experimental methods, optimisation, and lamination design. Various DOE methods have been utilised in many industries to model and optimise manufacturing systems (Bowden et al., 2019). The regression models developed by different DOE mechanisms have contributed tremendously to predicting the behaviour of existing systems at different parameter levels (Sahoo, 2013; Nguyen et al., 2015; Karna et al., 2012; Asghar et al., 2014). This chapter therefore focuses on process parameters involved in a SF laminating system. Furthermore, this chapter also focuses on experimental methods and discusses the importance of minimising process variation on each design parameter within a SF laminating system. Chemicals and materials used during experimentation are also shared herein. The importance of each design parameter during SF lamination of flexible packaging materials, including the surface modification technique of PE and PA films are further detailed in this chapter. In addition, a preparation of dyne solution using a mixture of formamide (CH_3NO) and ethyl cellosolve ($C_4H_{10}O_2$) at different concentration levels for testing surface energy on films is discussed. Lastly, modelling and optimisation using TM through Minitab software is highlighted. The chapter concludes with a brief summary.

3.2 Materials

3.2.1 Polyethylene (PE) and Polyamide (PA)

The PE material utilised during the experiment was extruded internally and treated at a minimum surface energy of 42 dyne/cm. The PA material was imported from an overseas supplier Kolon.

3.2.2 Lamination technology selection

As discussed in Chapter 2, various lamination technologies have been utilised for multi-composite polymer structures within the packaging sector; however, environmental concerns associated with solvent retention on the SB adhesives and disadvantages of WB adhesive have prompted additional research to be conducted on the isocyanate-based group which is SF adhesives, as it possesses no solvent retained after lamination (Ling et al., 2010). Thus, it is utilised in this study.

3.2.3 Chemicals

Some of the chemicals used during this study include ethyl acetate (EA), formamide, isocyanate, and an alcohol group. The EA solvent was utilised in this study to perform the cleaning of the adhesive system and machine rollers to achieve consistent distribution of the CW while maintaining the porosity of the application roller. Based on the information provided in Chapter 2, EA is more environmentally friendly than other solvents such as dimethylformamide (DMF), methyl ethyl ketone (MEK), and toluene, which can be used to perform the same task. EA is less volatile than the above solvents with a boiling point of 77 ^{o}C.

Table 3.1: List of chemicals used with their properties

Chemicals	Chemical Formula	Chemical and Physical Properties
Ethyl Acetate	$C_4H_8O_2$	BP:77.1 ^{o}C; Density: 902 kg/m^3. Colourless liquid with sweet smell.
Formamide	CH_3NO	BP:210 ^{o}C; Density: 1.13 g/cm^3. Clear liquid with Ammonia like odour.
Resin: Isocyanate group	R−N=C=O	BP:190 ^{o}C; Density: 1.16 g/m^3. Transparent yellow.
Ethyl cellosolve	$C_4H_{10}O_2$	BP:135 ^{o}C; Density: 930 kg/m^3. Colourless liquid with mild odour.
Hardener	R-OH	BP:190 ^{o}C; Density: 0.993 g/m^3. transparent yellow.

3.3 Experimental Methods

The experimental method starts with the pre-treatment of PE and PA materials, and then the preparation of the dyne solution.

3.3.1 Pre-treatment of PE and PA

The PE materials were produced at the extrusion machine internally using a 50:50 percent ratio of LLDPE and LDPE resin from Sasol. The PE material was pre-treated before lamination at a minimum surface energy of 40 dyne/cm. The PA material was received from Kolon (outside supplier) with a treatment of 48 dyne/cm. Both materials were stored at a minimum temperature of 20 ^{o}C before lamination. Moreover, both materials were wrapped

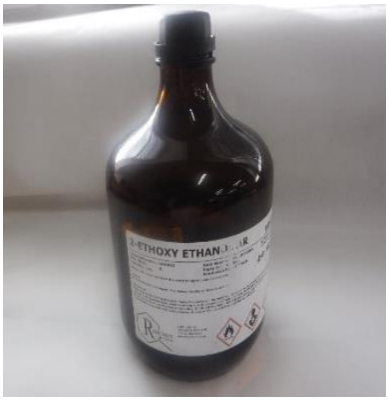
with a film to prevent contamination from the environment. The PA material had a silica gel inside while stored on wooden boxes to prevent moisture absorption. The PE and PA materials were kept at the store for less than a week to prevent corona treatment decay and excessive migration. Surface energy on each web was measured using a prepared dyne solution.

3.3.2 Dyne solution preparation

The corona dosage on PE and PA film before lamination was measured with a concentration of ethyl cellosolve mixed with formamide to yield 42 dynes/cm of wetting. To control wetting tension, the ethyl cellosolve concentration was kept at 28.5% with a ratio of 71.5% of formamide. To yield 38 dynes/cm solution, formamide and ethyl cellosolve were mixed at a ratio of 46% and 54%. The mixture turns to a purple colour after the mixing process is complete. The mixture is then applied on the film to identify the level of surface energy through wetting tension. Both ethyl cellosolve and formamide can still be mixed at a different ratio to measure the surface tension at different levels.

Formamide (CH_3NO)

+



Ethyl Cellosolve (Ethyl cello-solve)

Figure 3.1 Corona treatment testing solution

$CH_3NO + C_4H_{10}O_2 \rightarrow C_5H_{11}NO_2 + H_2O$ (1:1 stoichiometric ratio) (3.1)

Non-polar films such as PE and PA need their surface to be modified in order for ink and adhesive to be able to adhere as the result of improved surface energy (Awaja et al., 2009). These polymers are called “inert polymers”.

Figure 3.2 shows a photograph of the dyne solution prepared using the chemicals:

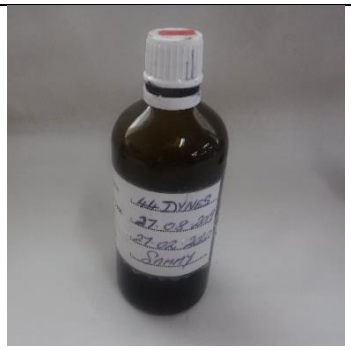
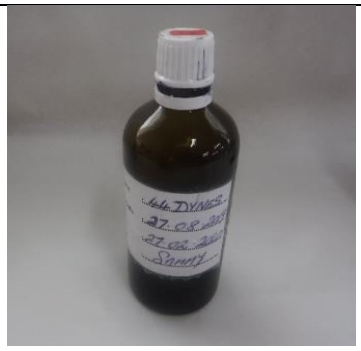
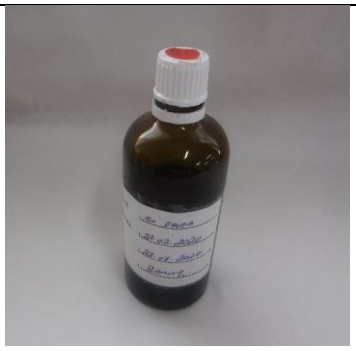
		
40 dyne. Exp date: 19/11/2020	44 dyne. Exp Date :19/11/2020	46 dyne. Exp Date: 19/11/2020

Figure 3.2: Dyne solution at different concentration levels

Because of the chemical properties of formamide, the prepared solution turns into a purple colour when mixed with ethyl cellosolve at different concentration levels. The dyne solution is used to test the level of surface energy at different corona treatment levels. After testing treatment level on the PE and validating the level of corona dosage, the material can be loaded on the SF lamination equipment to produce laminate.

3.4 Mix Ratio

In this study, adhesive (PU) and hardener (curing agent) are mixed together at a 80/20 ratio to yield the required bond strength between PE and PA web substrate. The port-life of the two-component adhesive as determined by the supplier is 30 minutes. For batch production environment, the working life of the adhesive needs to be monitored since anything above the specification may yield an undesired reaction. The mixture is therefore exposed to heat treatment to facilitate the bonding rate. MR measurements are crucial when two-component adhesives are utilised. When the adhesive ratio is not controlled, variation occurs. When the ratio of hardener increases during processing, degradation of adhesive material properties is exerted, thus a MR measurement is key in reducing process variation and poor AS.

3.5 Corona Treatment Measurement

There are three available methods for measuring the amount of corona treatment/surface energy on web substrates. These techniques are listed below:

3.5.1 Cotton-swab method

In this study, the *cotton-swab* method was utilised for measuring the surface energy of the PE and PA material. Three dyne solutions were prepared to conduct these measurements: 40 dyne/cm, 44 dyne/cm, and 46 dyne/cm. During the experimentation, the swab was immersed into a dyne solution before wetting the web. This was done to ensure that there is enough solution on the cotton. The rule on this, is that the solution must adhere on the web substrate for a period of more than 2 seconds. When the first test is conducted, a new clean cotton-swab should be used for a second test to measure the surface energy for the second test. Ideally, the solid line demonstrated by the dyne solution after wetting should stay intact with the surface of the web if the surface energy of the substrate is higher than the surface energy of the material. The application of the swab method is repeated until the dyne solution line on the web breaks. This signifies that the amount of surface energy on the material is then lower than the amount of energy on the dyne solution.

3.5.2 Dyne-pen method

During this method, a *dyne-pen* is used to validate the amount of surface energy on the material. The pen consists of formamide and ethyl cellosolve mixed at different concentrations. However, this method is considered to lack accuracy when compared to the cotton-swab method. This is because of the contamination that occurs on the tip of the pen when it has been used multiple times. This method is considered to be quick in comparison to other methods such as drawdown. During application, as the pen is pulled across the web, a solid line is demonstrated on the web as a result of the pen.

3.5.3 Drawdown test method

The *drawdown* method is considered to be the most accurate method within the flexible packaging industry. During testing, a sample of substrate is placed on a flat surface so that it cannot move. Thereafter, drops of the dyne solution at different concentration levels are placed on top of the sample to evaluate the amount of surface energy on the film. During the testing, a metering rod is used to spread the solution at the same time. Based on the requirement of the drawdown, the film tested should be free from contamination.

Table 3.2: Method for quantifying treatment dosage

Ethyl cellosolve (%)	Formamide volume (%)	Wetting tension: (Dynes/cm)
54	46	38
59	41	39
63.5	36.5	40
67.5	32.5	41
71.5	28.5	42
74.7	25.3	43
78.0	22.0	44
19.7	80.3	45
17.0	83.0	46
13.0	87.0	48
9.3	90.7	50
6.3	93.7	52
3.5	96.5	54
1.0	99.0	56

3.6 Effect of Corona Treatment Method on Polymers

Corona treatment is an established method for chemical modification of PE surface by means of a highly reactive ozone gas which breaks the hydrogen and carbon (H–C) bond to polarise the surface of the film during extrusion and lamination (Lindner et al., 2017). Lower distribution of corona discharge affects the AS and results in chemical inert of PE films (Awaja et al., 2009). High distribution of corona discharge results in a highly polarised material and thus increases the surface energy of the PE film. A 42 dyne/cm of corona discharge is the required minimal corona dosage, and 50 dyne/cm is the maximum treatment of PE. A corona dosage above the requirement will result in the production of a material with poor bonds, which will result in a poor quality of the product being produced. Corona dosage above the standard has a negative effect on the polymer surface as it results in molecular scission of polymers. For this study, the corona treatment of PA film was kept constant at 50 dyne/cm. The two figures (Figures 3.3 and 3.4) below demonstrate the effect of corona treatment on different substrate.

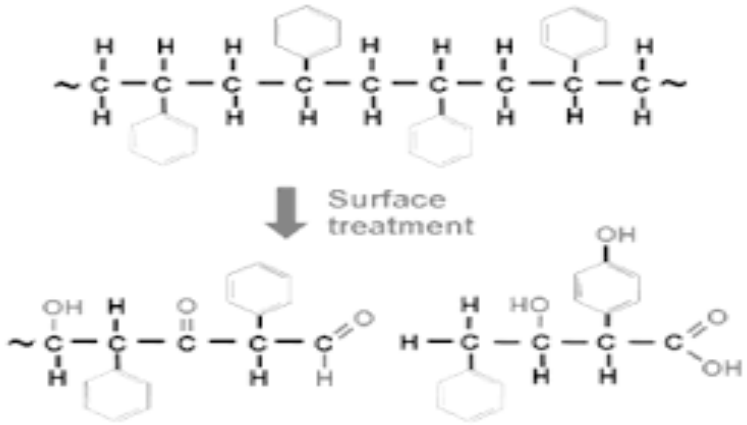


Figure 3.3: Effect of corona on PP film

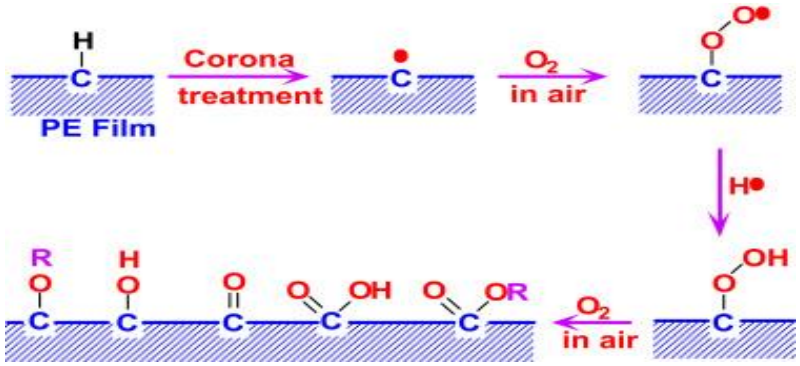


Figure 3.4: PE corona treatment process

According to Lindner et al., (2017), the magnitude and the contribution of the corona treatment on the bond strength depends more on the type of adhesive (SL/SB), film type, storage time before and after treatment, and corona dosage. As demonstrated in the figures above, the attack of corona treatment on PP is different when compared to the PE film. This is because of the molecular distribution that each web substrate possesses. The above figures further demonstrate how the oxygen polar group is introduced on top of the surface material during the corona treatment process. Through this process, the surface energy of the material is enhanced.

3.7 Application Temperature (AV) and Curing Temperature (CT)

In this research project, AV is studied at three levels, namely: the lower level at 35 ^{o}C, the intermediate level at 45 ^{o}C, and the highest level at 55 ^{o}C. In SF lamination, the heating adhesive system controls the viscosity of the adhesive and ensures that during application, adhesive is applied uniformly on the web substrates. Therefore, when considering utilising

SF adhesive, key parameters, which include the temperature before and after processing, become crucial to achieve higher AS through strengthening intermolecular forces between the web substrates. The intermolecular forces between adhesive and web substrates are said to be related to van der Waals forces, dipole-dipole, and chemical interactions. Thus, understanding the properties of adhesives plays a big role.

With excellent properties of holt melt adhesive, PU adhesive has been utilised in many applications within the packaging industry. PU adhesive is a thermoset polymer. These are irreversible types of adhesives and therefore cannot be recycled. The type of PU adhesive utilised in this study is an isocyanate moisture cure-based adhesive which is softer when compared to epoxies. However excessive moisture affects the curing rate during processing (Awaja et al., 2009). Therefore, it is very critical that reaction is driven in a moisture-controlled environment. Secondly, the AV needs to be controlled between 35 ^{o}C and 55 ^{o}C, since this type of adhesive is considered a non-Newtonian fluid. This is because PU viscosity changes with time, especially when heated. CT is the actual temperature at which laminated material is kept for the SF reaction to be facilitated to bond the PE and PA material. This is another significant parameter since any temperature below 15 ^{o}C will result in delamination because of uncured material and a side reaction that may occur, as demonstrated in Chapter 2 equation 2.5.

3.8 Machine Speed (MS)

According to Ling et al., (2010), the laminating unit consists of different rollers with various dimensions working at different principles. These rollers are running at a certain ratio in order to provide the required output in metres/minutes. In this case, there are three levels that were considered for MS: the lower level which is at 150 m/min; the intermediate level that is kept at 200 m/min; and the high level that is demonstrated when MS is kept at 250 m/min.

3.9 Viscosity (μ) Measurement

Viscosity measurements are monitored to reduce variation and improve the quality of the manufactured product. Viscosity depicts the functionality of substances, which includes surface tension and setting rate. Agitation and temperatures have negative effects on the adhesive viscosity and its fluidity at molten state. Adhesive systems belong to non-Newtonian fluids since their chemical behaviour changes with the flow of matter. When adhesive systems are heated, molecules are rearranged, which causes it to gradually deform to the form of stress. There is a great relationship between thickness of the liquid and its viscosity.

Through a static mixer, the laminant is fed at a volumetric flow rate of 5 mil/m^3 to the metering for bonding.

3.10 Rewind Tension (RT) and Taper Tension (TT)

During the experiment, the RT was kept between 60 and 80 Newtown to control the elastic properties of the substrate since a multi-composite polymer is used for SF lamination. This is because the green strength of SF lamination is very low when compared to SB lamination; therefore, a good setting of RT is required during lamination. Due to a very low cleavage strength of the adhesive, which later affects cohesion between molecules and adhesion polymer to the adhesive layer, the TT becomes another critical parameter during processing. During the experiment, the effect of TT during the SF lamination is studied at three levels, namely: 15%, 20%, and 25% taper.

3.11 Coating Weight (CW)

The CW describes the amount of adhesive distributed by the application roller to the web substrate. The CW in this project is evaluated under three different levels. These include the lower levels at 1.5 grams per square metre; the second level at 2 grams per square metre; and the highest level, which is kept at 2.5 grams per square metre.

3.12 Testing Methods

After setting all the process parameters above according to each run and level indicated by L_{18} OA, AS measurement was performed after 48 hours of curing. A 48-hour curing period was allowed to ensure that reaction between the hardener and the resin was fully completed with no side reaction.

To measure the bond strength between PA and PE, a sample of 10 x 10 mm was taken on each reel after 48 hours. Furthermore, the sample was placed on the Instron gripper as indicated in section 3.9.1. The PE side was placed on the lower gripper fixture, while the PA side was place on the upper gripper fixture to quantify both AS and tensile speed (TS).

Figure 3.5: Instron tensile tester

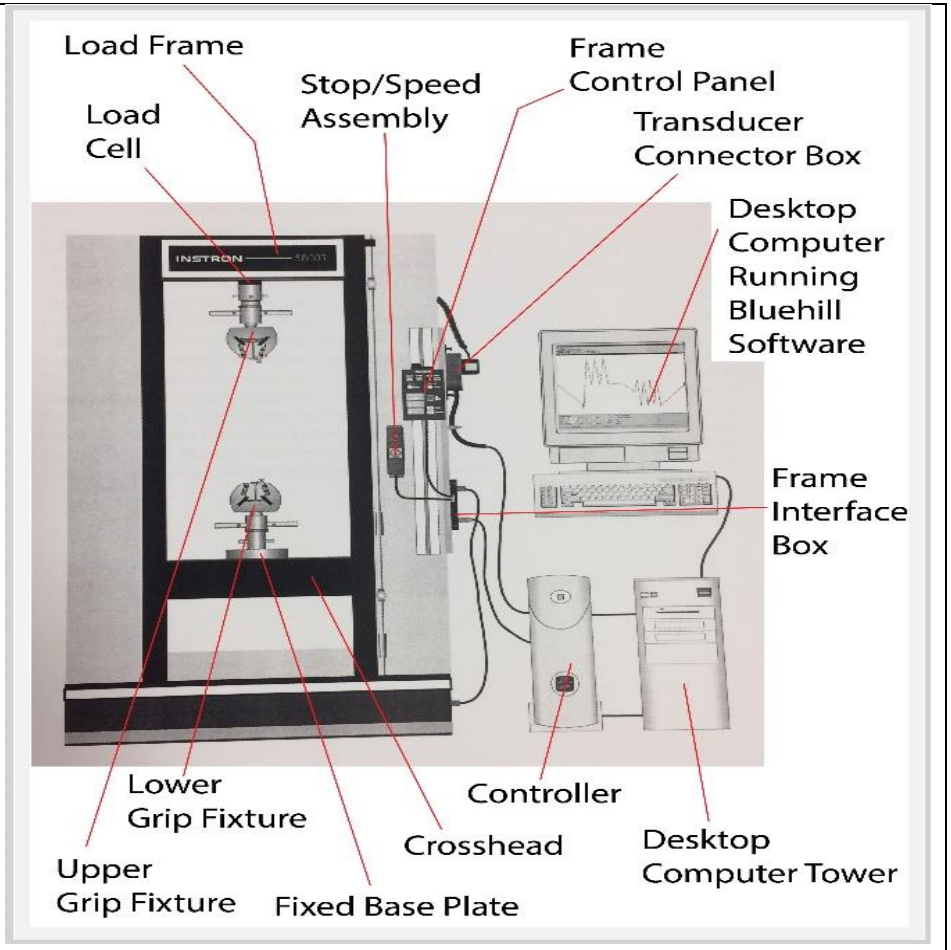
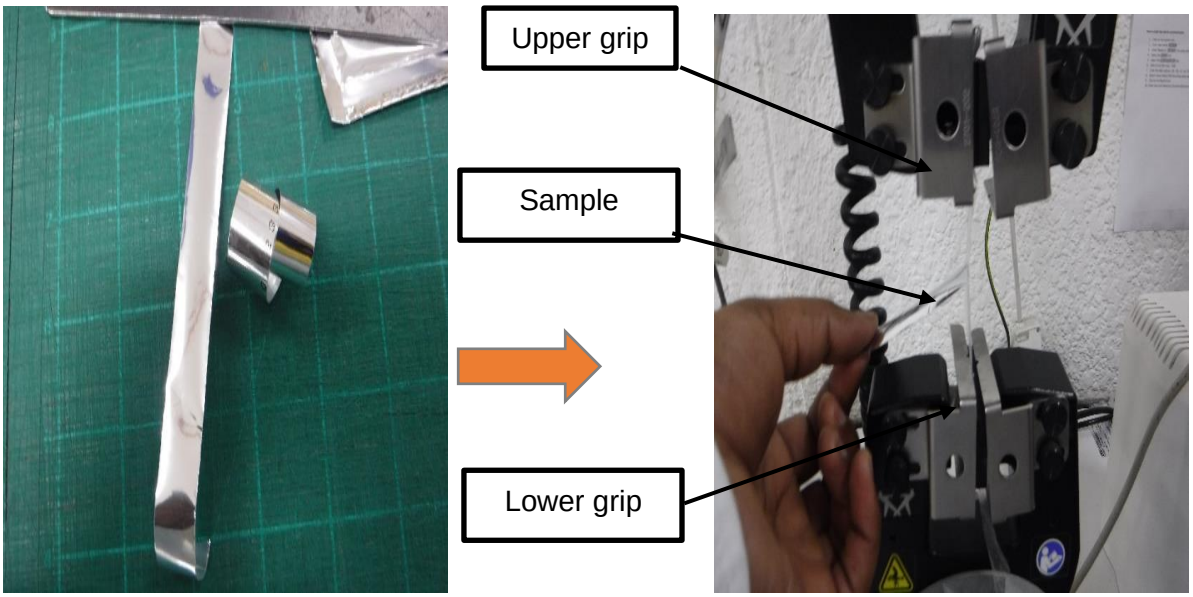


Figure 3.6: Instron tensile tester drawing



Sample from sample Sample placed on the Instron Grippers

Figure 3.7 laminate and Instron equipment used for determining AS

3.13 Analysis of Variance (ANOVA)

In this study, ANOVA was used to study the effect and contribution of each input variable to the response variable. A total of 8 process parameters were used to optimise the SF lamination using PA and PE as the model system. The Minitab software was employed to generate the ANOVA and polynomial equation.

With ANOVA, total sum of the squares (SST) is best described by the following equation:

$$SST = \sum_{i-1}^{n} \sum_{j-1}^{r} Y^2{}_{ij} - nr\overline{Y^2} \quad (3.2)$$

Where:

n is the number of the experiment,

R is the experiment data, and

$\bar{Y}$ is the grand mean

Sum of the squares (SS) for each factor is demonstrated by the equation below:

$$SSm = \frac{nr}{L} \sum_{k=1}^{L} (\bar{Y}_k - \bar{Y})^2 \quad (3.3)$$

Where k is the parameter level, and

Y_k is the bond strength response values.

$$\text{Total degree of freedom (DOF)} = \text{Total number of experiments} - 1 \quad (3.4)$$

$$\text{DOF per parameter} = \text{Number of level (L)} - 1 \quad (3.5)$$

$$\text{Variance} = \frac{SS}{DOF} \quad (3.6)$$

$$\text{F-ratio} = \frac{\text{Var}}{\text{Var of error(Ve)}} \quad (3.7)$$

$$\text{Pure sum of squares} = SS - Ve * DOF \quad (3.8)$$

$$\text{Percentage contribution} = \frac{\text{Pure SS}}{\text{ToTal SS}} \quad (3.9)$$

The standard OAs with their number of factors and the number of required experiments are listed in Table 3.3 below.

Table 3.3: Standard Orthogonal Array

Orthogonal Array	No. of Experiment	Max.# of Factors	Max.# of Factors at these Levels			
			Level 2	Level 3	Level 4	Level 5
L_4	4	3	3	-	-	-
L_8	8	7	7	-	-	-
L_9	9	4	-	4	-	-
L_{12}	12	11	11	-	-	-
L_{16}	16	15	15	-	-	-
L'_{16}	16	5	-	-	5	-
L_{18}	18	8	1	7	-	-
L_{25}	25	6	-	-	-	6
L_{27}	27	13	1	13	-	-
L_{32}	32	31	31	-	-	-
L'_{32}	32	10	1	-	9	-
L_{36}	36	23	11	12	-	-
L'_{36}	36	16	3	13	-	-
L_{50}	50	12	1	-	-	11
L_{54}	54	26	1	25	-	-
L_{64}	64	63	63	-	-	-
L'_{64}	64	21	-	-	21	-
L_{81}	81	40	-	40	-	-

During the experiment, all the output variables that needed to be optimised were selected, along with the inputs affecting the output response. The level of each parameter and OA were identified. All interaction and factors were assigned, and the experiment performed. Statistical analysis was used to determine the optimum condition. Factors and levels were also utilised during the experiment, as demonstrated in Table 3.3 under L_{18}.

3.14 Summary of Chapter

This chapter presented the experimental methods, optimisation, and lamination design parameters for bonding PE and PA materials. In addition, materials and chemicals used during experimentation were detailed in this chapter. The effect of corona dosage on PE and PP films were discussed. A mixture of formamide and ethyl cellosolve was prepared at different concentration levels for surface energy verification. Different methods used to

validate treatment levels on polymers were also demonstrated. A cotton-swab method was used during the experiment to validate the surface energy of each web substrate. The effect of AV and CT on isocyanate hot melt adhesive was fully described, along with the impact of a moisture-controlled environment on curing.

The following chapter presents and discusses the results of the experiment.

CHAPTER 4

RESULTS AND DISCUSSION

Chapter 4: Results and Discussion

4.1 Introduction

This chapter focuses on the processes carried out to perform experimentation which incorporates sampling, experimental setup, and the analyses of results to quantify the contribution of each design parameter on the AS between PE and PA. During the experiment, a TM based DOE was utilised to model and optimise a SF laminating unit to improve film mechanical properties and yield. Optimisation of SF lamination ensures that production waste and process inefficiencies are reduced while maximising profitability. Prior to running the experiment, preliminary tests were conducted using a Gage repeatability and reproducibility (R&R). Further details are provided in the subsequent sections below.

4.2 Preliminary Tests

This section focuses on the preliminary test performed. Preliminary tests were conducted to evaluate the Gage R&R in order to assess for any variation that may be due to the measuring instrument and also the personnel performing the experiment. This is critical as the fault of the measurement system may alter the result and provide a false analysis. To yield the required result on Gage R&R, a series of tests were performed by three different personnel during the measurement of CW which relates to the amount of adhesive distributed between the PE and PA web substrate during lamination. The CW parameter needs to be controlled through the metering roller gap during lamination. To control the amount of adhesive distributed, a 1 mm filler gage was utilised to set the gap between the metering rollers.

4.3 Gage Repeatability & Reproducibility Data Arrangement

Table A.1 (Appendix A) demonstrates 45 measurements conducted to qualify the Gage R&R study performed by operators A, B and C. The population parts were grouped accordingly and referred to as Parts 1, 2 and 3. In this study, the CW was measured in grams per metre (gsm). The lower limit for the CW is 1.5 gsm, while the upper limit is kept at 2.5 gsm. All CW measurements and ratios were conducted at the start-up of every batch. Although the data presented in Figure 4.1 and Table A.1 (Appendix A) are in uniform manner, the measurements were performed in random order. The Gage R&R performed in Figure 4.1 below was necessary to evaluate the performance of the measurement system while verifying its effectiveness for reducing process variation. Figure 4.1 describes the result of the Gage R&R that was performed using the data in Table A.1 (Appendix A). The data point

reveals a process that possesses instrument variation which needs to be reduced in order to ensure uniformity of CW distribution. Based on the analysis of the component of analysis graph, a high repeatability and Gage R&R is shown by the graph, and these contributed to the variation of the system. The R chart also depicts that operator B's measurement is conducted at a lower level when compared to operators A and C.

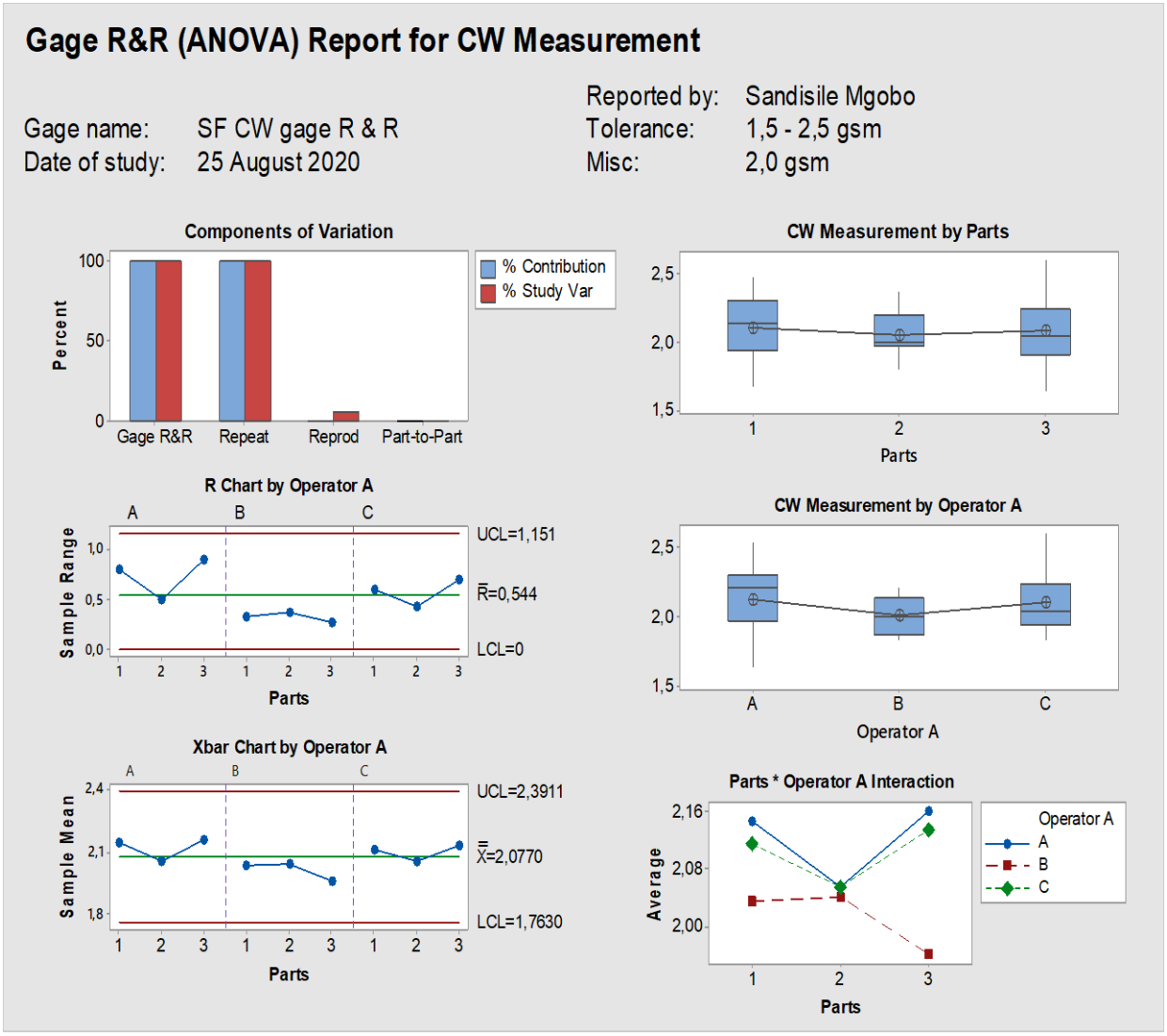


Figure 4.1: Gage R&R Analysis: Before Experiment Gage R&R Study – ANOVA Method

Figure 4.1 above further depicts limited variation, which is due to the material utilised. Table 4.1 was plotted in order to identify the significance of the interaction between the part, device, and the operator. Since process variation can also be due to interaction of process variables, the SS, F-test, and the probability value are demonstrated to identify the most significant variables.

Based on Table 4.1, the part to operator interaction is not significant since the P-value is 0.91. For a variable to be statistically significant, the P-value has to be equal to or less than 0.05.

Table 4.1: Two-way ANOVA table with interaction

Source	DF	SS	MS	F	P
Parts	2	0.01916	0.74187	0.532	
Operator A	2	0.10079	0.05o395	0.24711	0.91
Parts * Operator A	4	0.05165	0.012914	0.24711	0.91
Repeatability	36	1.88133	0.052259		
Total	44	2.05294			

α to remove interaction term = 0.05

After studying the statistical significance of the different sources in Table 4.1, a two-way analysis of variance, as shown in Table A.2 (Appendix A), was plotted to understand the influence of interaction on both the R&R. The Table A.3 (Appendix A) two-way ANOVA was calculated to understand the behaviour of the system when the interactions between the variables are limited and ignored. Furthermore, Table A.3 (Appendix A) shows a drop in the probability values with 0.362 when compared to 0.91 achieved in Table 4.1 for operator A. In addition, there is also a significant drop in the repeatability MS values from 0.0522 to 0.048 when interactions are not considered since the P-value portrays insignificance of variables.

In further understanding the two-way ANOVA without interaction, Table 4.2 represents the actual sources of variation and percentage contribution for each source. As presented by Figure 4.1, both total Gage R&R had high data points. Therefore, Table 4.2 illustrates that total Gage R&R had 100% contribution to the existing variation on the laminating unit while the repeatability had 99.72% contribution. According to the result, reproducibility was insignificant with a total contribution of 0.28%. Furthermore, Table 4.2 also depicts that the difference in parts did not pose a threat to the result since they had 0% contribution. The operator had 0.28% contribution which indicates insignificance with a var comp of 0.00032.

Table 4.2: Gage R&R parameter contribution variation and sources

Source	Var comp	% contribution
Total Gage R&R	0.0484627	100
Repeatability	0.0483247	99.72
Reproducibility	0.000138	0.28
Operator A	0.000138	0.28
Part-To-Part	0.000000	0
Total variation	0.0484627	100

The source of variation and the percentage contribution to the variation were indicated in Table 4.2 above. Based on this analysis, it was critical to plot Table A.4 (Appendix A) in order to study the variance and standard deviation, especially when the 95% confidence was placed on the studied data. In this case, the standard deviation was studied to understand how well the data points distributed around the mean values, especially when the sources of variation have been identified. Furthermore, the repeatability demonstrated a high standard deviation than any other source of variation with a stdv (SD) of 0.219. This means that the data points are further from the mean average when compared to reproducibility that has a standard deviation of 0.0117. The total Gage R&R had the second highest standard deviation with 0.220. The part-to-part had a standard deviation of 0.000. This means that the values are closer together with high precision and accuracy.

4.4 Sources of Variation and CW Distribution

The Gage R&R study was performed to assess all forms of variation on the process before conducting the actual experiment. In addition, the contribution of part-to-part, equipment and operational interference to the total variation were eliminated to improve the accuracy of the measurement equipment. A CW measurement which involves the distribution of the adhesive on the actual films was used to perform the Gage R&R study.

All Gage R&R measurements are presented in Table 4.1. The first column in Table 4.1 indicates the source of variation, whereas *operator* depicts the reproducibility in this study. The degree of freedom (DF) shows the total DOF for each source of variation. The DF demonstrated in Table 4.1 was based on the amount of data formulated during experimentation. The SS column indicates the SS which depicts the measured variation for each source. The MS stands for mean square which relates to each source of variation. The F and P values quantify the statistical significance of each source of variation. Based on the

results demonstrated in the component of variation graph (Figure 4.1), the existing variation in the system is due to repeatability and total Gage R&R. The high repeatability demonstrated by the result in Table 4.2 indicates a variation that is due to the measuring instrument. These results depict the need for a better measuring instrument. Therefore, a brief summary of the Gage R&R study performed is presented by Figure A.1 (Appendix A) to indicate the position and areas of improvement required to eliminate variation. As demonstrated, there were 3 operators involved in the study with 5 replications. Only 10% of the variation would have been considered insignificant, however there were two sources of variation that were identified by this study, respectively.

After identifying different sources of variation within the Gage R&R, process capability tests were conducted using the six-pack report in order to understand how well the data is distributed along the mean using the normal distribution curve. Figure 4.2 demonstrates that the process had a Cpk value of 0.89 with a standard deviation of 0.15, a Cp of 1.05, and PPM of 4083.06 within the required tolerances. These parameters depict a process operating below six sigma, therefore any experiment that could be performed under these conditions might distort the output variable. Thus, a variation on the measuring instrument needs to be eliminated to improve accuracy and precision in order to improve the performance of the laminating unit.

Capability analysis using coating weight distribution

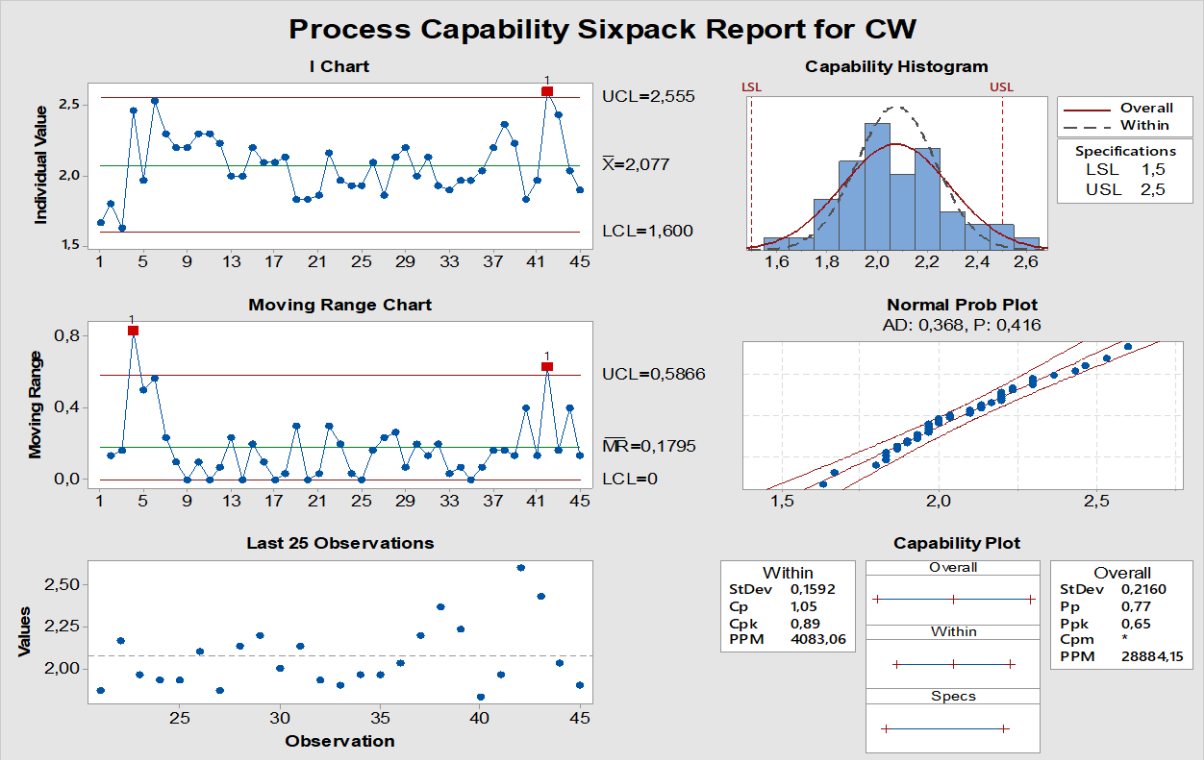


Figure 4.2: Process capability for coating weight distribution

Based on the moving rage plotted in Figure 4.2 above, there were two outliers identified which distort the movement of the data, even though some of the data points are distributed along the mean average. The observation plot shows a greater spread of the values between reel number 35 and 45. The capability histogram presented well distributed data along the mean value, however there are still some data points distributed above the upper specification, thus the variation and a measuring instrument needed to be reviewed. Although most data points fitted the normal plot with a P-value of 0.416, this is not enough to deliver a consistent output response. Therefore, the existing variation was due to the design of the measuring instrument presented in Figure 4.3 below. Therefore, a new design and method was required to improve the accuracy and precision. The feeler gage results presented in Table A.1 (Appendix A) were too poor to produce the required response output.

Figure 4.3: Feeler gauge

The accuracy and precision of operators A, B and C were examined with the feeler gauge in Figure 4.3. The utilised feeler gauge consists of 13 blades made from hardened spring steel as illustrated in Figure 4.3. The feeler gauge has a length of about 100 mm with a width of 12.7 mm. The measuring range is from 0.05 mm to 1.00 mm; however, for the purpose of this study, the 1.00 mm blades were used to measure the gap between the two metering rollers. The result of the Gage R&R is presented in Table 4.2. However, the system demonstrated a poor Gauge R&R study as indicated in Table 4.2. Therefore, the conclusion and recommendation made based on the result was to use the dials gauge demonstrated in Figure 4.4 below to improve accuracy and precision when setting up the gap between the metering rollers.

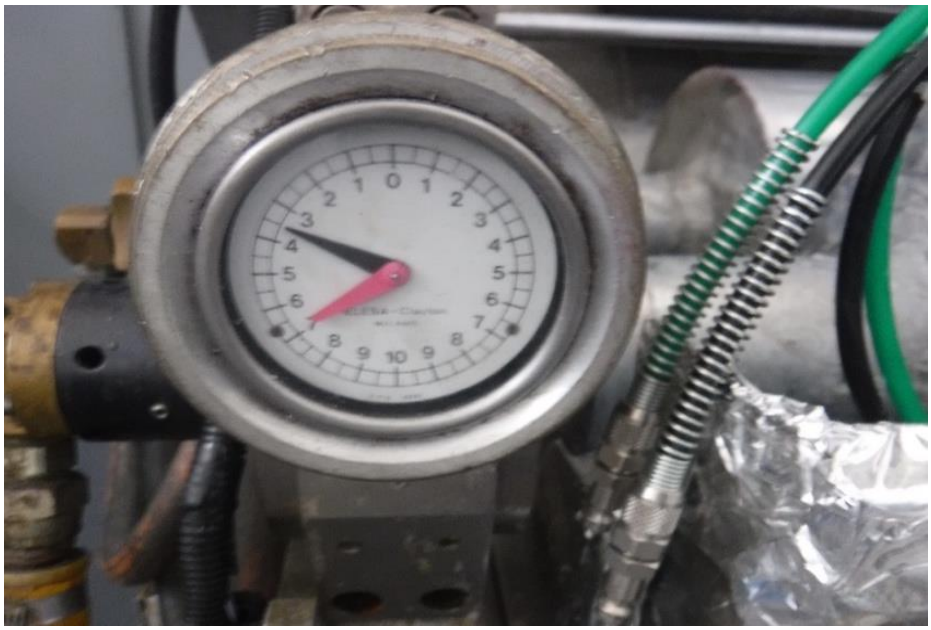


Figure 4.4: Dial gage indicator

The dial gauge indicator was used to improve the consistence of the adhesive distribution which will therefore demonstrate an improved Gauge R&R. The improvement in the Gage R&R will ensure limited process variation. The dial was set between 3 and 4, as shown in Figure 4.4, and interlocked to ensure that there is no variation during processing. The implementation of the dial method reduced human intervention in the adhesive method distribution.

After the feeler gage failed to produce repeatability during the first phase, a more structured method of measuring the gap between the metering rollers was implemented to improve consistent in distribution of adhesive while improving process capability of the SF laminating system. Table 4.6 further shows that the result is more consistent between the replicates. In Table A.2 (Appendix A), an average adhesive weight of 2 gsm can be seen as the instrument demonstrates its capability during the measurement. The two tables (Table A.2 and A.1) (Appendix A) demonstrate the same number of replicate and CW measurement conducted, but the dial showed an improved result in Figure 4.5. The results of Table A.2 (Appendix A) were utilised to plot all Gage R&R graphs for Figure 4.5. The more uniform data points can be seen in Figure 4.5 below, with limited interaction between parts and operators. These results were key in increasing the reliability of the data during experimentation to ensure that SF lamination operates at a high degree of accuracy and precision.

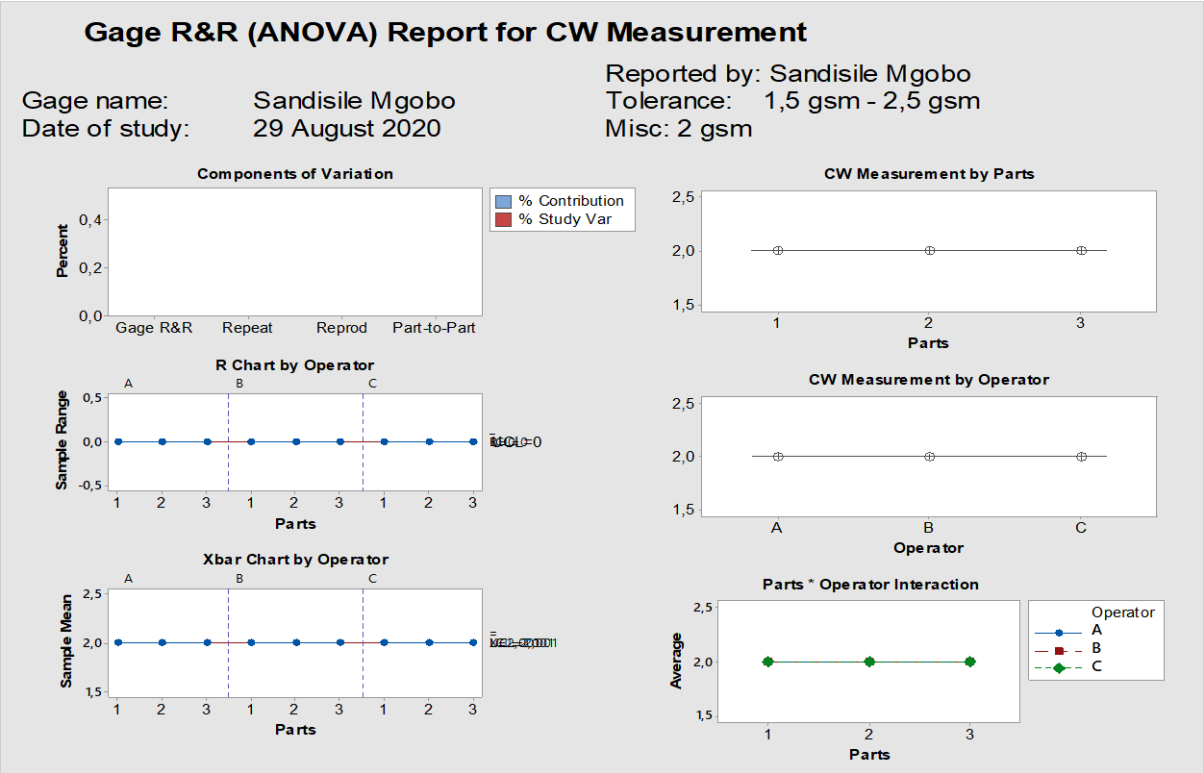


Figure 4.5: Gage R&R for process variation analysis

The Gage R&R ANOVA Table A.3 (Appendix A) was again plotted to study any interaction between the material, operator, and the instrument. Table A.4 (Appendix A) illustrates that there is no interaction between the parts and operator since the P-value is zero. The F-test presented is also zero for all sources, with the SS also zero, even though a total DOF of 44 for this system was calculated.

4.5 Process Variation and Interactions: Gage R&R and Experiment

Table A.4 was presented to understand different sources of variation within the system and their percentage contribution to the process variation within Gage R&R. As indicated in Table A.4 (Appendix A), there were no variations in the system after the improvement was made in the measurement system. All sources presented had a zero percent contribution according to the data presented in Table A.5 (Appendix A).

Since the process capability of the SF laminating unit was improved, the equipment was ready to run the experiment and produce the required result. The experimental runs were arranged according to the L_{18} OA presented in Table A.6 (Appendix A). The experiments were performed in random order. Table A.6 (Appendix A) further represents a total of 8

process parameters that were studied using the TM in order to improve the adhesion between the PA and PE film. All the process parameters involved in the SF laminating unit are presented in Table 4.3 below, with their operating level. Seven factors were kept at 3 levels, while only one factor was kept at 2 levels. Thus, a TM based L_{18} was utilised. Table 4.3 demonstrates all process variables involved in this study with a unit of measure for each variable presented in the last column.

Table 4.3: Solvent-free lamination parameters at different levels

Parameter	Independent Variables			Units
	L 1	L2	L3	
Rewind Tension (RT)	80	100	120	N
Taper Tension (TT)	15	25	35	%
Surface Energy (SE)	40	42	44	dyne/cm
Coating Weight (CW)	1.5	2.0	2.5	gsm
Machine Speed (MS)	150	180	200	m./min
Application Temperature (AV)	35	45	55	^{o}C
Mix Ratio (MR)	75	85	95	%
Curing Temperature (CT)	28	32	-	^{o}C

A series of experimental runs were conducted to improve AS and maximise laminate productivity as tabulated in Table 4.4, using a TM based OA. As displayed in Table 4.4, all investigated SF lamination design parameters were arranged according to L_{18}. During the run, 7 parameters were investigated from level 1 (L_1) up to level 3 (L_3). The total DOF was calculated to quantify the total number of experiments required for the study using variables presented in Table 4.3 and their levels. The calculations showed that the DOF for all parameters was 17, thus L_{18} OA was utilised.

After quantifying the total number of experiments, input variables were tabulated using TM OA (L_{18}) presented in Table A.6 (Appendix A). This was done as part of the planning and preparing for the experiment and arranging all process parameters according to OA (L_{18}). During the arrangement of the process parameters, curing temperature (CT) was kept at level 1 and 2; all other variables were kept at level 1 (lower level), level 2 (intermediate Level), and level 3 (high level), respectively.

Table 4.4 presents a total of 18 experiments conducted with their output responses. All variables were arranged according to their levels as presented in Table A.6 (Appendix A)

and Table 4.3. The measurement of the AS (N) and TS are also presented in Table 4.4. All measurements and calculations were done after 48 hours of the curing process. The curing process is one of the significant steps in SF lamination as it facilitates bonding between PA and PE, thereby allowing the reaction of the SF adhesive system to take place. Table 4.4 also presents the S/N ratio for both AS and TS. This is critical to statistically identify the most influential variable during lamination and quantify the contribution of variables to the output responses. The two considered output responses after lamination are AS and TS. The S/N ratio for AS is represented by SNRA1AS, while the S/N ratio for TS is represented by SNRA2. Since the aim of this research was to yield a much higher output response, a *higher the better* approach to the S/N ratio was identified with the aim of fully optimising the lamination process.

Table 4.4: Experimental result with output responses

Run	Columns								Response Variables					
	CT	RT	TT	SE	CW	MS	AV	MR	Adhesion Strength (N)	Tensile Strength (S)	SNRA1AS	SNRA2 TS	MEAN1 AS	MEAN2 TS
1	28	80	15	40	1.5	150	35	75	350	12.53	50.88	21.96	350	12.53
2	28	80	25	42	2	180	45	85	490	17.61	53.80	24.92	490	17.61
3	28	80	35	44	2.5	200	55	95	355	13.42	51.00	22.55	355	13.42
4	28	100	15	40	2	180	55	95	370	14.23	51.36	23.06	370	14.23
5	28	100	25	42	2.5	200	35	75	460	16.44	53.26	24.32	460	16.44
6	28	100	35	44	1.5	150	45	85	640	21.02	56.12	26.45	640	21.02
7	28	120	15	42	1.5	200	45	95	450	16.24	53.06	24.21	45o	16.24
8	28	120	25	44	2	150	55	75	550	19.13	54.81	25.63	550	19.13
9	28	120	35	40	2.5	180	35	85	300	11.84	49.54	21.47	300	11.84
10	32	80	15	44	2.5	180	45	75	565	20.03	55.04	26.04	565	20.03
11	32	80	25	40	1.5	200	55	85	300	11.84	49.54	21.47	300	11.84
12	32	80	35	42	2	150	35	95	445	15.39	52.97	23.74	445	15.39
13	32	100	15	42	2.5	150	55	85	440	15.01	52.87	23.53	440	15.01
14	32	100	25	44	1.5	180	35	95	590	20.90	55.42	26.4o	590	20.90
15	32	100	35	40	2	200	45	75	300	11.84	49.54	21.47	300	11.84
16	32	120	15	44	2	200	35	85	560	19.67	54.96	25.87	560	19.67
17	32	120	25	40	2.5	150	45	95	380	14.23	51.60	23.06	380	14.23
18	32	120	35	42	1.5	180	55	75	450	16.24	53.06	24.21	45o	16.24

Based on the result demonstrated in Table 4.4 above, a maximum AS of 640 N was achieved with a S/N ratio of 56.12. Under these conditions, CT was kept at 28 ^{o}C which is the lower level, the RT was achieved at the intermediate level of 100 N, TT was kept at the highest with 35%, the SE was recorded at 44 dyne/cm, with CW kept at 1,5 gsm, the MS was also kept at a lower level with 150 m/min, the AV had to be kept at the intermediate level of 45 ^{o}C, and ultimately, the MR was kept at the intermediate phase of 85 N. After the experimentation, it was necessary to analyse the behaviour of the process parameters in order to demonstrate the most influential process parameter to the AS (N) using the experimental data presented in Table 4.3. As described above, a higher the better approach was applied and identified critical parameters to this research, as presented in Figure 4.6 below for S/N ratio for AS.

4.5.1 Signal-to-noise ratio: Adhesion strength

Figure 4.6 presents the main effects plot for the S/N ratio. A minimum S/N ratio of 49.56 was accomplished during runs 15 and 9. Under these conditions, surface energy (SE) was kept at the lower level of 40 dyne/cm on both runs.

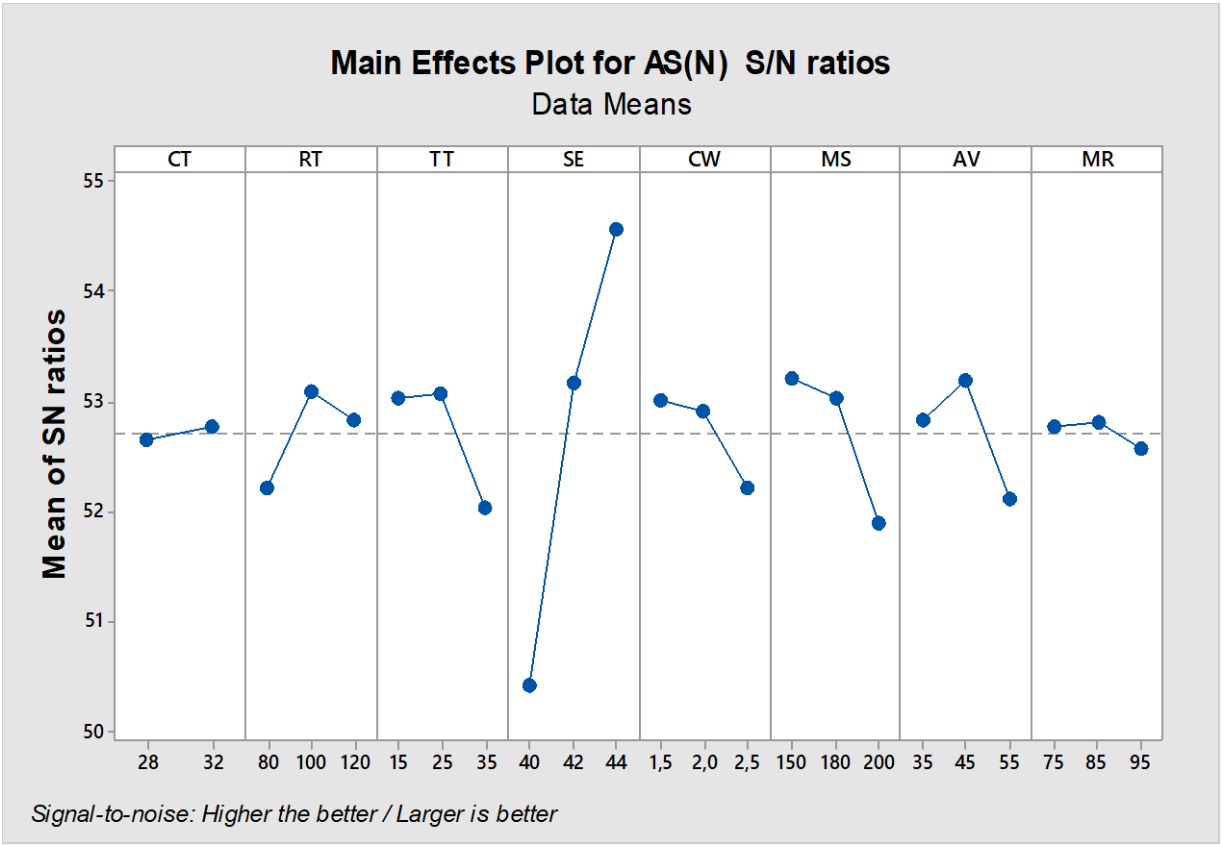


Figure 4.6: Main effects plot for means

The MS and AV were also kept at the same level for both runs at 180 m/min and 35 ^{o}C, respectively. The CT was kept at a lower level on run 9 which is 28 ^{O}C, and for run number 15, the CT was kept at a high level of 32 ^{O}C. These results portray that there is less effect caused by the CT on the output response. After determining the statistical significance on controllable parameters, it was also necessary to study how each variable affects the behaviour of another using the main effect plot for means (AS) in Figure 4.6 above.

Figure 4.7 below demonstrates an increasing trend for SE from 40, 42 and 44 dyne/cm, and a decreasing trend for CW since a lower mean can be produced at 2.5 gsm. The MS also demonstrates a decreasing trend and its effect on AS can be compared with the one for CW since they both produced a lower mean of means at level 3 (high level).

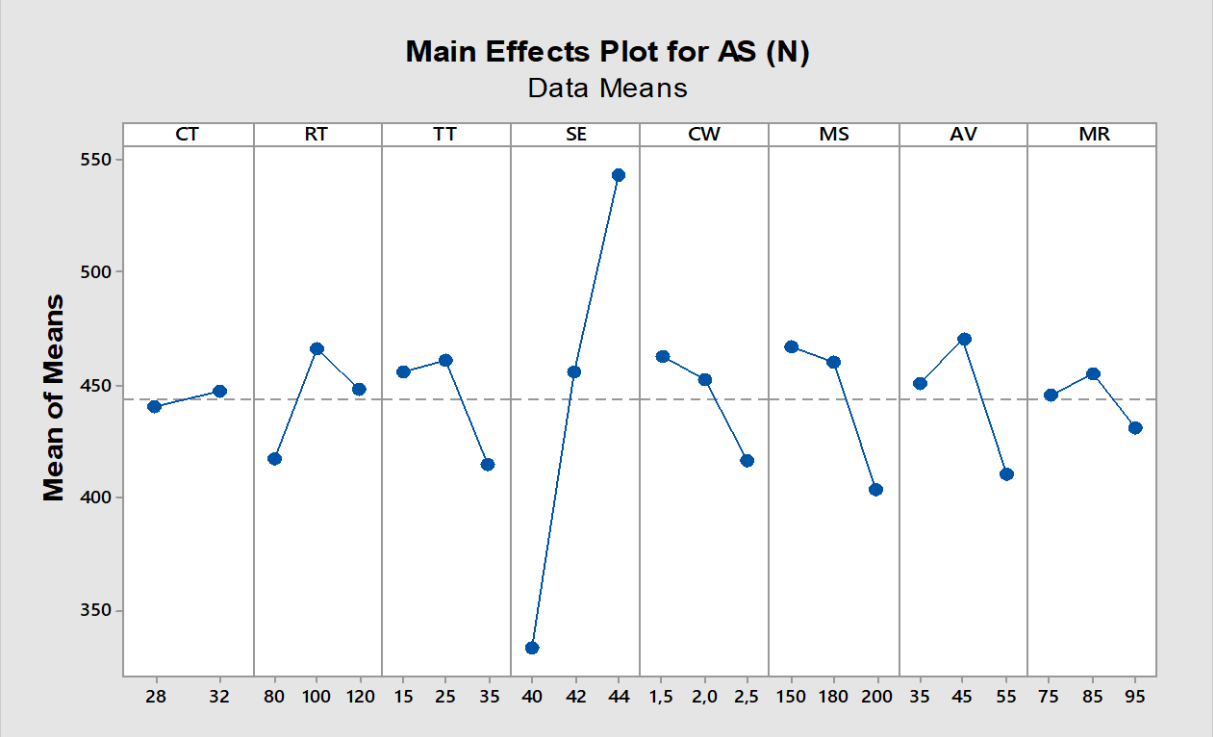


Figure 4.7: Main effects for means data means

Figure 4.7 above demonstrates that RT has a high effect at the intermediate level, while at high level its effect can be neglected. The MS and SE possess a greater effect on the output response since lower MS provides high SE. In that regard, these results indicate that at lower speed, more corona treatment is distributed on the film during the run, thus high adhesion is achieved under these conditions. The intermediate levels of RT have proven to be significant on the main effect of the means as this ensures that the effect of cleavage and shear stress is moderate for adhesion to occur precisely during processing. The TT was also found to be significant at the immediate level of 25%. MR proved to be statistically significant at the

intermediate level which is 85%. The MR result further demonstrates that AS and TS do not depend on high distribution of the adhesive system. Table 4.5 below presents the S/N ratio for process parameters involved and their behaviour against the AS (N). The S/N for each parameter at different levels were reviewed. The delta and the rank level for each parameter are tabled and arranged according to their significance.

Table 4.5: Response table for signal-to-noise ratios: Larger is better

Level	CT	RT	TT	SE	CW	MS	AV	MR
1	52.65	52.21	53.03	50.41	53.02	53.21	52.84	52.77
2	52.78	53.10	53.07	53.17	52.91	53.04	53.2	52.81
3	-	52.84	52.04	54.56	52.22	51.90	52.11	52.57
Delta	0.13	0.84	1.03	4.15	0.8	1.31	1.09	0.24
Rank	8	5	4	1	6	2	3	7

After analysis of the S/N ratio, it was also necessary to present this group of parameters involved in a lamination process in a response table of means, as demonstrated in Table B.1 (Appendix B). Table B.1 (Appendix B) also presents the means of means at different levels for each process parameter. Table B.1 (Appendix B) demonstrates that the highest mean can be achieved when the SE is set at level 3, while the lower mean can be achieved when the SE is at level 1, respectively.

4.6 Effect of Process Variables on the Output Response

Table 4.4 demonstrated the result of the experiment conducted using OA. All output responses and S/N ratios were demonstrated in the table. In Figure 4.5, the main effect plot of the S/N ratio was plotted to identify the most significant parameters in the SF laminating system. According to the S/N ratio plotted, the SE was the most significant variable with a value of 56.12 and an output response (AS) of 640 N. Based on these results, the polarity of the films become the most significant factor within the studied process parameters. This is demonstrated by the high value of the SE which is 44 dyne/cm at level 3. Therefore, the identified optimum conditions are presented in a separate table – Table 4.6 below:

Table 4.6: Optimum setting based on the main effect of plot for AS

SE	TT	CT	CW	MS	AV	MR	RT
44	35	32	1.5	150	45	85	100

The analysis conducted in this research was based on using the Taguchi based design of experiments to quantify the contribution of each variable on the output response. This was done to achieve high production of PA and PE laminate. The higher the value of S/N, the greater the production output. After using the above process parameters, a lot of material was recovered after production. The main plot in Figure 4.5 demonstrates the significant parameters to achieve high AS. The larger the better S/N ratio was chosen for the AS. The response in Table 4.5 for the S/N ratio quantified the delta for each factor during the experimentation, with SE having the highest value of 4.15 followed by MS with 1.31 and AV with the value 1.09 as the third significant variable. Table 4.5 and Table B.1 (Appendix B) also provide delta and rank each process variable according to its significance on the AS and TS. Again, the corona treatment (SE) distributed on the film proved to be the most significant variable with a delta of 210, and was ranked at level 1 according to importance. Moreover, according to the response in Table B.1 (Appendix B), MS is the second most significant parameter raked at number 2 with a delta of 46.4. The AV is the third most important variable with a delta of 60 and was ranked at number 3.

4.7 Result for Tensile Strength (TS)

The first section of the experiment was based on optimising the lamination process to achieve high AS between the PA and PE films in order to maximise the productivity. The second section involved measuring TS during sampling to find the appropriate time taken to break a well laminated sample of PA and PE film. The second approach was performed to identify the relationship between AS and the amount of time required to break a well laminated material. TS samples were taken from the same reel as for AS. The time taken to break each sample was measured using the same Instron. All the TS results were presented in Table 4.4 with the AS result. Figure 4.8 below is plotted to analyse the results and effect of each parameter on the TS. The application of S/N ratio was then used to understand the statistical significance of each variable on the TS.

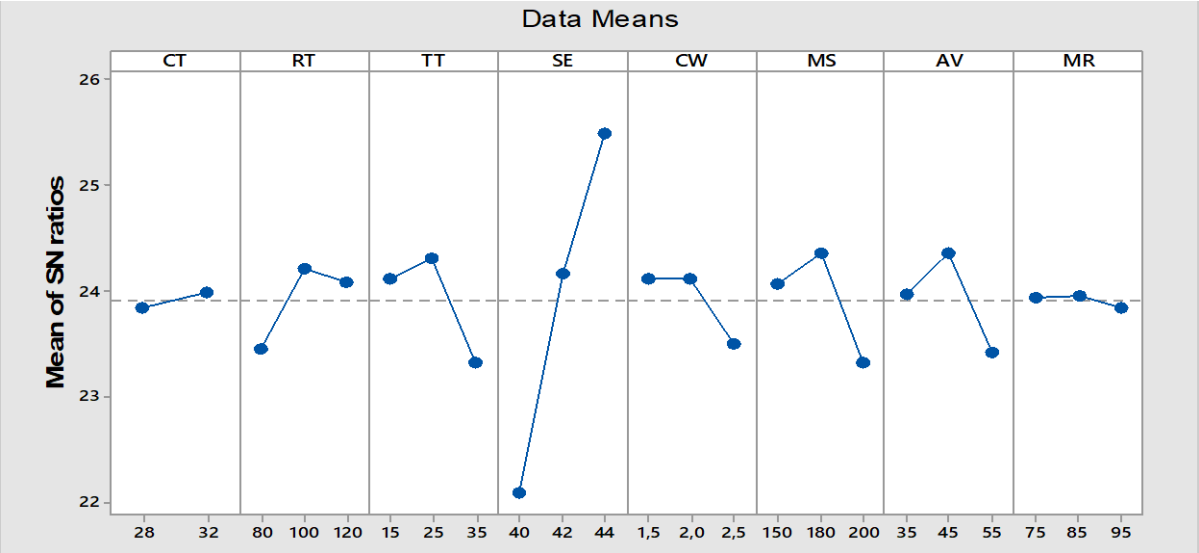


Figure 4.8: Main effects plot for SN ratios

Figure 4.8 above shows the main effect of the plots for the TS, revealing a similar behaviour of parameters as the one shown in the AS in Figure 4.7. The SE has once again proven to be critical as it showed statistical significance in Table 4.7. The TS was achieved when the operating conditions were set as follows: The CT at 32 ^{o}C; RT at 100 N; TT at 25%; SE at 44 dyne/cm; CW at the intermediate level with 2 gsm; MS at 180 m/min; AV at 45 ^{o}C; and lastly, MR at 75%. Figure 4.9 was plotted to understand the behaviour and the influence of the process variables on the strength of the studied films.

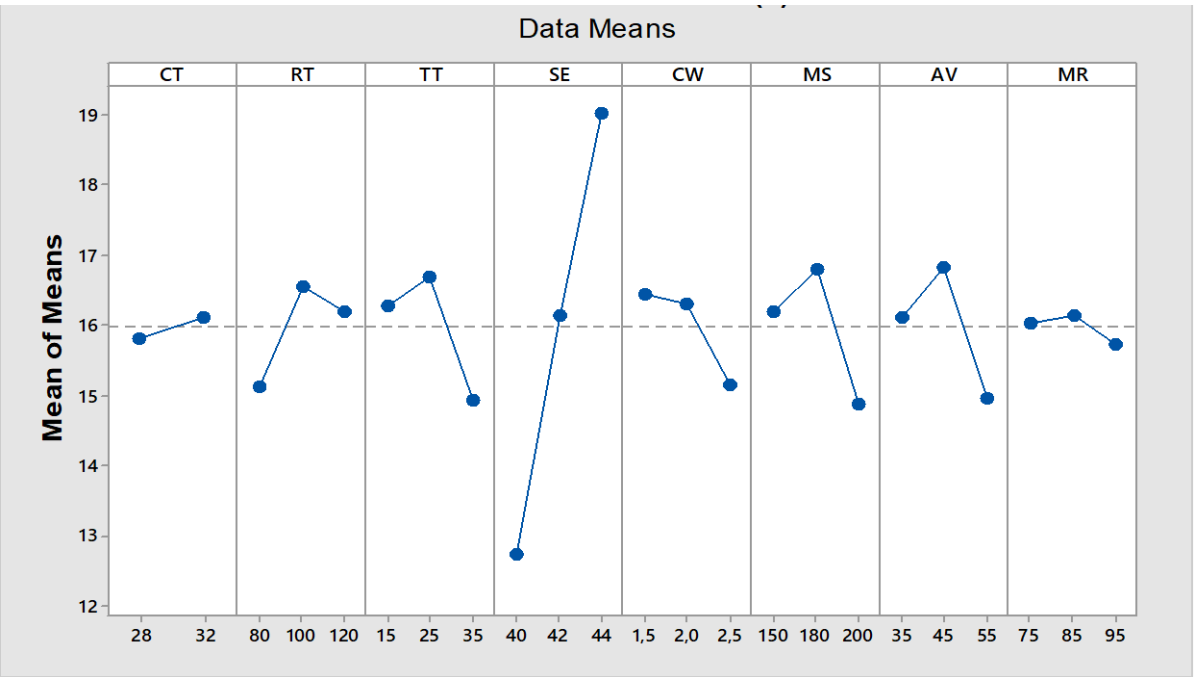


Figure 4.9: Main effects plot for means

Figure 4.9 further demonstrates that the MR variable is not significant when compared to the previous studied output response. The AS requires MR at the intermediate level (2) to achieve high AS; however, in this case, the TS required the MR to be at a lower level (1), but can still achieve the same result at the intermediate level (2). The SE continued to show an increasing trend from 40, 42 and 44 dyne/cm. Once again, the SE has become one of the most influential process variables at 44 dyne/cm. The RT, TT, MS, MR, and AV proved to be effective only at the intermediate level (2).

The analysis and the effectiveness of the process parameters were tabulated in Table 4.7 for S/N ratio for TS. The larger is better was again chosen to satisfy the required higher TS on the two laminated films. Table 4.7 further demonstrated a S/N ratio for each parameter at different levels. The delta and the rank were also calculated and documented below according to the significance of each process parameter.

Table 4.7: Response table for signal-to-noise ratios: Larger is better

Level	CT	RT	TT	SE	CW	MS	AV	MR
1	23.84	23.45	24.11	22.11	24.12	24.06	23.96	23.94
2	23.98	24.20	24.30	24.15	24.12	24.35	24.36	23.95
3	-	24.08	23.32	25.49	23.29	23.31	23.41	23.84
Delta	0.14	0.76	0.98	3.41	0.62	1.03	0.95	0.11
Rank	7	5	3	1	6	2	4	8

According to Table 4.7, SE is again the most significant variable with a delta value of 341 and ranked at number 1. The highest achieved S/N ratio is 25.49, while the lowest achieved S/N ratio is 22.11 at level 1 for SE, respectively. The MR had both the lowest rank and delta which is different from the analysis of AS where CT had both the lowest delta and highest rank in Table B.1 (Appendix B). Therefore, these results have proven that CT is more influential than MR in this regard. MS is the most influential variable according to the plot. TT is the third most significant variable, followed by AV which is the fourth most significant variable at number 4. The RT is ranked at number 5 with a delta of 0.75, while CW is ranked at number 6. CT had a delta of 0.14 with a rank of 7.

Table 4.8 below presents the response table for the means of each level with the rank and delta, respectively. The highest achieved response at level 3 of the SE is 19.03, while the lowest achieved response mean for the same parameter but at a lower level was 12.75. The

results were plotted to understand the behaviour of the different parameters in each level with respect to the TS.

Table 4.8: Response table for means

Level	CT	RT	TT	SE	CW	MS	AV	MR
1	15.83	15.14	16.28	12.75	16.46	16.22	16.13	16.03
2	16.13	16.57	16.69	16.15	16.61	16.81	16.83	16.17
3		14.96	14.96	19.03	15.16	14.91	14.98	15.73
Delta	0.14	1.73	1.73	6.28	1.3o	1.90	1.85	0.43
Rank	8	5	4	1	6	2	3	7

The second output response that was studied in this paper is the TS which defines the amount of time taken by the tensile tester to break a 10 mm x 10 mm laminated sample of PA and PE film. The highest TS recorded after the experimentation was 21.02 seconds. The results were recorded after the run number 6 on the OA. Figures 4.8 and 4.9 demonstrate the main effect for plot S/N ratio and the main effect of the plot for means. Based on these plots, the optimum operating conditions for optimising the SF lamination process and achieving a high output response for TS were 44 dyne/cm for SE, 180 m/min for MS, AV is at 45 ^{o}C, 1.5 for CW, 25% for TT, 100 N for RT, and 32 for CW.

Table 4.6 demonstrated the influential process parameters on TS. Again, SE is the most statistically significant variable with a delta of 3,41 and a rank of 1 as provided by the table. For Table 4.8, the output response for the mean, AV is the third most significant variable, while this is not the case for the S/N ratio (Table B.3) (Appendix B). Table 4.7 further depicts that CT and MR are the least significant parameters within the SF laminating system as the parameters are ranked 7th and 8th in position. Although the position of the least significant variable in Table 4.8 is different from Table 4.7, the parameters are still the least influential variable according to the rank and delta.

Figure 4.10 below was plotted to understand the effect of every parameter to one another and the possible interaction to achieve the required AS. The figure was plotted since the effect and behaviour of each parameter can depend on the level and influence of another parameter as was seen in the above analysis.

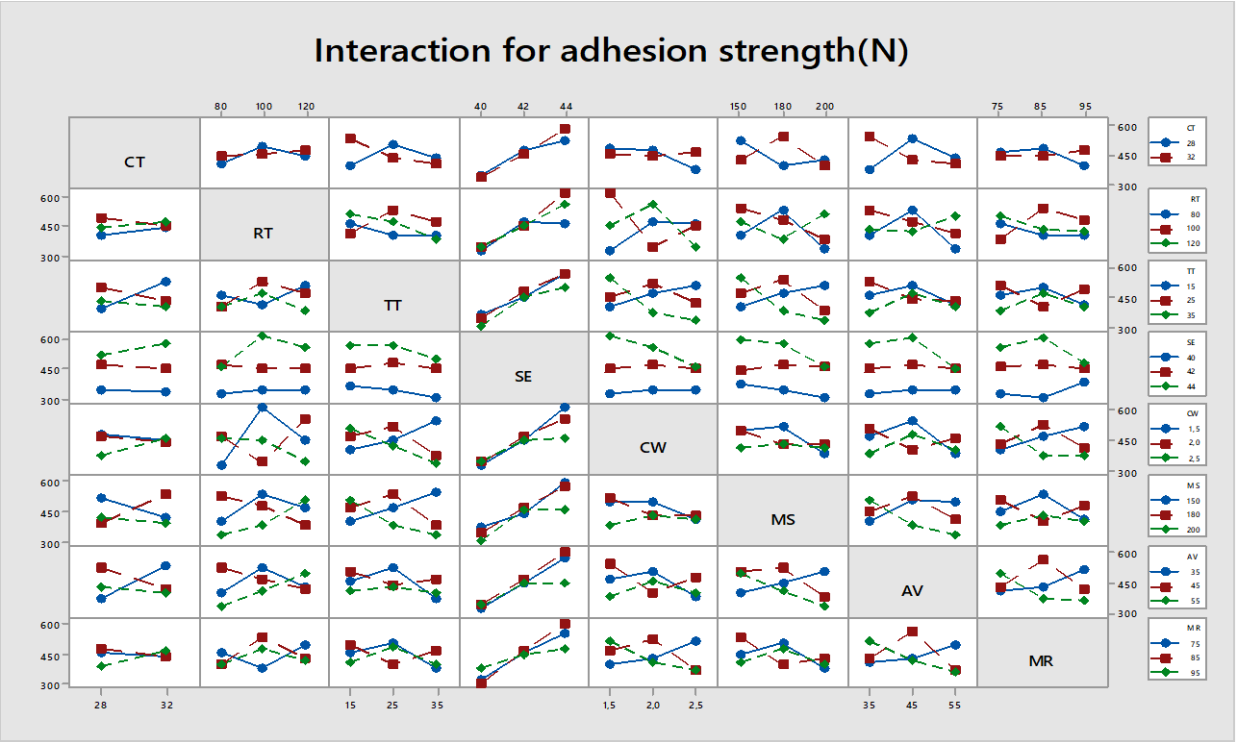


Figure 4.10: Interaction plot for adhesion strength (N)

Although it can be seen from Figure 4.10 above that there is minor interaction between SE and MR at the bottom of the figure, these interactions can be neglected since the MR and CT are not significant according to the S/N ratios studied for each parameter at different levels. A cross-over interaction exists between CT and TT, but is too insignificant to be considered. The SE and MS are the only variables that demonstrated exponential interaction, but were only partially significant. Therefore, it becomes critical to understand the level of interaction between the variables before generating predictive equations and linear models since these interactions impact the output when excluded in the equation, especially in cases of parameter significance. However, the figure demonstrates that interaction in this study is insignificant even when developing a model. After studying the effect and interaction between the variables, a linear model was developed, as demonstrated in Table B.2.

Table B.2 presents a linear model analysis conducted at each level of the parameter with an estimated model coefficient for the means. The developed model can be used to predict the mean values at different levels. Furthermore, a t-test and probability values are plotted in order to indicate the significance of these values under the studied level. The interactions are not included in the table since they possess limited interaction. Therefore, interactions would have been added only if they were significant in the estimated model. According to Table B.2, the SE is the only significant variable with a P-value of 0.029 at 40 dyne/cm. The

estimated constant for the model was 444.167. After presenting the estimated model for the means above, it was necessary to study the ANOVA for means in Table B.3 (Appendix B).

The ANOVA in Table B.3 (Appendix B) for the means presents the total DOF for the system and the residual error involved. The ANOVA in Table 4.9 presents the contribution of each process variable on the output response. The F-test and P-value test are also presented in the table below.

There were 17 DOFs calculated for this study with a total of 190062 for Seq SS. The maximum Adj SS and Seq SS were achieved only for the SE variable. CT had the lowest Seq SS and Adj SS value when compared to all the variables presented in this study. The highest achieved contribution was 70.25% for SE and the lowest achieved contribution was for CT at 0.12%.

Table 4.9: Analysis of variance for means

Source	DOF	Seq SS	Adj SS	Adj SS	F-Value	P-Value	% Contribution (SS/Total SS)
CT	1	235	235	234.7	0.07	0.814	0.12
RT	2	7408	7408	3704.2	1.12	0.471	3.90
TT	2	7758	7758	3879.2	1.18	0.459	4.08
SE	2	133525	133525	66762.5	20.27	0.047	70.25
CW	2	7158	7158	3579.2	1.09	0.479	3.77
MS	2	14533	14533	7266.7	2.21	0.312	7.65
AV	2	11200	11200	5600.0	1.70	0.370	5.89
MR	2	1658	1658	829.2	0.25	0.799	0.87
Residual Error	2	6586	6586	3293.1			3.43
Total	17	190062					

The above presented calculation and models only focused on the coefficients for means. The next section focuses on the linear model and coefficient for the S/N ratio. Table B.2 (Appendix B) presents the linear model analysis for the S/N ratio for all the process variables involved in a lamination process. Table B.3 (Appendix B) further presents the linear model analysis and estimated coefficient of parameters involved for the S/N ratio at different levels. The probability values and the F-values are also presented in the table. The constant achieved for this section is 52.7139. All estimations are done for values lower than the optimum operating condition. Table B.3 (Appendix B) was plotted to evaluate the contribution of the process variables to the output response. Since all the required models were created in Tables B.2 and B.3 (Appendix B), the estimated models were used to predict the AS and S/N

ratio at a higher operating level. This was done to validate and verify the accuracy and precision of the created models. Table 4.10 further presents the predicted values using the model. Therefore, an experimental error was calculated to verify the accuracy of the model as previously mentioned.

Table 4.10: Experiment and predicted data

Variables	Experimental Values	Predicted Values	Experimental error (%)
Mean	640	613,056	4.2
S/N	56.12	55.5737	0.97

Tables B.2 and B.3 (Appendix B) detailed the linear model analysis which estimated model coefficiencies for the means at lower levels than the optimum conditions identified. Based on the result, the estimated constant is 444.167 with S:57.39, R-Sq of 96.5% and R-Sq (Adj) of 70.5%. The ANOVA in Table 4.9 and Table 4.10 was presented to quantify the contribution of each process variable to the AS which is the main output response of the project. According to Table 4.9, SE has 70.25% contribution to the AS, while MS has about 7.65%, and AV is the third most contributed variable statistically (5.89%). Table 4.10 shows estimated model coefficients for the SN ratios with a constant of 55.7139 with S: 1.169, R-Sq = 96.4% and the R-Sq (adj) of 69.1%. Table 4.11 demonstrates the optimum AS value of 640 N with a predicted output response of 613.056 N at a same parameter level. Based on the actual experimental value, the predicted experimental error for AS is 4.2%. The actual optimum value for the S/N ratio from the experiment is 56.12 and the predicted value was 55.573, using the same parameter level of the optimum condition. The experimental error of the S/N ratio determined was 0.97%.

In order to study existing variability and interaction points for the AS, it was necessary to compute a regression analysis in the ANOVA Table B.4 (Appendix B). The P-values were presented to identify significant variables in the model developed. The F-test and DF were also presented in the ANOVA table.

The model summary demonstrates an R-sq of 85.75%, R-sq (adj) 73.09%, R-sq (pred) and S of 54.853. This result demonstrates a very good fit to the model established and a great coefficient of determination for both the independent and dependent variables for AS. Table B.5 (Appendix B) presents all coefficients for the generated model. As discussed, there is no special interaction plotted because of less variability and few changes on the slope of the model generated. Therefore, a regression model was developed as explained below.

4.8 Regression Equation

The regression equation was developed to predict the behaviour of the system.

AS (N) = -1388 + 1.81 CT + 0.771 RT – 2.04 TT + 52.50 SE – 46.7 CW – 1.184 MS – 2.00 AV – 0.71 MR

Fits and Diagnostics for Unusual Observations

Obs	Adhesion Strength (N)	Fit	Resid	Std Resid	
1	350.0	422.6	-72.6	-2.43	R
3	355.0	431.7	-76.7	-2.48	R

R Large residual

Residual plots for AS

Although Figure 4.10 demonstrated some interaction between the variables, for regression analysis these interactions can be neglected since they do not affect the behaviour of the input variables during processing. After creating a well-structured model, a residual plot was presented in Figure 4.11 to understand how well the data is distributed towards the created model.

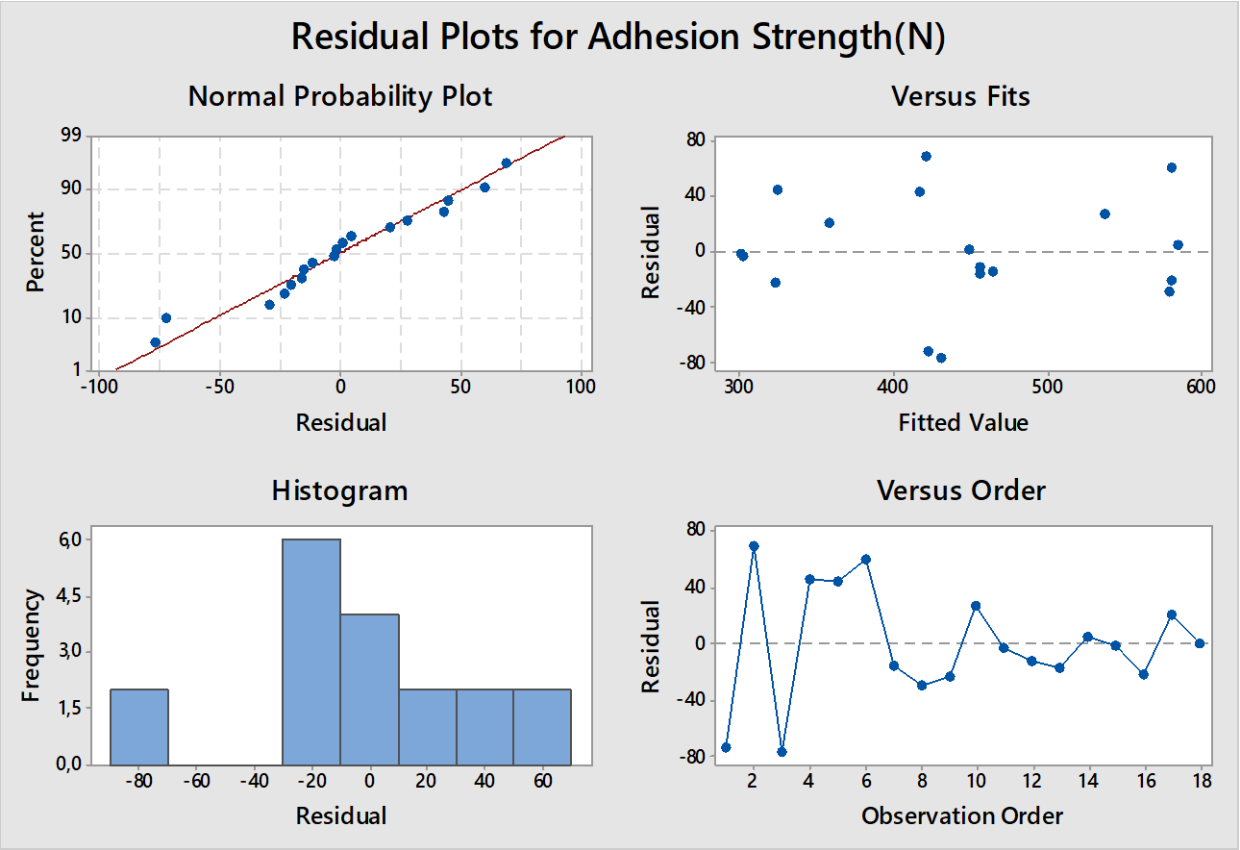


Figure 4.11: Residual plots for adhesion strength

The ANOVA method demonstrated in Table 4.9 was performed to identify the most significant input on the system. Furthermore, Table 4.9 validated that the SE is the most significant variable when compared to other process parameters with a P-value of 0.00. According to ANOVA, all variables with a P-value greater than 0.05 are not significant, while process parameters with a P-value less than 0.05 are considered to be significant. Another most significant variable in this study is the MS which has the P-value of 0.093. As demonstrated in the interaction, there seemed to be a great relationship between the MS and SE during processing. The interaction plotted demonstrated that the effect of SE distribution and the MS are the most significant process parameters as it was also quantified by the S/N ratio. The S/N ratio was evaluated to identify the influence of controllable parameters on the output response.

The ANOVA approach was used to quantify the effect of the input on the output. Figure 4.11 portrays that residuals are normally distributed according to probability plot. The residual versus fit value curves in the figure further demonstrates little and equal variability on the plot. There is systematic curvature on the residuals which depicts errors in the developed model. The histogram and observation order plot depict that improvement can be done to

ensure that the data is well distributed around the mean value in order achieved a best fit of the residuals.

4.9 Summary of Chapter

Process optimisation and modelling of an SF lamination was conducted in order to study the effect of the design parameters on the output response. To accomplish the objective of this chapter, Gage R&R was conducted first to minimise any sources of variation that could be the result of the system, instrument, or personnel. A total of 45 measurement trials per each operator were conducted to study the variation, and crossed Gage R&R was selected for this study. This is because each plastic roll manufactured/part is measured by individual personnel. For analysis, a Gage R&R ANOVA method was selected because it is considered to be more accurate than the X-Bar method. The Gage R&R by ANOVA technique considers part to an individual doing the measurement, where the Gage R&R X-Bar considers part-to-part.

To study R&R, 3 different operators were allowed to set the metering gap which depicts the amount of CW distributed on the film. Each individual conducted 10 different measurements on each part of a SF laminating system, apart from Gage R&R. Another aim of these experiments was to identify the effect of poor cleavage stress and shear stress on interfacial adhesion.

The contribution of each source of variation was studied with the ANOVA methodology, as demonstrated in Table 4.3. This focus was more on AV, MR, SE, MS, CW, CT. It was found that there is a greater relationship between the AS of the laminate and TS as demonstrated by the run number 6 where the AS is 640 N and the TS is 21.02 second. Both output responses were at an optimum level at run 6. Based on this, the higher the AS the greater the TS. According to the ANOVA, SE was found to be the variable that contributed the most during the SF lamination. MS was the second most influential process parameter during processing. Therefore, the result proved that SE needs to be controlled at level 3 in order to achieve high AS and TS.

Attention now shifts to model effectiveness verification in Chapter 5.

CHAPTER 5

MODEL EFFECTIVENESS VERIFICATION

Chapter 5: Model Effectiveness Verification

5.1 Introduction

In this last decade, the use of flexible packaging materials has increased due to their low cost and ease of processing in comparison to other types of packaging structures, such as rigid packaging and glass bottles. However, during processing when process parameters deviate from the required targets, loss is exerted from the process. TM further describes these deviations as being caused by process variation (Lofthouse, 1996; Kumar et al., 2012; Cristian & Popescu, 2012). The QLF is an effective method utilised to quantify losses of the product. In this chapter, the QLF is used to calculate losses on each reel produced (Sharma et al., 2007). The identified losses are based on customer dissatisfaction since the product is failing to meet the necessary requirement. When the produced product does not meet the desired condition, waste is generated which results in a re-work and use of utilities. This ultimately means an increase in production costs (Kumar et al., 2012). Thus, TM bases the QLF method on the fact that quality ought to be measured by how far the actual value is from the target and cease dependence on inspection. QLF techniques further stipulate that quality should not be measured by conformance on lower and upper specification but consistence on producing the required standard value (Gaitone et al., 2006). According to TM, process variation is the direct cause of waste and poor productivity, thus QLF is utilised to eliminate variation and the effect of uncontrollable factors during processing.

Figure 5.1 demonstrates the traditional approach of managing quality improvement, since the method is only based on the fact that the desired value should be based on the lower and upper specification.

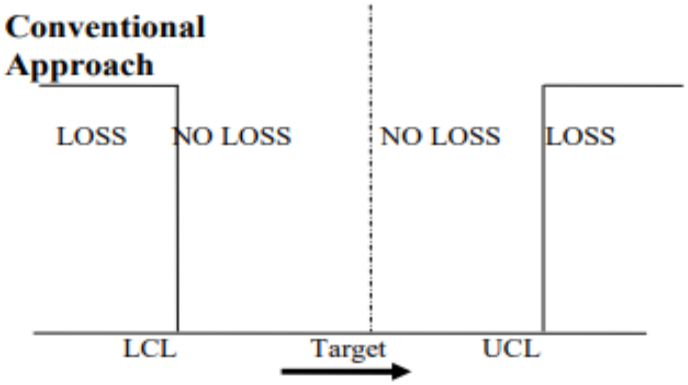


Figure 5.1: Traditional approach of managing quality

Figure 5.2 is based on the TM of maintaining a high degree and accuracy to ensure that the process output produces the desired result.

Figure 5.2: Taguchi approach of managing quality

The designed model has proven to be the key in achieving the required AS output result. The optimum AS value obtained in Chapter 4 was taken as the target value in order to calculate the constant (k) and loss function on each run. The QLF by TM can be categorised according to larger the better, smaller the better, and nominal the better. For the purpose of this study, a *smaller the better* approach was chosen to satisfy the research and ensure that the actual output does not deviate significantly from the desired value. The QLF uses statistical methods to drive process improvement in order to improve the output response, and this method follows an integrated approach in the DOEs. Ideally, the gap between the lower and upper spec shouldn't be so great as this detailed process variation and the significance of uncontrollable variables/NFs. Therefore, it has become critical to monitor the accuracy and precision of the SF lamination system.

TM designs can be separated into three categories, namely: *tolerance, parameter*, and *system* design. In recent years, there has been an increase in the demand of polymer structures that possess barrier properties to protect packaged products against the oxygen and water transfer rate (Yeh & Fan-chiang, 1997; Coltro & Borghetti, 2017; Sadiku-Agboola et al., 2010). These demands have raised the need for multilayer composite structures which can be obtained through SF lamination. Multilayer polymer composites are very complex, such that their molecular structures and polarity need to be activated to a greater height for them to be able to bond to one another because of their inert nature, and this has motivated the current study. As the world is increasingly moving toward polymers which are reusable, recyclable, and compostable, there has been a rise in the use of PU SF adhesive systems due their safety and environmental friendliness.

This chapter presents process modelling, selection of lamination adhesive, and the research methodology employed in this study. To validate the studied process, Minitab 17 software was used to study the contribution of different sources of variation on the output response. All trials performed in Chapter 4 were based on an industrial batch production process. As indicated in the literature reviewed in Chapter 2, there have been limited studies focusing on the contribution of process parameters on AS during SF lamination. After trials were performed and optimisation conducted, optimum levels of process parameters were determined. Furthermore, to achieve the objective of this chapter, the results discussed in Chapter 4 are used to create a model that can predict the behaviour of a SF laminating unit at different parameter levels. SF adhesives are crucial in accomplishing lamination of multi-composite structures for oxygen and water barrier properties.

To utilise the QLF and validate the effectiveness of the model, parameters in Table 5.1 below were used during the production of the new batch as optimum AS and TS were achieved through them. The highest achieved AS was 640 N and the lower limits (LCL_{AS}) and upper limits (UCL_{AS}) were used to calculate the QLF, respectively.

Table 5.1: Optimum operation condition

SE	TT	CT	CW	MS	AV	MR	RT
44	35	32	1,5	150	45	85	100

Optimum AS = 640 N, LCL_{AS} = 640 N - 2, UCL_{AS} = 640 N + 2

Optimum TS = 21.02 mm/s, LCL_{ts}= 21.02 mm/s - 2, UCL_{ts} = 21.02 mm/s + 2

Figure 5.3 below demonstrates the QLF before experimentation and QLF after experimentation. The QLF was calculated using the 18 runs performed to validate the effectiveness of the generated model.

Figure 5.3: QLF before the experiment vs QLF after the experiment

Figure 5.4 below demonstrates poor process capability of the AS distribution curve before experimentation, while Figure 5.5 further below shows a well distributed data point for TS. Both Figures 5.4 and 5.5 prove that the created model was effective to produce the design result.

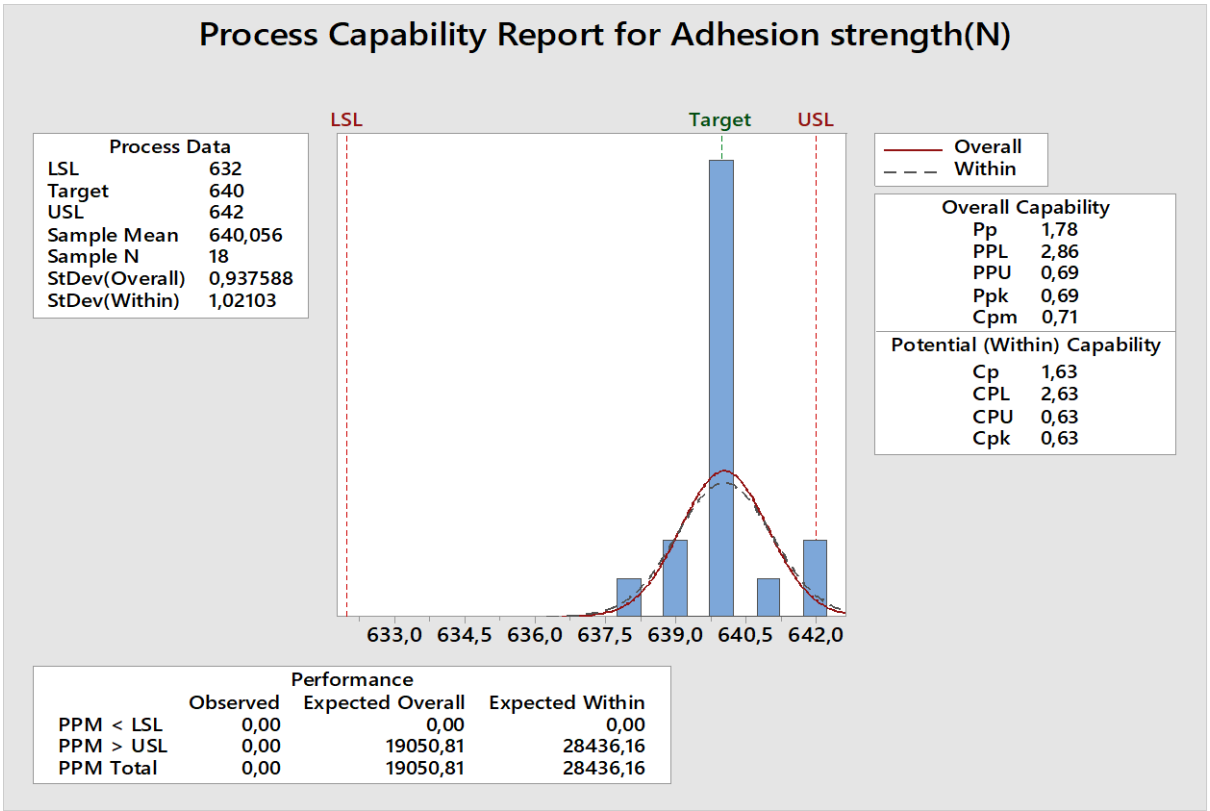


Figure 5.4: Normal distribution curve for AS

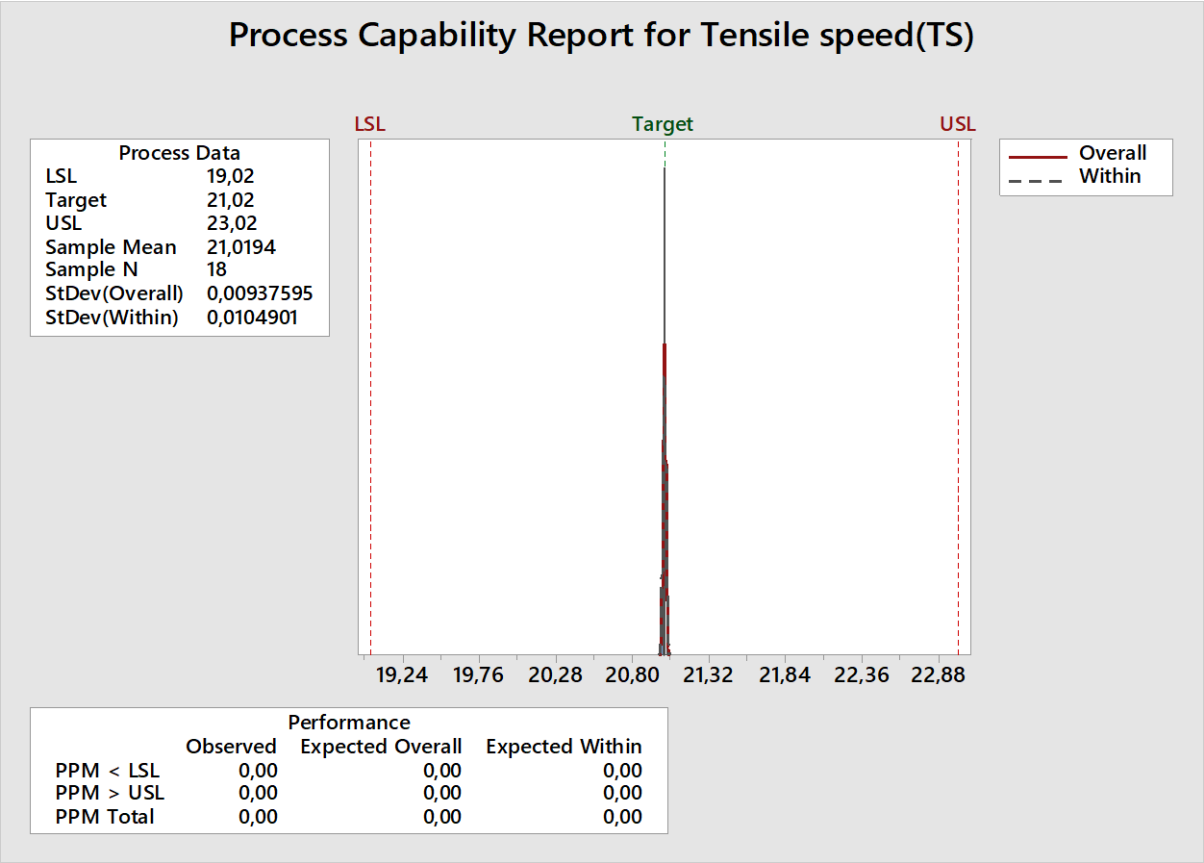


Figure 5.5: Normal distribution curve for TS

5.2 Process modelling and simulation overview

The discrete-event method was utilised to develop the model and simulation of the process using Minitab 17 software. Minitab is an efficient tool for designing an experiment with the aim of maximising the output response and developing simulation models that best describe systems at different levels. Through simulation and modelling, the performance of the existing produced product can be validated in order to increase efficiency while re-engineering the product for system design and configuration.

5.3 Process Description

SF lamination is a web multilayer process that combines chemical reaction and film tensioning during processing. The combination of a chemical reaction and web pull rate has brought complexity in the process to achieve consistent AS after bonding. As indicated in Chapter 2, the process has a lot of benefits, especially when there is a need for high barrier composite polymeric structures.

Figure 5.6 further below demonstrates a SF laminating unit with different sections within the system. During processing, the PE film is loaded on the unwind 1 where it is pulled by a

dancing roller to achieve consistent film tension throughout the unit. Therefore, the film is allowed to pass through the coating unit where the adhesive is distributed from the metering roller to the application roller all the way to the PE film. This is done to maintain uniform distribution of the adhesive. The composite PA is loaded on the unwind 2 section, where it is also pulled by the dancing roller to achieve a consistent tension. The PE and PA material are therefore joined together at a film joint section, as was demonstrated in Figure 5.3. All process parameters are based on the optimum levels achieved in Chapter 4. A first set was run in order to validate the amount of CW distributed. After 15 minutes of machine running, the machine is stopped to cut the sample to verify the amount of adhesive on the web. Once the adhesive distribution is validated and it is on specification, production is allowed to run accordingly.

5.4 Selection of lamination technologies

The use of adhesives in the flexible packaging industry depends on the polarity of the polymer molecular state, the environment at which the lamination process will be conducted, and the available system to process the adhesive. In many cases, bonding can be achieved through a single component; however, to ensure enough cohesion and adhesion, two-component adhesive systems are used due to their excellent physical properties. In this regard, a two-component isocyanate (OH) based PU adhesive was selected for this study to drive a SF lamination of PE and PA film. The two-component PU adhesive consisted of hardener and resins, which were mixed at a specified ratio during processing. Ideally, to enhance adhesion in any flexible packaging environment, polymer molecular structures are activated and re-arranged by applications such as flame treatment, plasma, and or corona treatment. However, in this study, corona treatment was applied to the web films to activate the molecules of the web substrates. The adhesive system was required to possess the thermal stability since processing is conducted beyond ambient temperatures. After selecting the best lamination technology, it was important to also identify the most critical components within the lamination equipment, as demonstrated in Figure 5.6, to ensure an efficient operation for the machine to produce the required result.

Figure 5.7 further below demonstrates a block flow diagram of the SF lamination unit. The block flow diagram demonstrates a flow of material during processing.

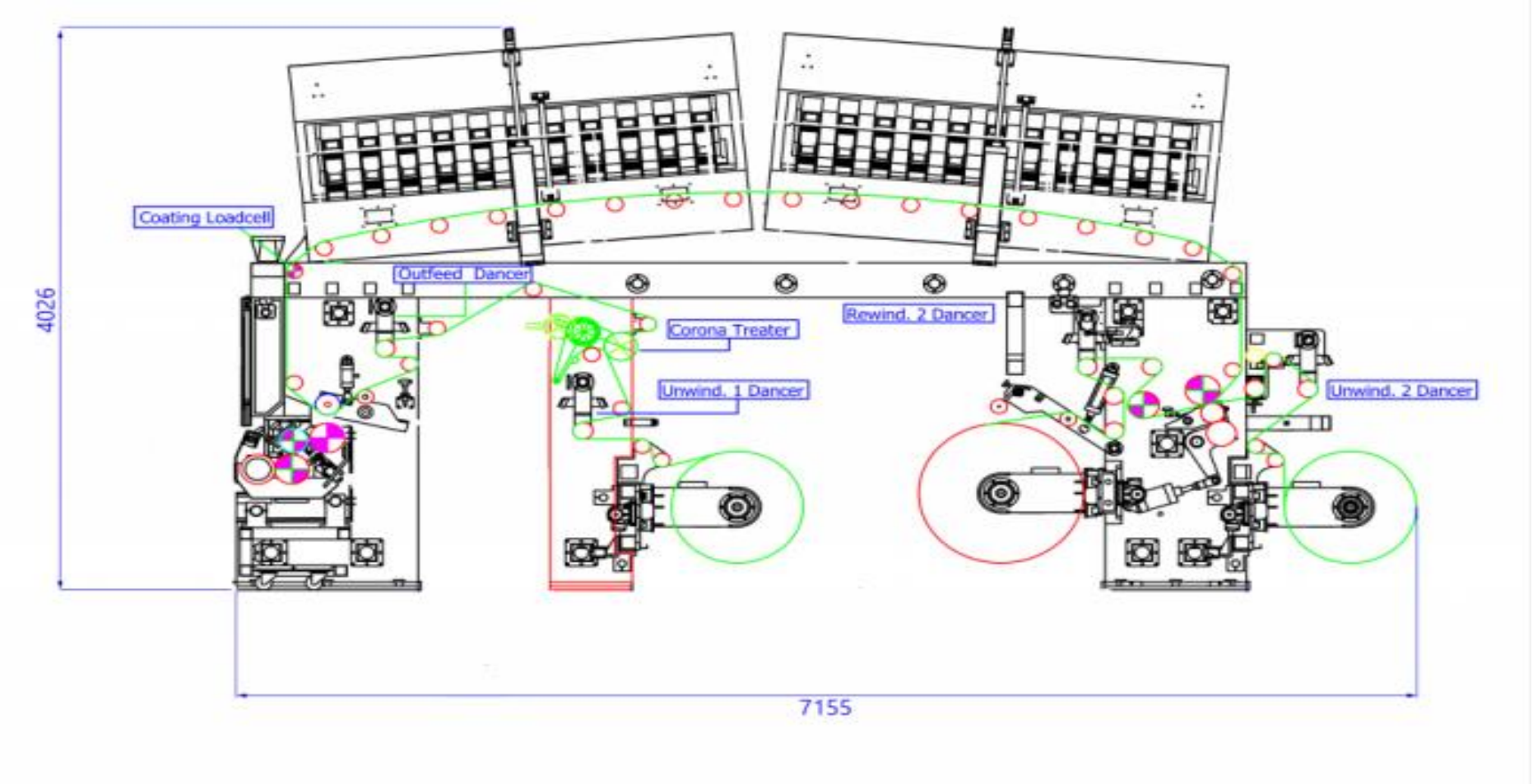


Figure 5.6: Configuration of a duplex solvent-free laminating system

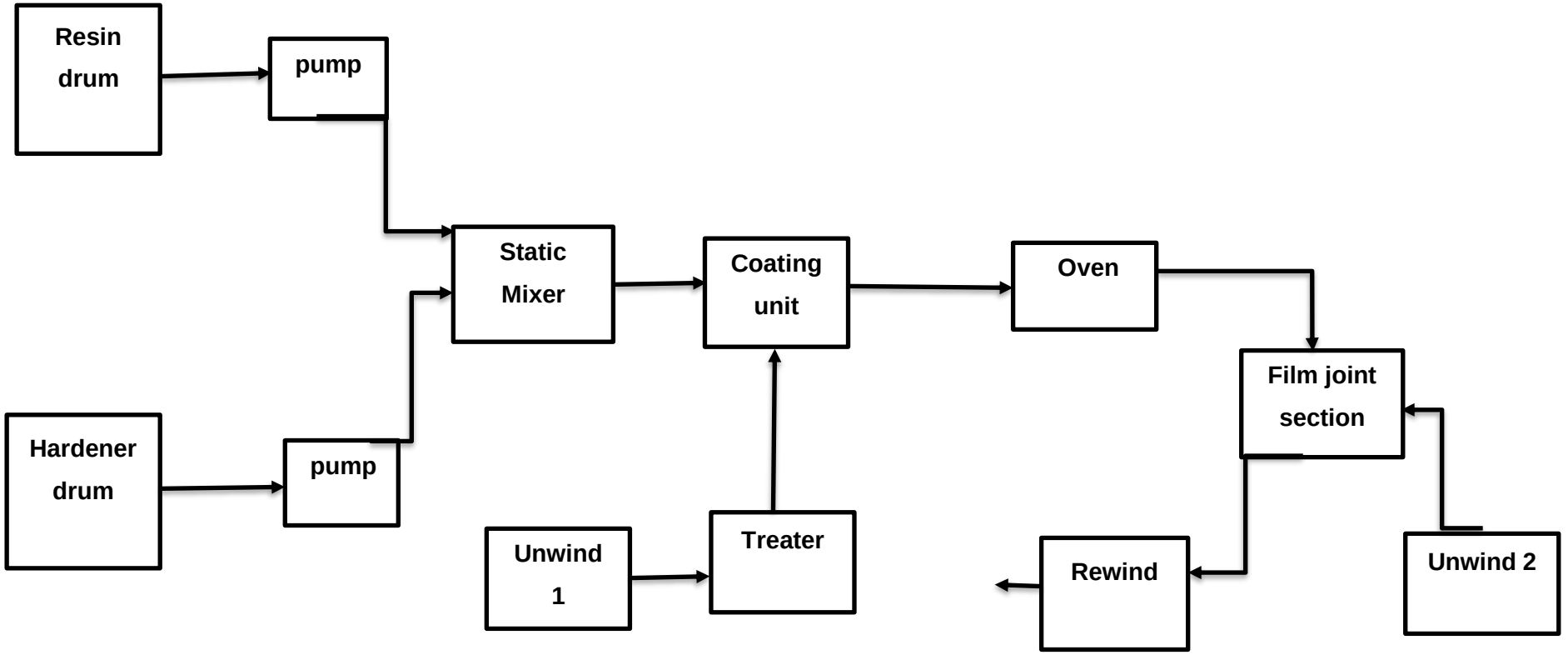


Figure 5.7: Block flow diagram of a solvent-free laminating system

5.5 Summary and conclusion of the feasibility study

This chapter demonstrated the result of the second experiment conducted using the optimum operating parameters achieved during experimentation and analysis of the result in Chapter 4. Based on the Taguchi QLF performed above, the model was very effective since optimum conditions were able to produce the expected output close to the required target of 640 N. As demonstrated by the normal distribution graph (Figure 5.4), all the achieved results were closely distributed around the target point. The above chapter further discussed the properties of SF lamination in order to provide constructive information on why the SF lamination was utilised over the SB lamination.

The following chapter focuses on SF economic feasibility.

CHAPTER 6

SOLVENT-FREE ECONOMIC FEASIBILITY

Chapter 6: Solvent-Free Economic Feasibility

6.1 Introduction

This chapter focuses on evaluating the economic feasibility of a SF laminating unit. The previous chapters – Chapters 4 and 5 – focused on developing a model with optimum process parameters and the effectiveness of the model, the selection of lamination adhesive, and the research methodology employed. The data generated during experimentation in Chapters 4 and 5 are used in this chapter to evaluate the economic feasibility of the model that was created. Chapter 5 also quantifies the contribution of each design parameter to the output response.

The presented literature demonstrated the importance of understanding the properties of different adhesive systems with their benefits on maximising output and increasing profitability (Davis & John, 2017). Furthermore, it is crucial to utilise adhesive systems that do not possess solvent retention and releases carbon dioxide (Ling et al., 2010; Wolf, 2010). In this day and age, the use of an SF adhesive system is imperative as the world has become conscious of reducing the carbon footprint by utilising polymers that are recyclable, compostable, and reusable (Zarybnicka et al., 2015).

To achieve the objective of this chapter, further investigation was necessary to understand the effectiveness of the model and its impact on the overall profitability of the plant. In this regard, the current value stream (VS) was mapped to quantify process inefficiencies within the value chain while future VS was plotted to demonstrate the benefit and effectiveness of the model that was generated.

6.1.1 Preliminary test: Gage R&R

Preliminary tests were conducted before experimentation to ensure minimum variation during processing. Firstly, a Gage R&R study was performed to confirm that repeatability and reproducibility were examined. Moreover, an extensive robust process was developed to make sure that human intervention is minimised in the process. As demonstrated, this methodology was key to ensuring that the experimental error for all predicted output responses are minimised.

6.2 Effect of Surface Energy (SE) on the AS and TS

The SE, also known as corona treatment, has proved to be the most influential design parameter within this study. As demonstrated in Chapter 4, all completed runs that have an SE parameter operating at level 3 produced a very high response when compared with the run which had SE operating at level 1. These results were also validated by the total contribution calculated for each process parameter. Based on the analysis conducted, it is crucial that the SE is kept at 44 dyne/cm for PE and 50 dyne/cm for PA to ensure that a maximum AS and TS are achieved, respectively. Table 6.1 demonstrates the quantities of material and cost of material during experimentation. According to the data presented, the whole project cost R168 600.

Table 6.1: Utilised resources

Raw material and resources	Units	*Quantity*	*Cost*
PE film	55 Kg	1000	R55 000
PA film	55 Kg	1000`	R55 000
Hardener	55 Kg	100	R5 500
Resin	55 Kg	200	R11000
Machine Rates	1250 Rands/hr	30	R37 500-
Labour			
QA inspector	40/hr	3	R1 200
Operator	30/hr	3	R900
Process Engineer	100/hr	1	R3 000
Cost per kilogram	Rand	55	-
Total			R168 600

Figure 6.1 represents the current value stream map (CVSM), while Figure 6.2 further below demonstrates the future value stream map (FVSM) after experimentation. Figure 6.2 shows that the model that was developed was effective, since less waste was generated, and productivity was improved by 30%. The FVSM further demonstrated an improvement in the cycle time (C/T) as well as quality of the laminate produced.

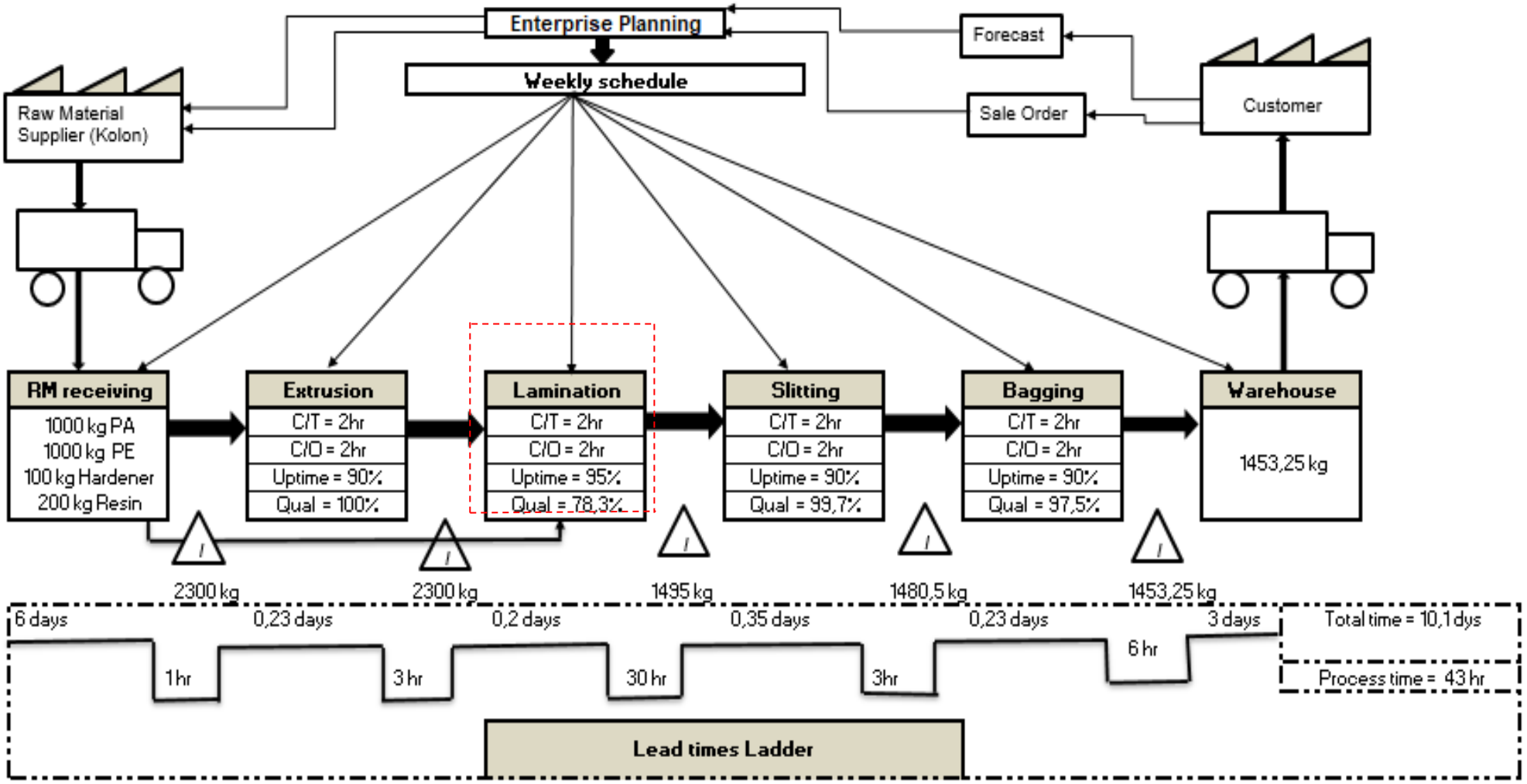


Figure 6.1: Current value stream map

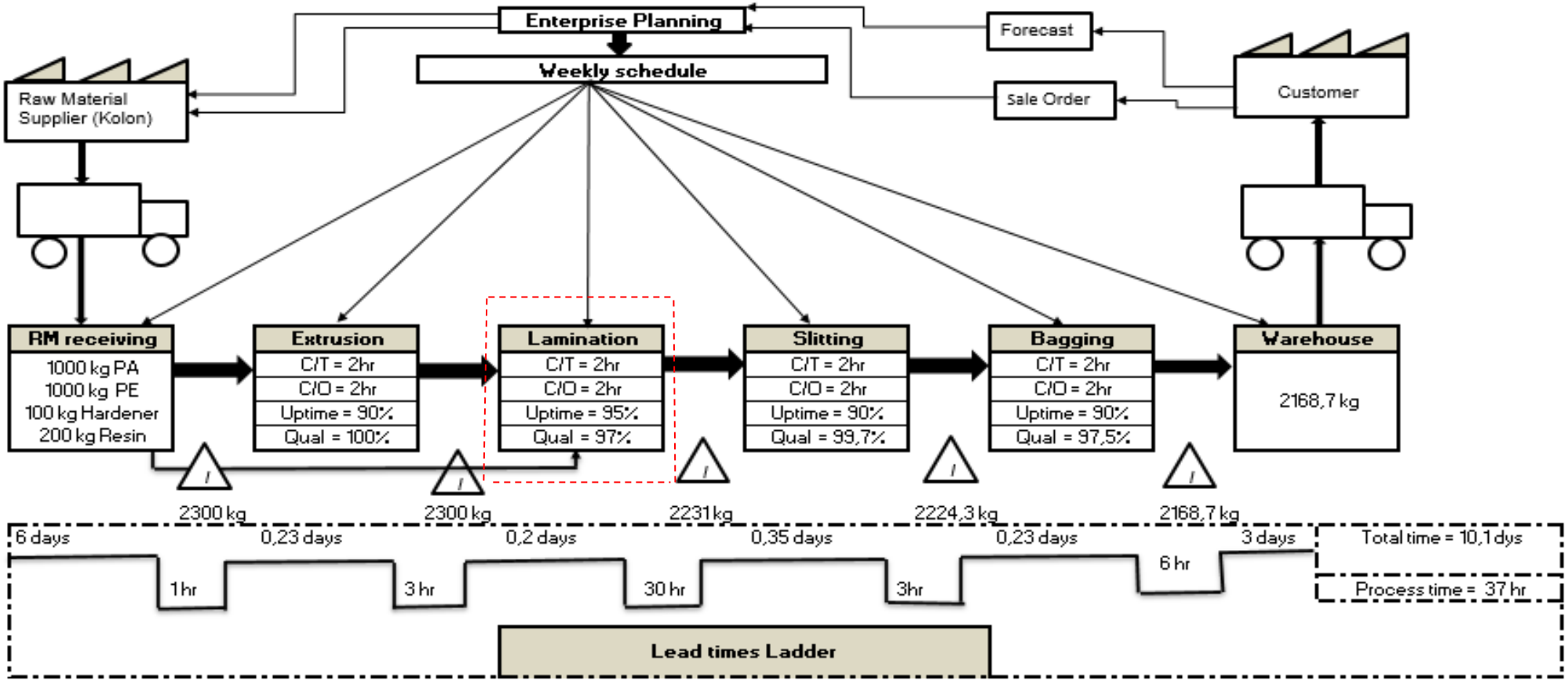


Figure 6.2: Future value stream map

6.3 Summary of Chapter

As detailed by the mass balance in Figure 6.1, a lot of material was utilised. Consequently, the process was not profitable compared to Figure 6.2 which demonstrated high output based on the optimum operating condition. In Figure 6.1, the input to the process was 2 300 kg of PE and PA with adhesive in between the two films. However, the output mass flowrate was 1 495 kg. As a result of poor accuracy and precision of process parameters, an amount of 805 kg was generated as a result of waste, achieving only 65% of the input. Based on the cost per kilo table above, an amount of R44 275 was the result of waste generated.

Based on the insights of the research so far, the following chapter concludes the study.

CHAPTER 7

CONCLUSION & RECOMMENDATIONS

Chapter 7: Conclusion and Recommendation

7.1 Introduction

Chapter 7 draws conclusions on the Taguchi based DOE in recovering laminate during SF laminating using OA. In this study, the effect of different process parameters involved in a SL PU system was investigated. These parameters included SE (dyn/cm), CW with a unit of measure of grams per square metre (gsm), MS which is a measure of (m/min), TT (%), RT (N), AV, MR, and CT (^{o}C). The research also detailed critical properties that need to be considered when choosing adhesive lamination for different polymer structures available for enhancing barrier properties. Different properties of SF, SB and WB adhesive systems were detailed in Chapter 4. A total of 18 experiments were completed following the TM OA. The mean effect plot and S/N graph were plotted. The effect of surface energy and adhesive distribution were evaluated.

There were two output responses that were studied in this project, namely: *adhesion strength* (AS) and *tensile strength* (TS). An amount of 640 N was achieved for AS with a S/N ratio of 56:12. The predicted output response for the same operating condition was 613.05 N. This shows a percentage error of 4.5% and 0.97% for S/N since the predicted S/N ratio was 55:57. All tests and trials were performed in an industrial SL laminating system. Prior to running the actual experiment, a Gage R&R study was performed in order to study all sources of variation that are related to instruments, personnel, and material used. This was done to ensure that there is uniform adhesive distribution, and that material variation is reduced. The experiment was performed to define the optimised operating condition and also generate a model that can be utilised to predict the behaviour of the system.

Based on the analysis conducted using ANOVA, SE was the most influential factor on the output with a contribution of 70.5%, followed by MS with 7.65%, and AV which consisted of a 5.89% contribution. To accomplish the above result, the optimum operating levels achieved were SE at 44 dyne/cm, TT at 35%, CT kept at 32 ^{o}C, CW at 1,5 gsm, MS at 150 m/min, AV which needs to be kept at 45 ^{o}C, MR which needs to be kept at 85 N, and RT at 100 N. After achieving all the mentioned parameters, a Taguchi QLF method validated the effectiveness of the model and quantified quality loss incurred on the process before optimising process parameters by the Taguchi methodology.

The normal distribution curve was plotted to assess how the parameters are distributed around the mean. The Cpk value was also identified using Minitab 17. The model was identified to be effective since process variables are well distributed around the mean.

In light of the findings of this study, the following recommendations are made for future research.

7.2 Recommendations for Future Research

Future studies should focus on:

- The effect of intermolecular forces between the PA and PE
- The effect of moisture on the resin and hardener
- The rheological behaviour of an adhesive system at molten state
- The polarity of web substrates during processing
- The hydrophilic and hydrophobic nature of polymers during the curing process

7.3 Final Conclusion

The main research question of this study was to investigate the effect of process parameters such as application weight, temperature, and film tension on the AS. The second question was to examine the effect of TM in reducing process variation in SF laminating system. The third question set out to examine the importance of Taguchi QLF in maintaining the AS target value. The first question was addressed through ANOVA (Table 4.9) which detailed the contribution of each process parameter involved in a SF lamination unit. Critical optimum parameters (Table B.5) were identified to enhance AS and reduce process variation using TM with the percentage error of 4.5% between the actual response and predicted response. Therefore, it can be deduced that TM is effective in eliminating variation. This result satisfies the second question since the target value of 640 N was obtained. After optimum parameters were identified, a model was created to understand the behaviour of the system and impact on the output. These results indicate that the third and fourth research questions were achieved with the established model. The CVSM and FVSM layout plotted in Chapter 6 (see Figures 6.1 and 6.2) were key in demonstrating the effect of TM and process modelling to reduce the plastic carbon footprint. These results satisfy the final key question of this study.

Having sufficiently answered the research questions and achieved the corresponding objectives indicated in Chapter 1, this study closes with an apt statement expressed by Pope John Paul II (n.d.):

Modern society will find no solution to the ecological problem unless it takes a serious look at its lifestyle.

This research seeks to do just that!

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APPENDICES

Appendices

Appendix A: Taguchi based design of experiment to achieve high adhesion strength and tensile speed

Table A.1: Gage R&R measurement data

Operator A	CW Measurement	Parts	Operator B	CW Measurement	Parts	Operator C	CW Measurement	Parts
A	1.67	1	B	2.10	1	C	2.13	1
A	1.8	2	B	2.10	2	C	1.93	2
A	1.63	3	B	2.13	3	C	1.9	3
A	2.47	1	B	1.83	1	C	1.97	1
A	1.97	2	B	1.83	2	C	1.97	2
A	2.53	3	B	1.87	3	C	2.03	3
A	2.3	1	B	2.17	1	C	2.2	1
A	2.2	2	B	1.97	2	C	2.37	2
A	2.2	3	B	1.93	3	C	2.23	3
A	2.3	1	B	1.93	1	C	1.83	1
A	2.3	2	B	2.1	2	C	1.97	2
A	2.23	3	B	1.87	3	C	2.6	3
A	2.00	1	B	2.13	1	C	2.43	1
A	2.00	2	B	2.2	2	C	2.03	2
A	2.2	3	B	2.00	3	C	1.90	3

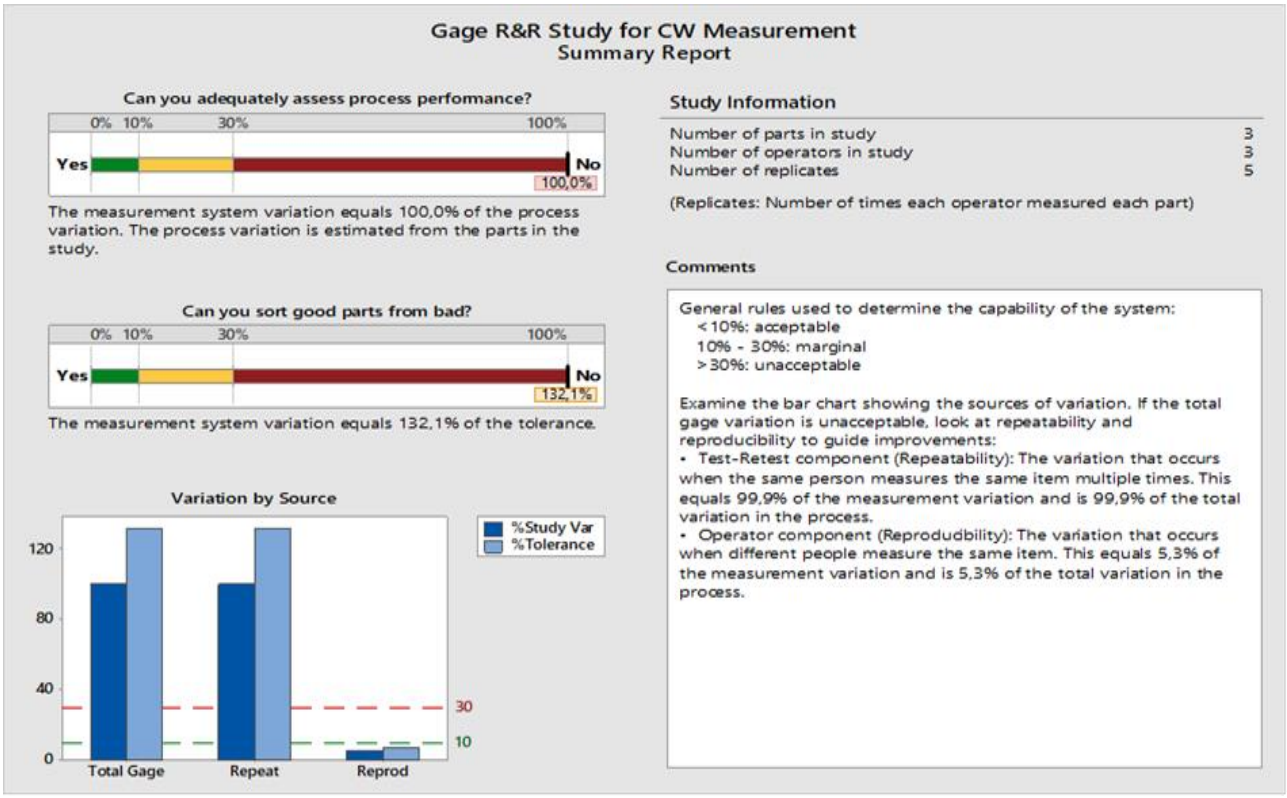

Figure A.1: Gage R&R summary

Table A.2: Dial gage measurement

Dial gage indicator result

Operator A	CW Recorded	Parts	Operator B	CW Recorded	Parts	Operator C	CW Recorded	Parts
A	2.00	1	B	2.00	1	C	2.00	1
A	2.00	2	B	2.00	2	C	2.00	2
A	2.00	3	B	2.00	3	C	2.00	3
A	2.00	1	B	2.00	1	C	2.00	1
A	2.00	2	B	2.00	2	C	2.00	2
A	2.00	3	B	2.00	3	C	2.00	3
A	2.00	1	B	2.00	1	C	2.00	1
A	2.00	2	B	2.00	2	C	2.00	2
A	2.00	3	B	2.00	3	C	2.00	3
A	2.00	1	B	2.00	1	C	2.00	1
A	2.00	2	B	2.00	2	C	2.00	2
A	2.00	3	B	2.00	3	C	2.00	3
A	2.00	1	B	2.00	1	C	2.00	1
A	2.00	2	B	2.00	2	C	2.00	2
A	2.00	3	B	2.00	3	C	2.00	3

Table A.3: Two-way ANOVA without interaction

Source	DF	SS	MS	F	P
Parts	2	0.01916	0.00958	0.19825	0.821
Operator A	2	0.10079	0.05o395	1.04284	0.362
Repeatability	40	1.88133	0.048247		
Total	44	2.05294			

Table A.4: Standard variance

		Study Var	% study Var
Source	Stdv (SD)	(6 x SD)	(%SV)
Total Gage R&R	0.220142	1.32085	100
Repeatability	0.219829	1.31897	99.86
Reproducibility	0.011748	0.07o49	5.34
Operator A	0.011748	0.07049	5.34
Part-To-Part	0.000000	0.00000	0.00
Total variation	0.22o142	1.32085	100.00

Table A.5: Gage R&R Parameter contribution variation and sources

Source	Var comp	% contribution
Total Gage R&R	0.000000	0.000000
Repeatability	0.000000	0.000000
Reproducibility	0.000000	0.00000
Operator A	0.000000	0.000000
Part-To-Part	0.000000	0.000000
Total variation	0.000000	0.000000

Table A.6 : L_{18} Orthogonal Array (OA) experimental set-up with Process Parameters at different levels

Runs	Columns							
	CT	RT	TT	SE	CW	MS	AV	MR
1	1	1	1	1	1	1	1	1
2	1	1	2	2	2	2	2	2
3	1	1	3	3	3	3	3	3
4	1	2	1	1	2	2	3	3
5	1	2	2	2	3	3	1	1
6	1	2	3	3	1	1	2	2
7	1	3	1	2	1	3	2	3
8	1	3	2	3	2	1	3	1
9	1	3	3	1	3	2	1	2
10	2	1	1	3	3	2	2	1
11	2	1	2	1	1	3	3	2
12	2	1	3	2	2	1	1	3
13	2	2	1	2	3	1	3	1
14	2	2	2	3	1	2	1	2
15	2	2	3	1	2	3	2	3
16	2	3	1	3	2	3	1	1
17	2	3	2	1	3	1	2	2
18	2	3	3	2	1	2	3	3

Appendix B: Linear model analysis and quantifying the most significant process parameters within the SF lamination

Table B.1: Response Table for means

Level	CT	RT	TT	SE	CW	MS	AV	MR
1	440.6	417.5	455.8	333.3	463.3	467.5	450.8	445.8
2	447.8	466.7	461.7	455.8	452.5	460.8	470.8	455.0
3	-	448.3	415.0	543.3	416.7	404.2	410.8	431.7
Delta	7.2	49.2	46.7	210.0	46.7	46.7	60.0	23.3
Rank	8	4	5	1	6	2	3	7

Linear Model Analysis: S/N ratios versus CT. RT. TT. SE. CW. MS. AV. MR

Table B.2: Estimated Model Coefficients for SN ratios

Term	Coef	SE Coef	T	P
Constant	52.7139	0.2755	191.349	0.00
CT 28	-0.0642	0.2755	-0.233	0.837
RT 80	-0.5071	0.3896	-1.302	0.323
RT 100	0.3814	0.3896	0.979	0.431
TT 15	0.3167	0.3896	0.813	0.502
TT 25	0.3564	0.3896	0.915	0.457
SE 40	-2.3025	0.3896	-5.910	0.027
SE 42	0.4568	0.3896	1.172	0.362
CW 1,5	0.3016	0.3896	0.774	0.520
CW 2,0	0.1942	0.3896	0.499	0.668
MS 150	0.4935	0.3896	1.267	0.333
MS 180	0.3249	0.3896	0.834	0.492
AV 35	0.1240	0.3896	0.318	0.780
AV 45	0.4813	0.3896	1.235	0.342
MR 75	0.0514	0.3896	0.132	0.907
MR 85	0.0934	0.3896	0.240	0.832

S = 1.169 R-Sq = 96.4% R-Sq (adj) = 69.1%

Linear Model Analysis: Means versus CT. RT. TT. SE. CW. MS. AV. MR

Table B.3: Estimated Model Coefficients for Means

TERM	COEF	SE COEF	T	P
CONSTANT	444.167	13.53	32.838	0.001
CT 28	-3.611	13.53	-0.267	0.814
RT 8O	-26.667	19.13	-1.394	0.298
RT 100	22.500	19.13	1.176	0.361
TT 15	11.667	19.13	0.61o	0.6o4
TT 25	17.500	19.13	0.915	0.457
SE 4O	-110.833	19.13	-5.794	0.029
SE 42	11.667	19.13	0.61o	0.6o4
CW 1,5	19.167	19.13	1.002	0.422
CW 2.0	8.333	1913	0.436	0.7o6
MS 150	23.333	19.13	1.22o	0.347
MS 180	16.667	19.13	0.871	0.475
AV 35	6.667	19.13	0.394	0.761
AV 45	26.667	19.13	1.394	0.296
MR 75	1.667	19.13	0.087	0.939
MR 85	10.833	19.13	0.566	0.628

S = 57.39 R-Sq = 96.5% R-Sq (adj) = 70.5%

Table B.4: Analysis of Variance for SN ratios

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value	% Contribution
CT	1	0.0743	0.0743	0.0743	0.07	0.837	0.0989
RT	2	2.5105	2.51o5	1.2552	0.92	0.521	3.341
TT	2	4.0824	4.0824	2.0412	1.49	0.401	5.433
SE	2	53.4991	53.4991	26.7496	19.58	0.049	71.2045
CW	2	2.2477	2.2477	1.1238	0.82	0.549	2.991
MS	2	6.1136	6.1136	3.0568	2.24	0.309	8.136
AV	2	3.6800	3.6800	1.8400	1.35	0.426	4.897
MR	2	0.1947	0.1947	0.0974	0.07	0.933	0.25
Residual Error	2	2.7321	2.7321	1.3661			
Total	17	75.1344					

Table B.5: Model coefficients

Term	Coef	SE Coef	T-Value	P-Value	P-Value
Constant	-1388	442	-3.14	0.012	0.005
CT	1.81	6.46	0.28	0.786	0.786
RT	0.771	0.792	0.97	0.356	0.356
TT	-2.04	1.58	-1.29	0.229	0.229
SE	52.5o	7.92	6.63	0.00	0.00
CW	-46.7	31.7	-1.47	0.175	0.175
MS	-1.184	0.29	-1.88	0.093	0.093
AV	-2.00	1.58	-1.26	0.238	0.238
MR	-0.71	1.58	-0.45	0.665	0.665

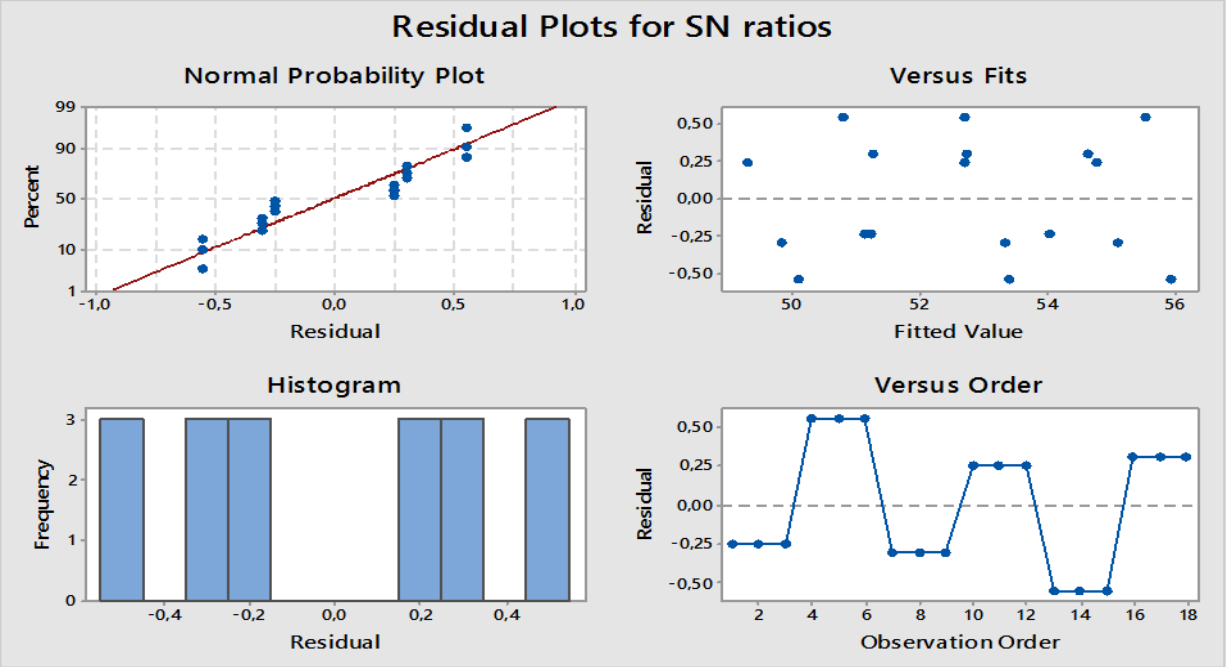

Figure B.3: Residual Plots for ratio

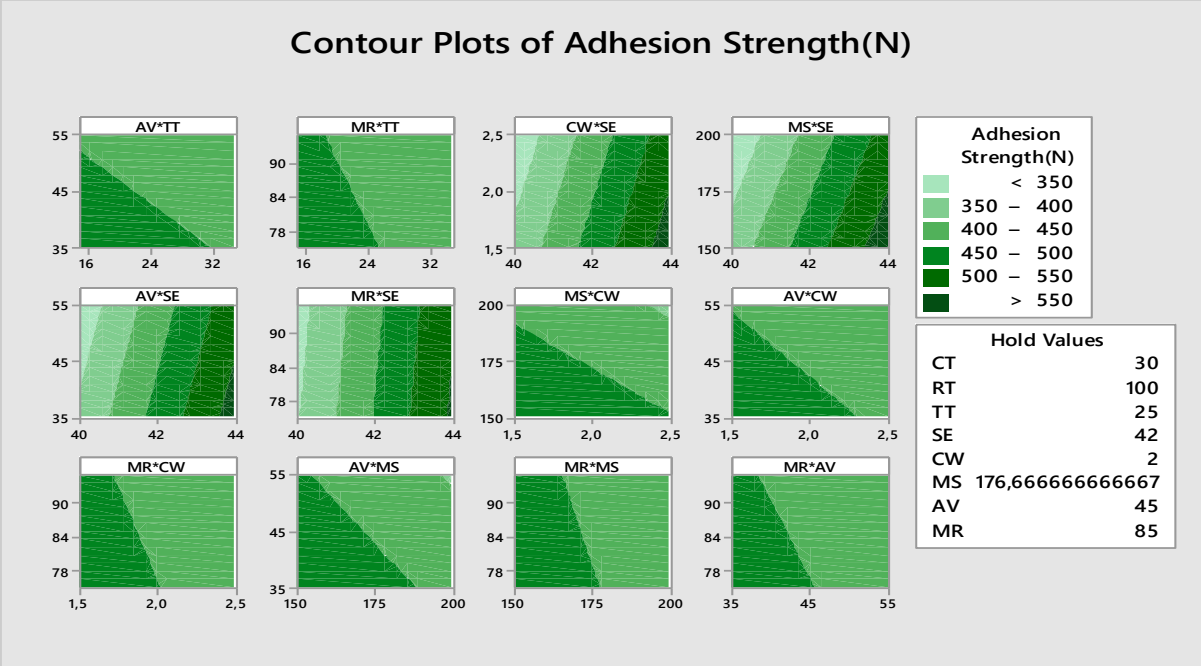

Figure B.4: Contour Plots of Adhesion Strength

Table B.5: Optimum operating condition

SE	TT	CT	CW	MS	AV	MR	RT
44	35	32	1,5	150	45	85	100

Appendix C: Parameters to achieve output and the effect of noise factors

C.1: Signal to Noise (S/N) ratio

To calculate the signal to noise ratio, a large the better approach was chosen since the aim of the study was to yield a higher output response on adhesion strength (AS) and tensile speed (TS).Therefore the below formula was used to calculate the S/N ratio for AS and TS.

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right)$$

C.2: Quality loss function (QLF) by Taguchi

Optimum AS = 640 N ,LCL_{AS} = 640 N - 2 ,UCL_{AS} = 640 N + 2

Optimum TS = 21.02 mm/s ,LCL_{ts}= 21.02 mm/s - 2, UCL_{ts} = 21.02 mm/s + 2

$L(y) = k (y – m)^2$

$(55) = k (2)^2$

K = 13.75

Y = actual output value, m = target

$L(y) = 13.75 (y – m)^2$

C.3: Parameter contribution to the output response (AS)

SS: sum of squares

Total SS for all process parameters = 190062

To calculate the contribution of every parameter to the output response, the following formula was used:

$$\text{Parameter contribution (\%)} = \frac{\text{Seq } SS}{\text{Total SS}}$$

For SE parameter: Seq SS = 13533

SE parameter contribution (%) = $\frac{13533}{190062}$ X100 = 71.2%

C.4: Process Capabilities after experimentation

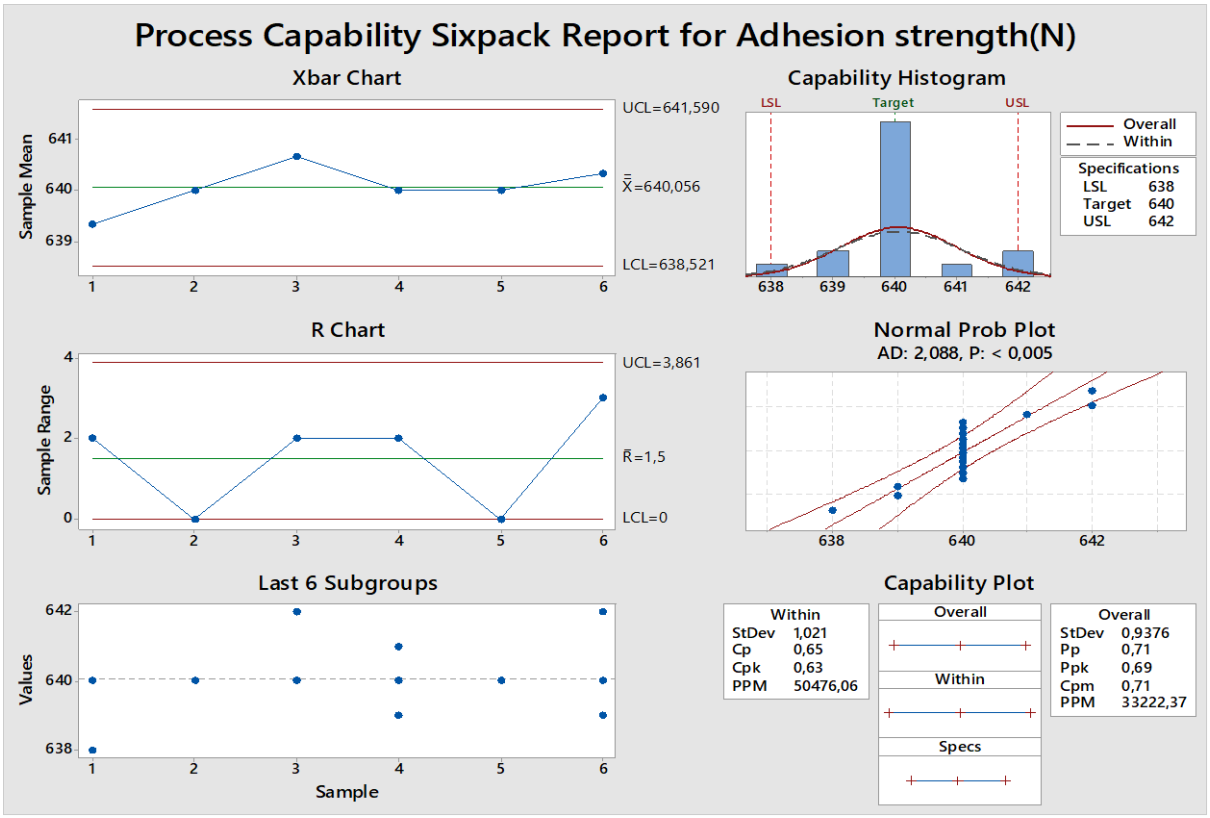

Figure C.3: Process capability study