AN ENVIRONMENTALLY SOUND GOLD RECOVERY PROCESS FOR SMALL-SCALE GOLD MINING

by

WENDY BOUWER (nee KOTZE)

A dissertation submitted in fulfilment of the requirements for the Masters Degree in Chemical Engineering Technology at the Cape Technikon

Supervisor: Prof. Francis W Petersen

Cape Technikon February 1999

"For the Lord gives wisdom; from His mouth come understanding"

Proverbs 2:6

DECLARATION

I hereby declare that the work contained in this thesis is my own original work, except for the assistance provided by certain persons, as acknowledged in the text.

Wendy Bouwer February 1999

ACKNOWLEDGEMENTS

The work contained in this thesis was performed at the Cape Technikon's School of Mechanical and Process Engineering in the Department of Chemical Engineering and commenced February 1997.

I am grateful to the following persons and/or institutions for their assistance and contributions in completion of this thesis:

- The School of Mechanical and Process Engineering, at the Cape Technikon for the use of their laboratory facilities and instrumentation.
- MINTEK for their financial support and continuous interest in the progress of my work. The helpfulness of the staff at times when I was in need of samples and/or the performance of analyses, will always be greatly appreciated.
- The Foundation of Research and Development (FRD) for their financial contributions.
- Mr van der Heyden, senior engineer at Caltex head office in Cape Town, for his assistance in obtaining the Caltex oil samples.
- Mr van der Hoven at the Athlone Powerstation, for hi assistance in obtaining the coal \overline{a} sample, used during all experiments.
- My supervisor, Prof. Francis Petersen for his support, guidance and continued $\overline{}$ encouragement during the course of my studies. His enthusiastic and diligent approach to life, as well as his dedication and loyalty towards his students will always be appreciated.
- The staff and fellow students at the Cape Technikon for their sincere friendship and consideration when I needed assistance. A special word of thanks to Paul van der Plas, for his unconditional assistance with the instrumental analysis on the Graphite Tube Atomiser (GTA), his helpfulness will always be greatly appreciated. A further word of thanks to Lucian Moses for his dedicated assistance during the performance of some of the final experiments.
- My parents (and sisters) for their faith in me. I sincerely appreciate their love, prayers, encouragement, emotional and financial support throughout my student life. Thank you for always being appreciative of my hard work.
- My husband, Evert, for his love and for always being understanding and supportive of my career. Thank you for always having faith in me and being there when I needed you most.
- To God be the glory!

ABSTRACT

The gold mining industry has mainly relied upon the use of a highly polluting chemicals, such as mercury and cyanide, to recover gold from its ores. As environmental legislation has become more stringent in all countries and environmental protection has become the focus of world-wide research, development of environmental sound processes has been favoured. The Coal Gold Agglomeration (CGA) process is such a process which was developed some years ago and has the advantage in that gold is recovered by a procedure which has little or no effect on the environment. The CGA process is based on the hydrophobic characteristics of coal, gold and oil. Gold particles which are substantially free become attached to the coal-oil agglomerates during collision, and eventually penetrate into the agglomerates. The resulting agglomerates are recycled to increase the gold loading, separated from the slurry, burnt. ashed and smelted to produce gold bullion.

Laboratory scale batch tests were performed on an artificial/synthetic gold ore, containing fine gold powder. The slurry was contacted with a mixture of coal and oil. i.e. coal-oil agglomerates, after which both the agglomerates and ore were analysed for gold. Operating parameters, such as the mode of contact between the coal-oil phase and the gold containing slurry, contact time of the slurry and the coal-oil phase. means of separating the coal-oil gold agglomerates from the slurry, coal to ore. coal to oil and water to ore ratios, type of oil. effect of collectors and the mineralogy of the ore on the gold recovery were investigated.

Results have shown that stirring the coal-oil phase and the slurty yielded higher gold loadings than shaking and the traditional rolling bottle technique. By increasing the time of contact between the coal-oil phase and the gold slurry, the final gold loading in the agglomerates increases, until an equilibrium value is reached. An increase in the amount of coal, together with a decrease in the amount of water used in the slurry, has shown to increase gold recoveries. Furthermore, by varying the concentration and volume of a collector. such as potassium amyl xanthate (PAX) enhanced the settling rate and enabled the effectiveness of separation. Moreover, it was found that the gold loading on the coal-oil phase increased after recycling it. Further tests were performed on a real ore sample and after X-ray Diffraction (XRD) analysis, it was found that certain minerals other than gold was transferred to the coaloil phase.

The theoretical foundation of the CGA process is based on the difference in free energy and was expressed as a function of the interfacial tensions and three-phase contact angles between gold, oil and water, together with the ratio of coal-oil agglomerate to gold particle radii. As the free energy is a measure of the thermodynamic stability and hence, partly a measure of gold recoveries, meaningful predictions as to gold recoveries were made by performing a sensitivity analysis on the variables connected to the free energy, It was, however, found that some operating parameters, which were linked to other factors, such as the maximum gold transfer into coal-oil phase and the separation efficiency of the agglomerates. were vital to be taken into account when predictions as to gold recoveries were made. Therefore, the gold recoveries were found to be a function of the thermodynamic stability as well as the maximum gold transfer into the coal-oil phase and the separation efficiency of the agglomerates, The meaningful information gained by performing the theoretical investigations were applied and linked to gold recoveries, thereby providing useful explanations as to the typical gold recoveries obtained during experimentation.

A comparative study on mercury amalgamation was done to evaluate the performance of the CGA process. It was found that the CGA process yielded better gold recoveries than amalgamation, which makes it the better process both in terms of recoveries as well as environmental safety, A further application of the theoretical knowledge was, however, very useful to explain the tendency of the CGA process yielding the better results,

CONTENTS ________________________ Page

LIST OF FIGURES Page

xiv

LIST OF TABLES Page

CHAPTER 1

INTRODUCTION

1.1 GOLD

1.1.1 Background

Gold, a well-known precious metal, has a history dating back to antiquity, where gold bits were found in Spanish caves that were used by Paleolithic people around 40,000 BC The yellow metal was named the "child of Zues" by the Greek poet Pindar and called *aurum* ("shining dawn") by the Romans. Furthermore, gold is the first element and the first metal to be mentioned in the Bible, where it appears in more than 400 references (Yannopoulos, 1991).

Gold accounts for approximately 0.000 000 5% of the earth's crust substance (a concentration of about 0.005 g/t) and occurs in ocean water as dissolved gold in a concentration of 0.01 to 0.05 mg/m³ (Hentschel and Priester, 1° 92a).

The metal is characterised by certain physical properties, which include the following:

- gold metal has a bright yellow colour,
- conducts heat and electricity,
- is nonmagnetic,
- has a high ductility (can be drawn into a thin wire without breaking),
- has a high malleability (where one ounce [31,3 g] can be beaten into a sheet of 9 m^2),
- is a very soft metal, rating 2 on Moh's scale,
- and has a melting point of 1 063 \degree C and a boiling point of 2 660 \degree C,
- is a very heavy metal, having a specific gravity ranging from 15 -19 g/cm³, depending on the silver content and declines with increasing silver content.

Among the chemical characteristics it is said that gold, in its natural form, is not attacked by either sulphur or oxygen in the presence of air or water and is resistant to weathering, as it tends to accumulate in secondary deposits (refer to section 1.1.2). However, the metal dissolves in the presence of halogens or complexing agents such as cyanide solutions as well as strong oxidising agents. such as *aqua regia.* Finally, gold has a good alloyability with other precious metals, such as silver and mercury (Hentschel and Priester, 1992a; Hacker. 1872).

The major importance of gold is as a store of wealth. where half of the gold ever produced throughout the world is either kept safe in government treasuries and central banks or in the hands of individuals. As gold is very soft and resistant to corrosion, it is often used in dentistry. Jewellery is another major use of gold, whereby the pure metal is alloyed with other metals, usually copper, to increase the hardness, as the pure metal is to soft. The quality gold alloy is expressed in "carats", where pure gold is "24 carat gold". Other standard alloys such as 22-, 18-, 15-, 12-,9-, denote the parts of gold in the alloy. eg. 22 parts of gold to 2 parts of other metal (Internet: reference no 51).

1.1.2 Natural occurrence of gold

Natural gold mainly occurs in two types of deposits, which are classified as primary and secondary deposits. Primary deposits are called gold-quarts lodes or veins and are ore bodies that may be of platonic or subvolcanic origin embedded in plutonites, vulcanites. metamorphites or sediments as country rock and enriched veins may form by mechanical or chemical action in cap zones, oxidation zones or cementation zones (Hentschel and Priester, I992a).

Secondary ore deposits, often called placers, are ore bodies formed by the breakdown of rock outcrops that were exposed to the surface of the earth and where subjected to the natural elements of weathering and erosion, however causing the eventual breakdown of the rock into fragments which are carried away by wind, water and ice. The redeposition of the fragments takes place in river systems, lakes or in the sea where the heavier, more durable gold is then concentrated into rich deposits. Placers are devided into two types. namely recent and fossil placers, where recent placers are of more recent origin and display all gradations between loose, slightly consolidated and consolidated sediments and the fossil placers yield extremely hard ore with processing properties very similar to that of a primary ore body. An example of a recent placer is the gold deposits of the Witwatersrand in South Africa (Hentschel and Priester., 1992a; Internet: reference no. 51). Further placer ores exist, such as alluvial deposits, whereby the gold/gold bearing rock has been transported short distances from their source (generally by gravity) and have been concentrated within the soil horizon. Alluvial deposits are formed by the concentration of gold particles within streams systems, under the action of running water and beach placers, where gold is concentrated in the beach sands by wave action, is a type of alluvial deposit. Leads are former stream courses, containing gold, where barren sands have covered the original passage of the stream and deep leads are gold deposits in former stream beds which have been covered with basaltic lava. Nuggets are formed, either as rich fragments of primary deposits, which have been transported and deposited in a sedimentary environment, or as chemical accretion of small gold particles into larger fragments. Some nuggets may have formed through the chemical action of host soils or sediments on a gold solution.

The gold content of primary deposits can range from $5-100$ g/t compared to that of a secondary deposit, which normally ranges from 0.1 -5 g/t, where the lower limits are taken as the extraction economical thresholds for the consecutive ore bodies. To obtain the maximum geological gold content of a primary ore deposit, it would necessitate natural enrichment processes reaching a factor of 1000 in order to sufficiently concentrate statistically distributed gold contents into profitable gold deposits.

Placer gold is mainly free gold due to the removal of the other minerals in the process of translocation by reason of their solubility, cleavage or weathering behaviour. In primary ore deposits the gold is normally scattered irregular throughout the quartzose matrix, and the gold and/or quarts may be intergrown with accompanying minerals. The most significant accompanying minerals in the unweathered veins include the following: quarts, feldspar, calcite and siderite as gungue material and pyrite, chalcopyrite, marcasite, galena, stibnite, free silver, arsenopyrite, pyrrhotite, molybdenite, sphalerite and bismuth minerals. The mineral composition of placers. however is much more complicated as many other minerals are "dragged along" during translocation.

Much literature exists whereby gold bearing ores are classified under certain headings, e.g. as by Henley (1975) the gold ores are classified into seyen broad groups according to the geological environments in which they are to be found e.g. gold-quarts lodes, epithermal deposits, young placers, fossil placers. deposits with desseminated gold. gold in non-ferrous metal ores and gold in sea water. McQuiston and Shoemaker (1975) proposed the following extraction orientated classification of gold ores

- *Native gold ores*, in which the gold can be removed by gravity separation. amalgamation and/or cyanidation.
- *Gold associated with sulfides.* occurring either as free particles or disseminated in sulphides. Auriferous pyrite with gold finely disseminated in its matrix is rather common.
- *• Gold tellurides,* which usually occur along with native gold and sulphides.

• *Gold in other minerals*, as with arsenic and/or antimony (e.g. aurostibnite, AuSb₂), with copper porphyries (as selenide and telluride), with lead and zinc minerals, and with carbonaceous minerals.

Regardless the type/name of classification, it is seen that gold metal exists in nature in various locations, which is an indication of the type of mineralogy of the ore body hosting the precious metal. Furthermore, it is found that gold metal exists either in its liberated/free form or in association with other minerals, which is determined by the type of deposit from which it originates.

1.2 **EXTRACTING GOLD FROM ITS ORES**

Since the discovery of gold, various means have been devised and tested to separate the precious metal from the ores containing it. The type of process utilised to extract gold from the ore body is vastly determined by factors such as: the mineralogy of the ore and the degree of liberation of gold particles. The raw ore body is crushed and ground in the attempt to liberate/free as much gold, which could be separated either by a chemical. physical or surface based process. It will be attempted to identify and provide a brief discussion of the main processes in use.

Processes such as cyanidation (chemical process) and mercury amalgamation (based on the surface chemisty of gold) is dealt with in this section.

1.2.1 Cyanidation

The cyanidation process was patented by JS MacArthur and his co-workers RW Forrest and W Forrest in 1887 and involves the production of geld and/or silver by leaching auriferous and/or argentiferous ores with a weak cyanide solution.

Figure 1.1 is a general flowchart of the processing steps required to extract gold from its ores by utilising the cyanide process and consists of five basic operations:

- I. The comminution circuit
- 2. The cyanide leach circuit
- 3. The solids-liquid separation area and/or a system of gold recovery from solution
- 4. The gold melting and refining area
- 5. The tailings disposal area

In the first step the ore is comminuted by means of crushers and mills to render it more useable by reduction of its particle size and te liberate the one component from another to permit subsequent separation of the valuable component (e.g. gold) from the gangue (Stanley, 1987). In order to enhance gold extraction by cyanidation, the gold grains (or micrograins in some ores) must be fully wetted by the cyanide so that the solid-liquid reaction of leaching takes place, therefore fine grinding of the ore liberates the gold particles and maximises the reaction rate and efficiency of leaching. An ore yielding acceptable gold recoveries (more than 88%) by direct leaching after being ground normally (60-75% -200 mesh). is defined as an *amenah:e* ore. whereas a *refractory* ore yields recoveries below 80% if normally ground and needs extremely fine grinding or pre-treatment before being exposed to cyanide (Yannopoulos, 1991).

The second step is the leaching circuit, where the gold ore is subjected to a leaching agent such as sodium or calcium cyanide, which dissolves the gold (or silver). Various theories have been proposed to explain the mechanism of gold and silver dissolution in aqueous cyanide solution (Habashi, 1987; Cornejo and Spottiswood, 1984) from which the overall reaction was derived and confirmed by Finkelstein (1972) as follows:

$$
2Au + 4NaCN + O_2 + 2H_2O = 2NaAu(CN)_2 + 2NaOH + H_2O_2
$$
...(1.1)

The other essentials for the reaction is oxygen, mainly to stabilise the cyanide radical, and calcium hydroxide, which maintains a protective level of alkalinity (pH 10 to **11**) (Young, 1987). Many types of leaching operations, such as heap leaching, vat leaching, batch and continuous leaching or a combination of both etc. have been researched and are **in** use (Young, 1987 and Yannopoulos, 1991). ¹¹ is not within the scope of this study to investigate the different means of leaching the gold ore, but rather to gain a understanding of the basic concepts concerning the cyanidation process as the leaching circuit is part of the basic operation of the cyanidation process and will vary for each gold operating plant.

Once the ore has been ground and leached with the weak cyanide solution, the pregnant solution contains anionic gold cyanide complexes (Equation 1.1), from which the gold must be recovered. One (or a combination) σ^2 the following processes is utilised to recover the gold from the pregnant solution (see Figure 1.1):

- Zinc cementation
- Activated carbon adsorption
- Ion-Exchange resin extraction

Continued research is being done and therefore, the abovementioned processes are not the only ones existing, but rather are the most widely used, and will hence be discussed briefly **in** the following sections. As the chemistry of these processes are very complex and can be treated here only in the barest outline, the reader is referred to works devoted to these subjects for further details, such as Helferrich (1962), Marcus and Kertes (1969) and Nicol *et al.,* 1987.

1.2.11 *Zinc Cementation*

The recovery of gold by zinc is based on the electrochemical order of the metals **in** cyanide solution, which dictates their solubilities in the solvent. The electrochemical order of metals in potassium cyanide solution are in the following sequence. from positive to negative: Mg, AI, Zn, Cu, Au, Ag, Hg, Pb, Fe, Pt. It is said that any metal in this sequence would tend to dissolve in cyanide solution more readily than the metal to its right, which explains why zinc displaces gold in a cyanide solution (Yannopoulos, 1991). The overall chemical reaction for cementation was proposed by Barin *et at.* (1980) and is as follows (Yannopoulos, 1991):

$$
Zn + Au(CN)_2 + H_2O + 2CN^- \rightarrow Au + Zn(CN)_4^2 + OH^- + 0.5H_2
$$
...(1.2)

Zinc cementation is classified as a *Solids-Liquid separation* (see Figure 1.1) system and involves the separation of the pregnant solution from the leached solids before recovery via Zinc precipitation (cementation). It is vital that the solids be washed thoroughly to minimize any loss of dissolved gold with the solids, therefore a *counter-current decantation (CCD)* series of thickeners is designed to provide solids-liquid separation with thorough washing of the solids. The solids are washed as they flow from the first to the last thickener and the liquid washings overflow and are pumped from the last to the first thickener, counter to the movement of the solids.

The *Merrill-Crowe zinc cementation process*, which is the next process step. was developed in the United States and consists of four basic steps (Yannopoulos. 1991: Bosley, 1987):

- I. Clarification of the pregnant cyanide, as fine solids in suspension may coat the zinc surfaces and reduce its activity.
- 2. De-aeration. as oxygen causes increased zinc consumption by wasteful side reactions oxidising the zinc (Crowe, 1918).
- 3. Addition of zinc powder and lead salts, as the formation of a so-called zinc-lead couple simulates the poles of a voltaic cell and hydrogen can be evolved at the lead cathode, thereby reducing the passivation of the zinc surface.
- 4. Recovery of the zinc-gold precipitate. Zinc dust is added to the de-oxygenated solution as it flows through precipitation filters, which are precoated with

diatomaceous earth and a secondary coating of zinc dust. The filters are totally enclosed to safeguard the precious precipitate.

[In a few instances, the gold solutions that are eluted from the activated carbon are treated with zinc cementation in stead of electrowinning. It was found that such solutions do not have to be de-aerated, since their oxygen level is very low due to prior hot elution and depressurizing (refer to Figure 1.1).

1.2.1.2 *Adsorbent-in-pulp systems*

Activated Carbon Adsorption

Activatea carbon is a highly porous material with a very large intraparticulate area per unit of mass, which gives it distinctive adsorptive properties. The recovery of gold from pregnant solutions by activated carbon consists of three distinct operations, refer to Figure 1.2 (Yannopoulos, 1991):

- I. Loading: the adsorption of gold (and/or silver) from solution onto the carbon.
- 2. Elution or stripping: desorption of gold (and/or silver) from the carbon. The gold cyanide complex is adsorbed onto activated carbon and is recovered from the dilute production solutions where it is delivered as concentrated gold solutions and not as gold metal, therefore the gold is merely desorbed into a more cuncentrated solution.
- 3. Metallic gold production: Electrowinning or zinc cementation of gold from the concentrated (eluate) solution.

More than one system exists whereby activated carbon is loaded and is described by the means of contacting the carbon with the slurry (Figure 1.2). for example, when the activated carbon is mixed with the leached slurry to adsorb the gold from solution (in separate tanks), it is named a carbon-in- pulp (CIP) system. When the activated carbon is added to the leaching tanks to adsorb the gold from solution as cyanidation proceeds, it is named a carbon-in-leach (CIL) circuit and the carbon-in-column (CIC) circuit involves the use of a packed column of activated carbon through which the clarified solutions is passed whereby the gold is adsorbed as they percolate. The CIP process is mostly used and preferred means of loading the activated carbon and has always been an economical and effective method for large scale gold and silver metal recovery.

Although activated carbon is an excellent adsorbent for soluble gold, it does not relinquish the adsorbed gold easily. Some of the elution/stripping techniques most commonly used is The Zadra method, whereby a warm sodium cyanide and sodium hydroxide solution is passed through an elution column and one or more electrowinning cells in series (Zadra, 1950) and The AARL (Anglo American Research Laboratory) method, which involves the preconditioning of the loaded carbon with a hot cyanide solution, followed by the elution, using deionised water (Davidson, 1986). All the elution operations, however, are based on mass transfer of the soluble gold compound, which is generated by a favourable concentration gradient and high temperatures (Yannopoulos, 1991).

The carbon used in the CIP circuit is regenerated (before being recycled to the last stage of the adsorption circuit) in order to get rid of the other adsorbed metals and organic contaminants (oils, greases, humic and fulmic acids). which causes carbon fouling and hence, reduces the activity and ability to adsorb. (Van der Merwe, 1991).

During the electrowinning procedure, gold metal is recovered by an electrolytic process, whereby the gold deposits onto the cathode. The laden gold cathodes may contain 70- 75% gold and 10-15% silver, the remainder being copper and steel wool. To fire-refine the electrowon gold, a pan is filled with 30% sodium nitrate (reacts to lower the melting temperature of the mix), 40% borax (dissolves the metal oxides and lowers the melting temperature) and 30% silica (combines with borax and contributes to the dissolution of metal oxides), all in percentage of cathode weight.

The charge $(1:1 \text{ cathode/flux})$ is placed in a silicon-carbide crucible in a tilting gas-fired furnace and heated to 1 093 $^{\circ}$ C. After melting, the mixture remains in the crucibles for about 1,5 hours after which it divides into two molten phases, bullion and slag. The slag is then skimmed off and the bullion further refined in clay crucibles (Yannopoulos, 1991).

Jon- Exchange Resin

Extraction of gold cyanide by means of ion-exchange resin is accomplished by the inert polymeric hydrocarbon matrix (usually polystyrene cross-linked with divinyl benzene) to which the functional groups are chemically attached. The three-dimensional network of the polymer matix absorbs water and swells when in contact with an aqueous solution, thereby allowing ions present in the aqueous solution to diffuse through the resulting gel to the sites of the active groups. The active groups can be anionic or cationic in nature and the interaction of with these groups and the cations or anions in solution is mostly coloumbic or electrostatic in nature. Therefore, the rate of the exchange reactions are high, reversible, and generally difussion-controlled and the selectivity of one ion over another is predominantly a function of the size, charge. and polyrizability to the ions (Yannopoulos, 1991; Nicol *et al.,* 1987». The chemistry involved in the ion-exchange technology is very complicated, as many different resins are available for use and will not be discussed here. Further reading could, however be done: Helferrich, 1962; Marcus and Kertes, 1969 and Nicol *et al., 1987.*

A typical resin-in-pulp (RIP) flowsheet consists of a milling section, followed by classification, whereby the classifier overflow is thickened. and the thickener overflow is leached with cyanide. The partially leached pulp is then contacted with ion-exchange resin in a resin-in-pulp operation. The resin is eluted at low temperatures and pressures after which it is then recycled back to the leaching opera'ion. The gold is then precipitated from the eluate, cast into anodes, and purified electrolytically. The problem with the elution of the resin is that the stripping is generally not complete, therefore the resin has to be incinerated after a few cycles to reclaim the contained gold values, which makes the RIP process unattractive due to the high cost of resins.

1.2.2 Mercury amalgamation

Amalgamation is a process applied to precious metal ores, where the precious metals (such as gold and silver) form alloys with mercury and is known as amalgams (Hentschel and Priester, 1992a). Authors have different opinions as to when this technology was developed, where Rose and Newman (1937) made an educated guess that the process has been in use for the last 2000 years and the two authors: Nriagu (1979) and D'ltri (1972) states that it has been in operation by the Romans since the $4th$ century BC. An even more recent date is suggested by Hentschel and Priester ;1992b) who states that this type of mining was applied in Bosnia under Emperor Nero 54 to 68 AD. The recovery of gold by mercury amalgamation is still a well known process today and has been used intensively in the small scale mining industry all over the world (Hentschel and Priester, 1992a).

The "chemistry" behind amalgamation involves the formation of the gold-mercury (or silver-mercury) alloy, which could contain two, three or four atoms of gold per mercury, giving compounds AU2Hg, AU3Hg and A14Hg, respectively (Hoffmann, 1994; Yannopoulos, 1991). Neither the gold or mercury undergo any chemical changes during the formation of the alloy, but it is rather the attraction and wetting of the gold by the mercury which takes place(Yannopoulos, 1991).

The amalgamation process consists of three basic operations in the following order (Hentschel and Priester, 1992a):

1. The raw ore is processed with the mercury, where fine alluvial gold, free gold from a ground ore or gold from the concentrate of a gravity-separation circuit is recoverable by the mercury. The contact process could be accomplished in gold pans, sluices, drums, tubs, amalgamating barrels or copper plates, edge mills, stamp mills and amalgamating tables.

- 2. The amalgam is separated from the ore. This is usually done by gravity separation, whereas both mercury and gold have higher densities as the gangue.
- 3. The gold is separated from the amalgam in two steps:
	- firstly the amalgam is separated from excess mercury by wrapping a wet cloth/chamois leather around the amalgam and wringing the cloth over a gold pan so that the mercury drips into the pan. In larger scale operations amalgam presses are used and operate on the same principle of viscosity differences, whereas the amalgam stays behind due to the larger viscosity and therefore the less viscous mercury is separated easily when pressure is applied (Hentschel *et aI.,* 1992a; Internet reference no. 54, Hoover and Hoover, 1912).

secondly, the amalgam is distilled or retorted at temperatures above the boiling point of mercury (357 \degree C) so that the mercury evaporates and gold powder remains. This is done either on open fire or closed circuit retorts to minimise the polluting effect.

The simplicity of the technique, low investment costs and comparatively high gold recoveries, has made the amalgamation process an attractive means of generating funds in the small scale mining industry (Hentschel and Priester, I992b; Subasinghe and Maru, 1994). Gold ores that are suitable for amalgamation must contain already liberated gold particles, having a grain size ranging from 2 mm to $20-50 \mu m$, that has not been encrusted, for example by ferrous oxides. *The* shape of the grain. together with the interfacial tension of the mercury and water are the defining factors for the lower limit of the gold grain size to be recovered (Hentschel and Priester, 1992b).

1.3

THE DANGERS OF EXISTING GOLD PROCESSING METHODS

Over the past years the gold mining industry has relied mainly upon processes such as cyanidation and amalgamation to recover gold from its ores. It is seen, however that these processes pose a threat to the environment due to the large scale use of chemicals such as cyanide and mercury, which are two highly toxic reagents that require very careful handling. **It** will be attempted in the following subsections to brief the reader on the dangers of these chemicals.

1.3.1 Cyanide, both nsefull and dangerous

It is seen from section 1.2 that cyanide has been used extensively in the mining industry to dissolve gold and silver from pulp. Although many noncyanide leaching agents have been investigated in the past, the use of cyanide has dominated the large scale gold mining industry (SchIettwein and Petersen, 1998).

Cyanide exists within the mining solutions in four different forms, and hence the toxicity of the cyanide present in the mining wastestreams, is therefore related to one of these forms i.e. free cyanide, iron cyanide, weak and dissociable cyanides (WAD) and cyanide related compounds.

Although the following sub-headings discuss the effect of cyanide in certain dosages with respect to the human body. it is vital to note that bird, wild and aquatic life has been influenced to a large extent as well (Hocker, 1872).

1.3.1.1 *The effect ofcyanide on the hW)ian body*

Cyanide is very harmful to the human body, as hydrogen cyanide is absorbed through ingestion or inhalation at a very fast rate and it is then carried into the plasma. Many proteins and enzymes contain key components, such as sulphur, copper and iron, which

bind strongly with HCN. An enzyme named cytochrome oxidase, which exists within the cells ofthe body, is vastly affected and can therefore lead to ashyxiation and tissue death, and eventually failure of all vital functions, followed by death (Smith and Mudder, 1991). In short it could be said that cyanide, if consumed in lethal dose at once, blocks the transport of oxygen accross the cell walls and causes death by suffocation, despite the fact that the victim has fully-oxygenated blood. The central nervous system is the first organ to succumb (Hocker, 1872).

The long term health effects of cyanide has largely been ignored, however, there is good reason to suspect that cyanide in lethal dose has serious health effects in long-term chronic exposures at low levels. Correlations have been observed between chronic lowlevel cyanide uptake and specific diseases in humans, and experiments on animals have demonstrated progressive damage to nervous and other tissues (Goldfrank, 1986 and Oak Ridge National Laboratory, 1978).

1.3.1.2 *Lethal concentrations*

The toxicological concems lie mainly with the complex forms of cyanide, as these complexes break down and hence, release the free cyanide into the system.

The mean lethal dosage for free cyanide to affect humans is within the range of 50 to 200 mg (i.e. 1-3 mg/kg), where death can occur within one hour. Furthermore, it was found that 10mg or less free cyanide per day is non-toxic, where 5 mg/day has shown no harmful effects. To reduce the risk of cyanide toxicity, it is advised that 2 litres of water should be consumed per day per person. Once a person has been exposed to HCN in concentrations ranging between 100 and 300 ppm (mg/L), death will occur within ID to 60 minutes, whereas an exposure to 2000 ppm HCN will cause death within 1 minute (Huiatt *et al.*, 1982). The LC_{50} (i.e. the lethal concentration of a toxicant that causes death of 50% of the population of organisms in a specified period) of free cyanide absorption is approximately 100 mg/kg of body weight (Smith and Mudder, 1991).

1.3.2 Mercury poisoning

The simplicity and high recoveries yielded by mercury amalgamation has made this process the perfect means of income for the uneducated and needy miners to whom a high technology process would not appeal, as additional funds and effort are required for a higher education. The miner has only one main concern: winning gold! The impact of mercury on the environment is either chosen to be ignored or due to lack of knowledge is not known to these men, which makes the extent of poisoning and pollution even more serious than anticipated.

Mercury is a highly toxic metal. With regards to its toxicity, three major chemical forms of the metal must be distinguished: mercury vapour (elemental mercury), organic mercurials and salts of mercury (Câmara et al., 1997).

1.3.2.1 *The loss ofmercury to the environment*

Mercury is lost to the environment at different stages of a small-scale mining operation and in different forms (Hoffmann, 1994):

- I. The first loss of mercury to the environment is in the tailings after it has been contacted with the ore, whereas the excess mercury is washed away in the form of floured mercury (very small mercury droplets). When in the aquatic environment, microorganisms can convert mercury into methyl mercury, which is more toxic than the inorganic forms; the compound is taken up by rlankton algae and is concentrated in fish via the food chain, reaching much higher concentrations than the originally found in the environment (Câmara *et al.*, 1997). People living up to 200 miles downstream then eat the fish.
- 2. Futher mercury *losses* occur when the excess mercury is removed from the amalgam by wringing out the solids in cotton cloth/chamois leather, as discussed

in section 1.2.2. This mercury occurs in the form of metal mercury droplets, which are lost to the environment by further handling and vapourise in time.

- 3. After the above step, the remaining amalgam then contains half gold and half mercury by weight. The mercury is then simply roasted off, great amounts volatilizing. Here the major loss of elemental mercury occurs to the atmoshpere.
- 4. Finally the amalgam, containing about 3 to 5 % mercury in its composition is then taken to gold-selling stores located in urban centres, where it is reburned to endstage purification and its gaseous residues emitted to the atmosphere without treatment (Camara *et aI.,* 1997).

1.3.2.2 The effect of mercury on the human body

Metallic mercury is not particularly toxic when ingested, because of its low absorption from the gastrointestinal tract. Mercury vapour, on the other hand is extremely dangerous because it is completely absorbed by the lung and then is oxidized to divalent mercury cation by catalase in the erythorcytes (Magos, *et ai,* 1978). The vapour crosses the membranes much more readily than does divalent mercury, which explains why a significant amount of mercury vapour enters the brain before it is oxidized. Toxicity to the Central Nervous System is thus more prominent after exposure to mercury vapour than to divalent forms of the metal. The mercury is then partially deposited in tissues combined with sulfhydryl groups. Excretion of the fraction that is not absorbed is via the urine and feces. The half-life of elemental mercury is approximately 60 days (Friberg and Vostal, 1972).

Another method of exposure to mercury is through indirect means of ingesting food contaminated with mercury. This primarily consists of fish, which metabolize mercury into methylmercury, this exposure causes the victim to succumb to Minamata desease. Minamata disease is named after a bayside village in Japan which suffered hundreds of deaths resulting from a nearby plant dumping mercury into the bay (Internet reference no 53).

Some of the syptoms found after short-terms exposure include weakness, chills, metallic taste, nausea, vomiting, dysphnea and coughing, heart palpitations, tremores, loss of coordination, whereas chronic exposure to mercury vapour produces a more insidious form of toxicity that is dominated by neurological effects.

1.4 AN ENVIRONMENTAL SOUND GOLD RECOVERY PROCESS

Environmental protection has become the focus of world-wide research in the gold mining industry and favours the development of new technology, whereby the use of cyanide and/or mercury is either lessened or eliminated. Such a process, whereby the use of any hazardous chemicals are eliminated, was developed by the British Petroleum (BP) research tearn in the early eighties and is called the Coal Gold Agglomeration (CGA) process. It is aimed to provide the reader with a background knowledge on the CGA process and its relevance to the global mining industry.

1.4.1 The Coal Gold Agglomeration (CGA) process

The Coal Gold Agglomeration process is based on the recovery of hydrophobic/oleophilic gold particles from ore slurries into agglomerates fonned from coal and oil. The oil acts as the bridging liquid between the coal and gold particles, where the coal is the carrier of the mineral and enables effective separation of the oil phase. The coal-oil agglomerates are recycled to increase their gold loading, after which they are separated and further treated to produce gold bullion (House *et al.,* 1988a; Bonney, 1988; House *et aI.,* 1988b; House *et aI.,* 1988c; Buckley *et aI.,* 1989; Bellamy *et al.,* 1989). Figure 1.7 shows a schematic representation of the basic concepts involved in the CGA process.

Gold bearing ores containing liberated/free gold particles, such as alluvial/free milling ores, gravity concentrates or gravity plant tailings was found to be suitable for the CGA process, as associations with other minerals/metals, such as pyrites, aresenopyrites etc. reduce the oleophilicity of gold, and hence limits gold recoveries (Bonney, 1988; House

et at., 1988b; Bellamy *et al.,* 1989). High gold recoveries, irrespective ofthe gold grain size (ranging in size from 1-500 μ m) are found, therefore providing an alternative to amalgamation or intensive cyanidation (House *et aL* 1988b; House *et aI.,* 1988c; Bellamy *et aI., 1989).*

1.5 THE OBJECTIVES OF THIS STUDY

It was aimed in this study to focus on the following objectives:

- to understand and optimise the CGA process route as a small-scale gold mining operation and a possible alternative to mercury amalgamation,
- to determine the effect of the mineralogy of the ore on the effectiveness of the CGA process,
- to propose a simple mathematical model to predict the thermodynamical behaviour of the CGA process.
- to perform a comparative study, whereby the CGA process is compared to the mercury amalgamation process.

Figure 1.1 - A General flowchart of processing ore to gold by utilizing the Cyanidation process.

Figure 1.2 - General flowsheet for the recovery of gold by carbon adsorption systems.

Figure 1.3 - A schematic representation of the CGA process

LITERATURE REVIEW

2.1 SMALL-SCALE GOLD MINING

The reader is now facilitated with sufficient knowledge about gold, its ores and the extraction thereof to be equipped with an understanding of the overall gold mining industry. It is seen, however, that the gold mining industry, at large, is dominated by operations such as cyanidation and mercury amalgamation.

Cyanidation has been used extensively in the large scale gold mining operations, especially in the use of the CIP and RIP circuits, which requires highly skilled operators and technological equipment. In view of complicated processes, difficult control, dependence on large amounts of reagents etc., they are regarded as less suitable for smallscale mining (Hentschel and Priester, I992b).

Mercury amalgamation, on the contrary, has been applied mainly ir small-scale mining operations, whereas the simplicity of the process, its relatively high recoveries and low investment costs has been the encouraging factors for many uneducated and unemployed peasants to enter the gold mining industry. Small-scale miners are defined as (Internet reference no 45) follows: individual casual workers who, from time to time pan streams for gold; small organised groups of four to eight individuals carrying out their operation on a full time basis using simple equipment and tools, and a co-operative of ten or more individuals.

Small-scale gold mining, such as mercury amalgamation, is an essentially artisinal or small-industrial form of raw material extraction and is characterised by the following (Hentschel and Priester, 1992a):

- little or no mechanisation in the form of machines and engines, resulting in a large proportion of heavy manual labour,
- low safety standards,
- generally low level of training,
- lack of technicians, resulting in deficient planning and organisation of extraction and processing activities,
- relatively poor exploitation of available resources due to selective extraction of rich ores in combination with low recovery rates, i.e., specific yields,
- low wages,
- low labour productivity,
- in part only seasonal employment in mines, or only as long as the world market prices are appropriately high,
- little awareness of environmental hazards,
- chronic shortage of capital,
- widespread illegal activities as a result of unfavourable mining laws and due to a lack of mineral rights/licensing.

It is seen that a small-scale gold mining operation is known for the simplicity of the process, whereas large scale gold mining operations are much more complicated and organisea, yielding much larger amounts of precious metal. Although many different small-scale gold mining operations do exist (gravity concentration, flotation etc.) (Hentschel and Priester, 1992a), it was decided not to introduce these to the reader, as the use of mercury has been preferred by the miners and dominates the small-scale gold mining industry by far. These operations cause extensive pollution and toxicity to both the miners and urban populations nearby.

Small-scale gold mining. i.e., mercury amalgamation, has been in operation for many years and many countries have a share in the gold won from these operations. It will be attempted in the next sub-section to provide a layout of the countries engaging in these operations so that the reader is facilitated with the knowledge of these parts of the world which are largely affected by the negative environmental impact of the amalgamation process.

2.1.1 Global distribution ofsmall-scale gold mining by regions

Most of the world's small-scale gold mining operations are situated in (Hentschel and Priester, 1992a):

South America, i.e., Honduras, Nicaragua, Colombia, Ecuador, Peru, Bolivia, Chile, Brazil, Suriname, Venezuela and the Dominican Republic,

Africa. i.e., Ghana, Kenya Tanzania, Zambia. Zimbabwe, Ethiopia, Guinea, Liberia. Nigeria, Gabon, the Central African Republic, Burundi and Madagascar and illegal operations in South-Africa, mainly in the Mpmalanga province (Internet reference no 68),

Asia, i.e., India. China the Philippines, Papua New Guinea, Indonesia and Malaysia.

and these are the countries greatly affected by the environmental disadvantages of smallscale gold mining.

The abovementioned countries are not the only gold mining regions, but mainly those which engage in small-scale gold mining as primary gold related income. The aforementioned countries are shown in dark colour on the map in Figure 2.1.

2.2 MERCURY AMALGAMATION

The amalgamation process could be utilised both in the small-scale and large-scale gold mining industries. In the large-scale mining industry the mercury is added to the gravity concentrate (Refer to Figure 1.1) to recover the coarser gold particles before entering the cyanidation circuit, because the larger gold particles are not dissolved that easily by cyanide. For the purpose of this study, however, the amalgamation process will be referred to as that process utilised in the small-scale gold mining industry only, as the abovementioned procedure is a part of a large scale gold mining plant. Furthermore, the large-scale gold mining industries mainly focus on the recovery of dissolved gold, whereas amalgamation is most suitable for ores comaining free/liberated gold particles, such as alluvial ores.

2.2.1 A small-scale **mining operation**

Although the amalgamation circuit could vary for different small-scale miners, depending on the type of equipment available to them, the basic principle of the alloy formation (gold-mercury alloy) is exactly the same. As discussed in section 1.2.2, the amalgamation process could be divided into three different stages, which will be discussed in more detail within the following sub-sections. It is aimed to provide the reader with an overview of the different types of equipment and methods most commonly used to recover gold by amalgamation in the small-scale gold mining industry.

2.2.l.1 Raw ore processing with mercury

The equipment used for this purpose can be divided into two categories i.e. those designed purely for the purpose of contacting the gold bearing ore with mercury (plate-, barrel- and pocket amalgamators) and secondly mercury is used in conjunction with gravity separation techniques/equipment whereby the gold recovered by these techniques are then trapped within mercury (batea, sieves, sluices, centrifugal concentrators etc.).

The amalgamation process is best suited for ores containing free/liberated gold particles for two basic reasons: firstly, the process is dependent on the formation of the goldmercury alloy and secondly, the density of the gold relative to the gangue particles facilitates the separation by gravity, whereas associations with other minerals renders the gold to be "lighter" and inhibits separation by gravity, which means the gold will most likely never reach the mercury.

The type of raw ore to be treated determines the number of dressing and preparation steps to precede the actual concentration of gold into mercury, whereas the amalgamation process is both, however, applied to sedimentary (placer ores) and primary intergrown gold ores (Branches *et al.,* 1993). Some of the equirment most commonly used for the recovery of gold utilising, mercury, will briefly be discussed below to provide the reader with background knowledge on the amalgamation practices. These processes are, however, suitable for the primary ores as well, whereas the amalgamating step is preceded by crushing and/or grinding.

Amalgamating copper plates

Amalgamating copper plates are used for recovering gold from fine stamped or milled gold ore and consists of a sloped copper plate or Muntz metal (60% Cu and 40% Zn) that have a electrolytically applied layer of silver and mercury coating. The slope of the plates should be such that the mineral particles do not sediment (depending on the density of the heaviest accompanying minerals). The gold migrates to the bottom of the pulp (gravity separation), which flows over the plates, where it comes into contact with the mercury and amalgamates (Hentschel and Priester, 1992a). It has been found that plate amalgamation is not applicable to ores coarser than 1,5 mm, heavy sulphides, oxidising substances or As- containing ores (Internet reference nr. 52).

After removing the amalgam, the plates are cleaned by scouring with sand, and washing with a strong solution of soda ash after which it is degreased with a 1% NaCN solution

followed by a thorough water wash. After the water wash, a mixture of sand, mercury and ammonium chloride (to remove base metals) is rubbed on with a stiff brush, which is repeated until the plate is covered with a thin film of mercury. The plates are recoated with a new layer of silver foil/silver nitrate when needed. For optimal performance, the following factors are very important when amalgamating with copper plates: the angle of the plates, the flowrate of the feed and, finally the size of the feed, which could cause abrasion of the amalgam and/or mercury when sized to coarse (Hentschel and Priester, 1992a; Internet reference no 52). Large amounts of metallic mercury is lost to the environment together with the discharge when the ore slurry is fed over the plates.

Amalgamating barrels

Amalgamating barrels are used to amalgamate preconcentrates and work on the same principal as a ball-mill, differing in the speed of rotation, whereas in amalgamation, the action should rather be attrition than tumbling. The barrel is charged with feed ore, water, roughly three times as much mercury as the anticipated quantity of recovered gold, grinding media, caustic soda, detergent and some cyanide to oxidise sulphides and clean gold surfaces. Without the preparatory treatment (the last three additions mentioned above), great losses of mercury due to "flouring" occurs (Penman, 1987). In the smallscale gold mining industry, the miners simply load boulders into a cement mixer to arrive at an amalgamating barrel, which leads to large amounts of "floured" mercury, as many of these miners do not have access to chemicals, such as cyanide etc.

At the end of the rotating time, the content of the barrel is subjected to different means of vibrating to facilitate the effective separation by gravity separation The mercury and amalgam gathers at the bottom for recovery, needless to say that large amounts of metallic mercury is discharged together with the tailings as it is washed away by running water.(Hentschel and Priester, 1992a).

Pocket amalgamators (Jackpots)

The operation of the pocket amalgamator/jackpot is very much the same as a round thickener. The pulp enters the deep, conical vessel through a central feed pipe extending to just above the top of the mercury. The pulp is forced to change direction, which causes the heavier gold particles to sediment, be wetted by the mercury and amalgamate. The amalgam has a higher specific gravity than the pure mercury, which causes it to sink to the bottom, leaving pure liquid mercury at the pulp/mercury interface for further amalgamating action (Hentschel and Priester, 1992a).

The batea

The batea is a circular metal/wooden pan with a pointed bottom, similar to a Chinese hat. Separation of the gold occurs by gravity, as the batea is given a circular eccentric motion under slowly floating water, whereby the lighter minerals spill over the edge into the river. Mercury is added to the bottom of the pan in order to "capture" the gold particles and as the lighter minerals are spilled, some of the floured mercury is lost to the river. This technique is suitable for very rich deposits because of its low throughput and is very commonly used in the small-scale gold mining industry.

Although many other methods, besides these mentioned, are used, it is not discussed, as those are based on the exact same principal of gravity separation of gold which is trapped into the mercury. From those mentioned, it was, however. seen that the operations are mostly careless, leading to large-scale mercury losses.

2.2.1.2 *Separating the amalgam from the ore*

The amalgamation process is based on the separation of the main components, gold and mercury, by gravity, as these usually have much higher specific gravities than the other components present in the ore slurry. For each type of equipment the procedure might differ, but the principal of separation by gravity is applied in each. As the ore slurry is processed, the gold particles migrate downwards where it is *trapped* into the mercury. Separation normally occurs by the washing away of the ore slurry, leaving the amalgam behind.

As the amalgam is separated from the ore, mercury particles are lost to the tailings, and hence the environment in which these are to be dumped, whether it be a river or stockpile.

2.2.1.3 *Separating the goldfrom the amalgam*

It is mentioned in a previous section $(1.2.2)$ that the separation of the gold from the amalgam takes place in two steps:

- I. As the amalgam contains about 50-60% weight mercury and 40-50% weight gold (Hentschel and Priester, 1992a; Veiga and Meech, 1997), the first step is to separate excess mercury by means of wrapping a wet close woven cloth/chamois leather around the amalgam and wringing it until the excess mercury drips from the cloth. This separation is based on the viscosity difference of the amalgam and pure mercury, whereas the amalgam remains in the cloth due to its higher viscosity. It is known, however, that the amalgam is not a chemical compound, but rather a physical mercury coating over gold, which explains why such separation by pressure is possible. In the larger small-scale mining operations an amalgam press is used for rapid separation of the amalgam and mercury (Hentschel and Priester, I992a).
- 2. The amalgam remaining in the cloth still contains large amounts of mercury, which is then distilled on open fire having to a temperature above the boiling point of mercury (357 °C) (Hentschel and Priester, 1992a). After this basic procedure, a further 3-5% mercury remains in the gold-amalgam *(bullion)* , which is then taken to gold-selling stores located in urban centres, where it is reburned

to endstage purification and its gaseous residues emitted to the atmosphere without treatment (Câmara et al., 1997). The risk to human health, affects not only occupationally exposed personnel, but also the general population in the surrounding areas.

2.2.2 Losses **of mercury to the environment**

As it is basic knowledge that the amalgamation process has a deleterious effect on the environment, it will be attempted in this section to provide insight into the actual dangers of the process in terms of mercury losses and statistics in some countries. It is seen from section 1.3.2.1 that mercury is lost to the environment at four different stages of the process and is summarised below (Hoffmann, 1994; Nriagu and Wong., 1997):

- I. The loss of metallic mercury in the form of "floured" mercury (mercury beads) to the environment from the tailings after it has been contacted with the ore (section 2.2.1.2),
- 2. a further loss of "floured" mercury occurs when the excess mercury is removed from the amalgam by wringing out the excess mercury through a cloth/chamois leather (section 2.2.1.3),
- 3. metallic emissions of mercury (mercury vapour) occurs when the mercury is roasted by the miners and (section 2.2.1.3),
- 4. when mercury is further refined in the gold-selling shops by further roasting (section 2.2.1.3).

It is seen that the mercury losses to the environment as a result of utilising the amalgamation process for gold recovery occurs in two main forms nl. metallic mercury (nr. I and 2) and mercury vapour (nr. 3 and 4).

In Brazil, for instance, it was estimated that 235 of the 340 tons of mercury imported in 1989 has found its way to the gold fields (Hoffinann, 1994) and the total mercury losses to the Amazon River Basin is estimated to be I 200 tons up to 1991 (Homewood, 1991). Roughly 50% of the mercury used is lost to the environment due to poorly conducted amalgamation practice (Veiga and Meech, 1997). According to Pfeiffer and Lacerda (1988) 45% of these losses are released into the rivers and 55% into the atmosphere. Cetem (1989), however has found that the distribution of mercury losses is as follows:

- 70% by volatilisation during amalgam distillation,
- 20% dragged with the amalgamation tailings and
- 10% volatized in the gold shops when gold is melted.

2.2.3 **The toxic affect of mercury to the human body**

The toxicity of mercury depends on the particular chemical form it takes, whereas the worst offenders are organomercurials, such as methyl mercury CH_3Hg^+) and elemental mercury in the form of a vapour, whereas metallic mercury in the liquid form, is not absorbed by the human body and is not poisonous in this form (Dreisbach, 1980; Hentschel and Priester, 1992b; Reynolds, 1993).

2.2.3.1 *Elemental mercury vapour*

Mercury vapour is inhaled by the human and is absorbed through the aveolar membrane in the lungs, whereas the outcome can be respiratory failure and death (Goldfrank *et al.,* 1990; Bryson, 1989). Acute mercury poisoning through inhalation takes place in phases, starting with a metallic taste, abdominal pain, coughing, vomiting, etc. and finally ulceration; in the gums and extreme light sensitivity (Lein *et al*, 1983). Chronic mercurial poisoning (mercurialism) exhibit a more insidious onset and may not be evident for several weeks or months (Florentine and Sanfilippo., 1991; Joselow et al., 1972). Symptoms include severe nervous disturbance and psychological changes (Ellenhom and Barceloux., 1988; Bryson, 1989; Bretherick, 1987).

2.2.3.2 *Methyl mercury*

Methyl mercury is formed when mercury is digested by fish where it undergoes methylation and accumulates to higher concentrations than originally "dumped" in the environment (Subasinghe and Maru, 1994).. The miners, as well as humans living up to 200 km downstream then eat the fish causing acute poisoning i.e. *Minamata,* named after the Bay in Japan where the first epidemic caused by large scale methyl mercury poisoning took place in 1950 (lrukayama, 1977; Kurland *et al., 1960).*

Methyl mercury is absorbed by blood on account of its high stability and solubility in fat, which aliows it to cross the blood brain barrier very easily, where it then accumulates in the cerebral cortex and cerebellum, causing changes to the central nervous system (CNS) (Branches *et al.,* 1993). Similar toxic effects than mercury vapour is found, haveing a more selective and permanent action on the CNS (Reynolds, 1993; Dreisbach. 1980). Typical symptoms is normally associated with sensory disturbances, constriction of the visual field, hearing impairments, impairment of speech, etc. (Tsubaki *et al., 1977;* Magos, 1997). Methyl mercury tends to diffuse across the placenta causing congenital neurotoxicity and excretions in breast milk causes further damage once the baby is born (Reynolds, 1993). As the signs and symptoms of malaria often mimic those of mercury poisoning, the diagnosis is very difficult, whereas malaria is indigenous to the areas where amalgamation operations feature.

2.2.4 Measures taken to lessen the "mercury problem"

Informal mining, such as the use of mercury in the small-scale gold mining industry in underdeveloped countries, as a rule uses rudimentary mining and processing methods, which reflect the unreliable environmental control practised by these miners. The fact that mercury poisoning is not only confined to the miners is the most disturbing fact of all, as innocent people living in the nearby vacinity of miners ani 'or gold shops suffer from the toxicity of mercury as well (Câmara et al., 1997; Veiga and Meech, 1994).

Although the "mercury problem" has been recognised by higher authorities, it is the miners who has to grasp the seriousness of the problem before any significant changes will occur. In Brazil, for instance, the government has tried to close down the informal gold mining operations (called *garimpos)* in 1987, whereas new ones have sprung up in other gold-mining regions (Hoffmann, 1994). A further step was taken to restrict the sale of mercury to the small-scale gold miners *(garimpeiros),* which lead to smuggling and black market sales increasing. The government even offered a less expensive device to recycle the mercury, but the response was poor (Emerson and Kepp., 1991). The *garimpeiros* have developed their own technology and formed unions to continue their work in the areas they occupy and would even go as far as operating illegal, irrespective the measures taken by the Brazilian government (Veig<. and Meech, 1997).

A "safer" technology, such as the use of retorts to distil the amalgam in a closed circuit, have not been popular among miners, due to the discolouring effect caused by iron and arcenic compounds, which results in a lower price being offered by buyers (Subashinghe and Maru, 1994; Hentschel and Priester, 1992b). This technology, however, does not ensure no mercury losses, but rather decreases the effect to some extent.

It is seen that the small-scale gold miners do not accept any changes to the existing amalgamation process, as the technique is effective for their purposes, providing a simple process which ensures an "easy" income. The essence of the problem, however, is that the miners are not educated as to the toxicity of the substance they so freely use to generate funds. The solution may simply be to educate them as to the dangers and toxicity of their existing technology and, at the same time, providing a equally simple and as effective environmentally sound technology.

The Coal Gold Agglomeration (CGA) process is such a new technology, suited for the small-scale gold mining industry and might be the perfect alternative to the use of mercury. The purpose of this study, however, is not to provide an infrastructure and action plan to educate the small-scale miners, but rather to understand, develop and

optimise the CGA process as a possible and valid alternative to the amalgamation process.

2.3 THE COAL GOLD AGGLOMERATION (CGA) PROCESS

The Coal Gold Agglomeration (CGA) process involves the use of coal-oil agglomerates to recover free/liberated gold particles from an aqueous slurry and was developed and patented by the British Petroleum (BP) research team in the early eighties (House *et al.,* 1988a; Bonney, 1988).

Coal and oil is added to a slurry and after intensive agitation coal-oil bridges are formed. which then build up into flocs and finally into agglomeraties. The hydrophobic/oleophilic gold particles, which are substantially free, become attached to the coal-oil agglomerates when they collide and eventually the gold penetrates into the agglomerates (Bonney, 1988). The oil is the active ingredient whereby the gold is collected. Although it is possible to recover gold particles by agglomerating them with oil, the amount of gold in the ore is usually to small and few to fonn agglomerates (Calvez *et aI.,* 1998). Therefore, it is needed to use another hydrophobic material such as coal, which in turn enables easier handling of the oil, preventing oil losses by dispersion to the water phase and hence, facilitating the separation of the oil phase.(Buckley *et aI.,* 1989).

The CGA process is most suitable for gold ores containing free/liberated gold particles, as it is the hydrophobic quality of the gold metal (Au^0) , which enables the separation thereof by the hydrophobic coal-oil agglomerates. Any associations with the gold metal, such as quarts gangue and/or coatings of a general precipitate, possibly calcium. magnesium or iron, reduce the gold hydrophobicity/oleophilicity and hence, limits gold recovery (House *et aI.,* 1988c). A collector, such as potassium amyl xanthate (PAX). is, however, added to enhance the oleophilicity of the gold. Gold particles ranging from \leq μ m to 500 μ m was found to be recovered with equal ease (House *et al..* 1988b; House *et aI.,* 1988c; Buckley *et aI.,* 1989; Bellamy, *et al..* 1989).

A simple flowchart of the basic operations involved in the CGA process is shown in Figure 1.3. It is seen that the agglomerates are either separated and further treated to produce gold after contact or recycled to increase their gold loading, whereas gold loadings of 5 to over 10 kg gold per tonne agglomerates can be achieved. The loaded agglomerates are treated for gold recovery by combustion (burnt) to volatize the oil and bum the coal, followed by dissolution and precipitation or by direct smelting (Bonney, 1988; Hughes-Narborough, 1992).

It was aimed in this section to investigate all past studies into the CGA process, identifying possible shortcomings, which served as the basis from which further work was conducted. Section 2.3.1 deals with the "discover₂" and hence, the novelty of the CGA process, whereas the section following deals with past research into the process.

2.3.1 The CGA process - A novel process

The use of agglomerates made of hydrophobic materials (such as coal) and oil to recover valuable metals, such as gold, has stemmed from research being done (by BP in 1983) in the recovery of coal from inorganic wastes by agglomerating them with oil (Allen and Wheelock, 1993; Bonney, 1988). The dynamics of the process or "liquid phase agglomeration", involves the agglomeration of particles (coal and gold) in aqueous suspension when a second immiscible liquid (oil) preferentially wets the particles and aggregates them, forming spherical agglomerates (Szymocha *et aI.,* 1989).

The CGA process was only identified as a gold recovery process in the mid eighties and patented by various companies and/or persons, such as Cadzow in 1984, BP Australia Ltd. in April 1986 (Mainwaring *et aI.,* 1986), Bateman Engineering International Ltd. in July 1986 (Sidall, 1986), Cbarlton Mineral Associates Pty. Ltd. lodged an application in August 1986 (Bonney, 1992) and finally House *et al.* in 1988(a), who is employed by BP and named it the CGA process.

The CGA process has therefore lost its novelty, but attempts to understands, refine and even optimise the process and various aspects thereof, have, however, been investigated, as will be discussed in the following section.

2.3.2 Past studies and findings on the CGA process

Initial research had been conducted by the British Petroleum (BP) research and development team, London, where many researchers have participated and during the coarse of new discoveries, published their findings. These papers include those written by Bonney (1988), House *et af.* (I 988b), House *et af.* \1988c), Buckley *et af.* (1989) and Beliamy *et af.* (1989), whereas Davy (Stockton) Ltd. was granted the technological licence by BP to commercialise the process, as attempted in the paper published by Hughes-Narborough in 1992. Any other papers mentioned in the text was published by other institutions and companies than BP.

2.3.2.1 *Batch and Pilot plant operations*

Preliminary batch tests was conducted by BP on a synthetic ore mixture (a mixture of silica sand: $d_{80} = 100$ µm and pure gold: $d_{80} = 20$ µm) and recoveries under optimal conditions (slurry densities, agitation intensities, agglomeration concentrations, residence times and gold head grades) were 80-95%. The results obtained from the batch operation then provided the data base from which a pilot plant was designed, which yielded gold recoveries between 75 and 82% for very low grade natural ores -0.56 g gold/ton ore (Bonney, 1988). Further pilot plant studies were done to investigate the long term behaviour of the agglomerates and it was found that the CGA process was capable of consistent operation, was technically feasible at a I tph (ton rer hour) scale and the agglomerates had not deteriorated in their activity during prolonged recycle, whereby gold loadings onto the agglomerates are increased after each cycle (House *et af.,* 1988c; Bellamy *et af., 1989).*

Attrition scrubbing, which is normally done to brake up cemented aggregrates or clean gold surfaces from abradable coatings, was performed on certain ore types as a preconditioning step, whereby it was found that the success of this operation as a preconditioning step was dependent on the type of ore. Results have shown that abradable coatings on gold had no effect on the recoveries by CGA and attrition scrubbing was unnecessary (House *et al.,* 1988c), whereas the break-up of aggregrates caused the liberation of gold and hence, increased gold recoveries (Bellamy *et al., 1989).*

Finally, the BP team has made it their mission to do comparative studies, whereby the CGA process was compared to cyanidation, gravity concentration and flotation. The findings were as follows:

- CGA vs. Cyanidation: CGA promises low residence times because of fast kinetics (± 30 minutes), as compared to the 24-48 hours of cyanidation, low capital costs, low reagent costs, no expensive detoxification is required and finally reduced the environmental impact.
- CGA vs. Gravity Concentration: CGA is capable ofrecovering finer gold particles (-5 μ m), yields higher gold recoveries of 90% (as compared to 50%), yields a high concentrate grade from a low grade ore, has a simple flowsheet and, therefore increases revenue through higher recoveries.
- CGA vs. Flotation: CGA reduces concentrate treatment costs due to the high grade product, reduces labour cost due to the simple process control and increases revenue due to the recovery of finer and coarser gold.

The CGA process, however, does have major disadvantages above these "large scale" gold mining operations such as, lower recoveries as by cyanidation etc. and is therefore, in this study, considered as a possible alternative to the smaller scale operations, such as amalgamation, where application is easier.

2.3.2.2 *CGA on various ore types*

Tests were conducted on various ore types, and it was found that the CGA process was most suitable for ores containing free/liberated gold particles, such as placers containing fine gold (not recoverable by gravity concentration, low grade gravity concentrates, free milling ores (where the gold can be liberated at a reasonable ore particle size) and gravity plant tailings (House *et al.,* 1988c; Bellamy *et al.,* 1989; Buckley *et al.,* 1989). House *et al.* (1988c) conducted experiments on four gold bearing materials - synthetic silica/gold mixture, black sand gravity concentrate from Indonesia, gold tailings form France and a tailings sample from Australia and found that CGA performance was independent from gold head grade and gold particle size $(500 \mu m)$ gold particles have been recovered). Klein and Sampaio (1997), conducted tests on a low grade (0.18 g/t) slime flotation tailing having a major mineral matrix formed by sericitite-muscovite (>60%) and found that the addition of additives, such as depressants, collectors and other flotation related additives (frothers etc.) were vital for treating such an ore with the CGA process. A synthetic mixture of gold powder, pure silica and sulphides was used by Calvez *et al.* (1998) to study the effect of sulphides on the CGA process, whereby it was found that sulphides had no effect on recoveries and the same conclusion was made about quartz and heavy minerals by Gaidarjiev *et al. (1996).*

In conclusion, it was found that that low gold recoveries with certain ores were due to the mineralogy of the ore (where gold is entrapped within other minerals) and not due to the process itself, whereas no further study was conducted to determine the exact effect of other minerals on the CGA process and to which extent these minerals \"ere present in the agglomerates.

As oil and coal are vital components of the CGA process, many studies have focused on the effect of different oils and coals to ascertain which are most suitable in terms of gold recoveries and agglomerant performance.

Costa *et af.* (1994) has found that diesel oil and coal formed the better agglomerates, as compared to charcoal and synthetic graphite and oils such as kerosene, soya oil and corn oil. Gaidarjiev *et af.* (1996) conducted a further study, whereby three types of Bulgarian coals i.e. black coal from the Balkan field, and lignite samples from the Maritza-lztok and Chukurovo basins, were investigated, using either vegttable oil (olive), kerosene or pine oil as agglomerating agents. It was found that the black c al and olive oil yielded the best gold recoveries, but was much lower than found by Costa *et af.* (1994). Klein and Sampaio (1997) has found that charcoal agglomerated better than the Brazilian coals used for their investigation, whereas a more viscous lubricating oil (SAE 20-40) seemed to be the best agglomerating agent and recoveries were better than for a so called SAE 10 oil and diesel oil. A further investigation was conducted by Hammelman *et af.,* 1994, on diesel and soya oil together with a metallurgical type coal, and findings were that gold recoveries were *satisfactory.* Finally, Calvez *et af.* (1998) has used a bituminous coal together with a diesel oil and kerosene as agglomerating agents and found that diesel oil yielded "stronger" or more stable agglomerates and better recoveries and stated that this was due to the fact that a lighter oil (as kerosene) yields "weaker" agglomerates, as measured by Labuschagne *et af.* (1989). Unfortunately, none of these studies have focused into the physical and chemical properties of either the coals and oils, which makes their findings inconsistent and incompatible. Therefore, further investigations into the effect of the physics and chemical character of the coals and oil on the CGA process need to be conducted

The ratio of coal to oil has proven to be an important factor, whereas it was found that the size and stability of the agglomerates were largely affected by the amount of oil and the

size of coal used, and hence alters the surface area available for gold to contact itself (Costa *et al.,* 1994; Gaidarjiev *et al.,* 1996; Calvez *et al.,* 1998). The choice of separation (either screening or flotation has been used in all of the above studies), however, dictates which size of agglomerate is favourable and weighs up to the fact that a smaller agglomerate provides a larger surface area and hence, ensures an increase in gold uptake (Gaidarjiev *et al.,* 1996; Calvez *et al.,* 1998). It could therefore be concluded that trial and error tests need to be conducted before engaging in any CGA investigations by using the amounts of coal and oil mentioned in literature, as these differ depending on factors such as oil type, size of the coal and the type of agitation speed and should only be used as a guideline. It is emphasised that optimal gold recoveries is the main objective and to assure this, the resources available should be investigated, rather than using coals and oils as dictated in literature, whereas those available might prove the better.

Further investigations were focused into the agglomerate to ore ratio, whereby it was found that a larger amount of agglomerates caused an increase in gold uptake, as more surface area was available for gold capture, but opinions differ as to the amounts used (Costa *et al.,* 1994; Calvez *et al., 1998).*

2.3.2.4 *Miscellaneous studies*

As mentioned earlier, BP has been responsible for the gross amount of research and development of the CGA process, whereas many researchers have in the past years based further studies on the results and findings published by them. It will be attempted in this section to provide an overview of additional findings over the past years, whereas previous sections have covered the overlapping studies.

Costa *et al.* (1994) performed a comparative study, whereby the CGA process was compared to the amalgamation process and it was found that the CGA process yielded better results than the amalgamation process on experimental basis. Further research,

however, needs to be done to ascertain the theoretical comparison of the two processes, which could lead to valuable predictions.

A further comparative study was performed by Klein and Sampaio (1997), whereby the CGA process was compared to flotation, using the exact same ore sample, whereas the agglomerates were added to the one sample and exposed to the same procedure as a normal flotation operation. It was found that recoveries were higher by the introduction of the coal-oil agglomerates, the reason being that the gold has a higher affinity to the oil phase than the air phase in flotation.

Calvez *et al.* (1998) has found that gold recoveries by the agglomerates were very low after adjusting the pH to very low values (pH = 2), whereas recoveries were not significantly affected by pH values between 4 and 12, which proves that the adjustment of pH is not vital for optimal recoveries. It is further speculated that low recoveries found at $pH = 2$ is due to the removal of organic and/or oxide contamination on the gold surfaces by excess H^+ , which, according to Zisman and Bernett (1970) and Schrader (1992) makes a gold surface less hydrophobic.

A Scanning Electron Microscope (SEM) was used by Calvez *et al.* (1998) to study the penetration of the gold into the surface of the agglomerates, whereby gold particles were found to penetrate as deep as $60 \mu m$ into the surface of the agglomerates. These findings are consistent with those by Cadzow *et al.* (1989), by whom it was stated that the agglomerates are capable of high gold loadings due to the fact that the gold particles are drawn within the agglomerates, leaving fresh spaces available for gold capture.

Although much is now known about the type of recoveries and grades obtained by the agglomerates, practically no study has been done to investigate the thermodynamic behaviour of the CGA system, which could lead to information on the theoretical effect of certain parameters, thus leading to valuable predictions.

2.4 SIGNIFICANCE OF LITERATURE REVIEW

The Coal Gold Agglomeration process has been the focus of much research in the past, which has lead to the believe that this "new technology" has many advantages to existing small-scale mining technology for gold extraction. Although the CGA process has many advantages to some large-scale gold mining operations, such as cyanidation (faster kinetics, cost advantages, no detoxification needed etc.) and flotation (see section 2.3.2.1), it is not the aim of this study to investigate this process as an alternative to such operations. As the CGA process has proven to be a rapid, versatile and robust technique, it would be a more useful operation in small-scale mining operations, alternatively to the mercury amalgamation process.

It will, therefore be attempted in this study to investigate the CGA process as a valid alternative to the conventional amalgamation process, studying simpler means of performing the CGA process in terms of contacting and separating the agglomerates. In order to be accepted in the small-scale mining industry, the CGA process has to be as simple and robust. Therefore, the operating parameters will be revised and investigated to find the optimal conditions for highest gold recoveries, focusing on parameters such as contact time, collector addition, means of contact and separation, the ratios of coal:oil and water:ore, oil type etc. and performing sensitivity analysis on each to ascertain their effect on gold recoveries. Although some of these parameters have briefly been investigated, it will be attempted in this study to investigate and optimise the process route of the CGA process so as to provide a valid alternative to amalgamation.

A further need exists within the understanding of the CGA process which involves the thermodynamic behaviour of this simple technology, which could lead to valuable information on certain operating parameters. As found from the literature review, no significant studies were focused on this topic. It will, however, be attempted in this study to provide a mathematical model on the thermodynamic behaviour of the CGA process and utilising it as a tool to predict the behaviour of both the CGA process. A theoretical

comparison of the amalgamation and CGA practices could then be studied, leading to the essence of better recoveries by the CGA process.

The significance of the Literature Review is summarised as follows:

- To study the CGA process as a valid and possible alternative small-scale gold mining operation to mercury amalgamation,
- To revise operating parameters and thereby obtain the optimal conditions for the CGA process, focusing on parameters such as:
	- contact time, $\frac{1}{2}$
	- mode of contact, $\mathbf{L}^{\mathcal{A}}$
	- means of separation, $\frac{1}{2}$
	- experimental ratios e.g. coal:ore; coal:oil; water:ore, $\frac{1}{2}$
	- $\omega_{\rm{max}}$ oil type.
- To ascertain the effect the mineralogy of an ore has on the effectiveness of the CGA process.
- To study the effect of recycling on the performance of the CGA process.
- To provide a mathematical model on the thermodynamic behaviour of the CGA process, which could assist in making valuable predictions. Furthermore, the model will be used to compare the CGA and Amalgamation processes on theoretical basis.

Figure 2.1 - Countries in which substantial small-scale gold mining activities occur are thc dark coloured areas (Hentschel and Priester, 1992a).

Figure 2.2 - The mercury cycle and input of mercury into the environment through mining (Hentschel and Priester, 1992b).

CHAPTER 3

THEORETICAL CONSIDERATIONS

3.1 INTRODUCTION

The Coal Gold Agglomeration (CGA) process has been described in detail in previous chapters and it is therefore assumed that the reader has a basic understanding of the concept of separation of liberated gold particles via coal-oil agglomerates (Refer to Figure 3.1). It will be attempted in this chapter to develop a thermodynamical model, which could lead to a better understanding of the theoretical working of this process.

The CGA process is a surface-dependent process (Labuschagne. 1986). which has been identified as a liquid-liquid solid separation system (Henry et $al.$ 1979) whereby gold particles (solid) are "entrapped" into the oil phase (liquid) from the water phase (liquid). Preferential wetting of oloephilic/hydrophobic particles (coal and gold) by a hydrocarbon oil forms the fundamental basis of the separation of these particles from the aqueous suspension by oil agglomeration (Mehrotra et al., 1.83), whereas the first step is the coaloil agglomerate formation. Upon particle contact. the hydrophobic qualities of the coal allows the immiscible liquid (oil) to spread over the coal surface, resulting in a film, which in return forms a "bridge" with other coated coal particles. resulting in the formation of flocs and, hence agglomerates (Keller and Barry, 1987)

As the gold particles have a hydrophobic nature, they are attached to the oil phase (coaloil agglomerates) upon agitation, whereby the oil film is thcn ruptured, causing the gold particle to be spread with oil and hence, entrapped within the agglomerate (Souci Lu, 1991; Zhongfu and Souci Lu, 1991).

From a thermodynamic standpoint, the driving force for wetting by oil and subsequent agglomeration of coal and gold particlcs is the reduction in the total surface encrgy of the system (Mchrotra *et al.,* 1983). Therefore, the CGA process was divided into theoretical States I and II, as seen in Figure 3.1. It is seen that State I is a representation of the initial contact of the three main components (gold, water and the coal-oil agglomerates), whereas State II is the representation of the coal-oil-gold agglomerate surrounded by a film of water afler being contacted for a period of time.

Figure 3.1 - Stages I and II represent the state of the different components initially (I) and after contacting (ID, where the coal-oil agglomerates are represented by oil in the diagram.

The aim was to identify initial and final conditions according to which a thermodynamical model was developed, which states the total free energy in terms of the following parameters: the ratio of radii of agglomerate to gold particles (k), the threephase contact angle $(\theta_{\text{gold/oil/water}})$ and the interfacial tension between the two liquids (Yoil/water). Therefore, the CGA process is viewed in terms of total free energy, which is a measure of the stability of a given system, whereas a lower value represents a more stable system (Henry et al., 1979).

3.2 ASSUMPTIONS

An empirical model will be derived from first principles to stipulate the thermodynamic behaviour of the CGA process. As no numerical explanations exist for non-ideal conditions, the ideal conditions were assumed and include the following:

- Although the surface of coal is a patchwork assembly of hydrophobic and hydrophilic sites, it was assumed that the coal particles used for the agglomerates had a homogeneous hydrophobic surface.
- The agglomerant (oil) is assumed to be totally immiscible in water.
- The model assumes that the "bridging" phenomenon takes place only by liquid transport supplied by an established surface film (see Figure 3.1: State II), thus the bridging of a gold particle and coal-oil agglomerate.
- It is further assumed that the gold particles are perfectly spherical and are perfectly surrounded by an oil film (i.e. completely wetted and dispersed in the oil phase). which in turn is surrounded by a film of water, whereas the coal phase is seen to be present for practical purposes i.e. enabling separation and easier handling of the oil phase etc. and was assumed to be irrelevant in all calculations.
- Both the CGA process and mercury amalgamation is assumed to be based on the fact that gold is wetted by the oil mercury and are considered to be "perfect" liquid-liquid solid separation processes. all other possible mechanisms are assumed to be irrelevant, which makes a comparison of these processes possible (Henry et al., 1979).
- \bullet A stability analysis was performed to predict if the end state of the process under consideration i.e. CGA (or Mercury amalgamation). is thermedynamically stable at equilibrium. whereas other stability considerations-factors (such as mixing. temperature and other operational conditions etc.) were disregarded.

• The transfer of gold particles from out the water phase into the oil phase, when viewed from the thermodynamic standpoint, was considered only in the initial and fmal free energy states in order to determine the thermodynamic feasibility of the process.

3.3 **MODEL FORMULATION**

Consider a spherical particle 1 (gold) of radius r_1 , initially suspended in a continuous liquid phase 3 (water), in which there is also dispersed droplets of a second immiscible liquid 2 (oil), as shown in Figure 3.1, State I. Assume that the desired final state of this system, from a separation viewpoint, is complete wetting and dispersion of the particle (gold) into the dispersed liquid phase (oil), as shown in Figure 3.1, State 11. The free energy of States I and II can then be given as (Barrow, 1989):

$$
F_1 = A_1^*(\gamma_{13}) + A_2^*(\gamma_{23}) \qquad \qquad \dots [3.1]
$$

$$
F_{\rm H} = A_1^{\dagger} (\gamma_{12}) + A_3^{\dagger} (\gamma_{23}) \qquad \qquad \dots [3.2]
$$

the free energy of each state (J/m*²)* where: F_1 and F_{II} A_1 , A_2 ^{*} and A_3 ^{*} - the surface areas of each given component (m²) - the interfacial tensions between the components (J/m^2) Y_12, Y_13, Y_23

The thermodynamic stability of the final state depicted in Figure 3.1 can be obtained by a free energy analysis, the most stable state being that with the lowest free energy and the most likely change being that associated with a negative free energy difference (Henry et al., 1979; Barrow. 1989). The free energy difference of the change in state was determined and combined with Young's equation (Adamson. 1990):

$$
\gamma_{12} - \gamma_{13} = \gamma_{23} \cos \theta \qquad \qquad \dots [3.3]
$$

which is a relationship between the various interfacial tensions and the three-phase contact angle (θ) , measured through the oil phase on the gold surface in the presence of water. The resulting equation is as follows:

$$
F'_{II-I} = \gamma_{23} [(A_3^{\dagger} - A_2^{\dagger}) / A_1^{\dagger} - \cos\theta] \qquad \qquad \dots [3.4]
$$

where: $F_{\text{II-}I}$ - the total free energy per unit area (J/m^2) γ_{23} - the interfacial tension between 2 (oil) and 3(water) [J/m²] θ - the three phase contact angle, measured through 2 (oil) onto the solid surface of I (gold) in the presence of 3(water).

By utilising the well known equation for the volume of a sphere:

$$
V = \frac{4}{3}\pi r^3 \qquad \qquad \dots [3.5]
$$

it was possible to convert the areas in equation [3.5] to radii. A simple relationship between the radius, contact angle and total free energy per unit area was found as follows:

$$
F'_{II-1} = \gamma_{23} [(k^3 + 1)^{2/3} - k^2 - \cos\theta] \qquad \qquad \dots [3.6]
$$

The above equation (3.6) expresses the total free energy/unit area (F'_{II-I}) as a function of γ_{23} , θ and k, which could now serve as an empirical model.

Equation 3.6 can also be used to evaluate the effect of \'arious agglomerants. which is characterised by various interfacial tensions (γ_{23}) and contact angles (θ), on the thermodynamic stability of a *specific process,*

The total free energy per unit surface (F'_{II-1}) was non-dimensionalised by the interfacial tension (γ_{23}) , a measurable value which is fixed for a given system. so that a simpler

relationship between the total non-dimensionalised free energy $(F'')_{H-I}$, the three-phase contact angle (θ) and k exists:

$$
F''_{H-1} = (k^3 + 1)^{2/3} - k^2 - \cos\theta \qquad \qquad \dots [3.7]
$$

where: F''_{H-1} - non-dimensionalised total free energy

 k - the radius of the coal-oil agglomerate divided by the radius of the **gold particle (rcoal-oll** agglom~ratcs/rgold **particles)**

The objective of non-dimensionalisation was to provide a simpler equation which was easier to handle when evaluating the stability/free enegy of a *specific process* with respect to k and θ . For future reference it should be noted that the dimensionalised (F'_{II-1}) and non-dimensionalised (F"_{II-I}) free energies are linked in the following manner:

$$
F'_{II-I} = \gamma_{23} (F''_{II-I}) \qquad \qquad \dots (3.8)
$$

The thermodynamic stability, however, is not only of importance whcn evaluating a *specific process,* but is one of the determining factors when comparing different processes of similar theoretical background. Therefore, it is vital that the value of the interfacial tension (γ_{23}) be taken into account in this instance as the magnitude of this parameter could largely affect the total free energy/surface area (F'_{II-1}) and hence, the thermodynamic stability. Furthermore. the utilization of equation 3.7 is then no longer sufficient as it does not contain the term γ_{23} and equation 3.6 is utilised.

3.4 SENSITIVITY ANALYSIS

3.4.1 Introduction to the analysis

For a given system *(specific process)*, the values of γ_{23} and θ are constant, and are characteristic of the agglomerant used. A.s the specific agglomerant (oil) dictates the values of γ_{23} and θ , it was necessary to investigate the effect of each of these parameters

on the thermodynamic stability, as the choice of agglomerant might vary and, hence effect the thermodynamic stability of a *specific process*. In order to simplify the evaluation of the thermodynamic stability of such a system with respect to θ , it was necessary to eliminate the presence of γ_{23} , which explains why equation 3.7 instead of equation 3.6 was used to perform the sensitivity analysis. For a specific system to be thermodynamically stable, the free energy must be negative. Obviously a higher absolute negative value for the free energy will result in a more stable system. Therefore, it is fair to eliminate γ_{23} from the equation, as the interfacial tension (γ_{23}) is by convention always positive (Glasstone *et aI.,* 1981) and would not affect the stability of the system. Hence, $F'_{II-I} = \gamma_{23} \left[(k^3 + 1)^{2/3} - k^2 - \cos\theta \right] = \gamma_{23} (F''_{II-I}).$

Therefore, the analysis presented here clearly reveals whether or not a *specific* liquidliquid particle separation process is thermodynamically stable and hence. feasible for a given system in terms of the specific three phase contact angle (θ) , and the agglomerate (or liquid droplet) size/solid particle size ratio (k) for that system. It will be attempted to identify the specific values of these governing parameters that are needed for thermodynamic stability and predict the ranges under which a *specific process* would be a feasible operation. The above will be done by performing a sensitivity analysis on equation [3.7], which is the relationship between the non-dimensionalised free energy (F''_{II-I}) , agglomerate/solid particle size ratio (k) and three-phase contact angle (θ).

3.4.2 Background on contact angles (θ)

The physical working of a liquid-liquid solid separation process has been ascribed to the wetting of the solid by a second immiscible liquid, which makes the recovery of the solid particles into this liquid phase possible. According to literature(Boucher and Murrel, 1982), the term wetting is used only when the liquid under consideration spreads over the surface of the particular solid and the extent of spreading (wetting) is defined by the magnitude of the contact angle of the liquid on the solid surface. whereas the limiting

case for the contact angle is zero. The cases of $\theta > 0^{\circ}$ are divided, by convention, into wetting and non-wetting conditions (Boucher and Murrel, 1982):

$$
0^{\circ} < \theta < 90^{\circ}
$$

20° $90^{\circ} < \theta < 180^{\circ}$
30°
30°
40°
40°
90°
40°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°
90°

3.4.3 The effect of k and 8 on thermodynamic stability

In order to find the relationship between the three-phase contact angle (θ) and the thermodynamic stability, a plot of the non-dimensionalised total free energy $(F_{II,J})$ versus the ratio of agglomerates to solid particle rad \vec{a} (k) at selected contact angles between 0 and 90 \degree (wetting conditions), is shown in Figure 3.2. From the graph it is seen that the value of F''_{H-1} decreased as the value of the three-phase contact angle (θ) decreased, which indicates that a more stable thermodynamic configuration is found at contact angles close or equal to zero and confirms the fact that such a process is dependent on the wettability of the solid by the second immiscible liquid phase. Furthermore, it is seen that the relation of F''_{II} to k is an asymptote, which implies that the value of F"n.! *strives* to its lowest possible value at the contact angle under consideration (the cosine of the three-phase contact angle). as the value of k increases. Although the value of $F_{\text{II-1}}$ will never reach this lowest value, it is seen, however that the value of k only effects the non-dimensionalised total free energy to a certain extent. hence. for large values of k the effect is so minimal that it could be ignored.

A plot of F''_{H-1} vs. θ is depicted in Figure 3.3 for selected values of k. The findings from this graph confirms those that were made from the previous graph. where F''_{II-I} is only at its lowest (which by convention is a negative value and implies the most stable configuration or thermodynamic state. under the following conditions):

where the three-phase contact angle is closest or equal to zero ($\theta \rightarrow 0^{\circ}$), and

It is further seen from Figure 3.3 that for every value of k there exists a critical threephase contact angle $(\theta_{\rm crit})$ below which the free energy of State II is less than that of State I (F^u_{II-1} is negative) and are the values of θ where the F^u_{II-I} curves intersects the θ axis. By utilising this information, it is possible to define the **range** in which the thennodynamic stability would be favoured the most, which is presented in Figure 3.3 by the rectangular marked ABCD. The ideal range of θ for feasible operation (rectangular ABCD, Figure 3.3) was calculated by selecting the minimum and maximum values for k and calculating the maximum allowable values of θ accordingly, utilising the point where the "F"_{II-1}-line" for each k_{min} and k_{max} intercepts with the θ -axis as follows:

By assuming that $k_{min} = 1$ (and $k_{max} = \infty$), equation 3.7 was used to solve for θ where $F''_{II-I} = 0$:

Solve for θ at $k_{min} = 1$: $F''_{II-I} = [(1^3 + 1)^{2/3} - 1^2 - \cos \theta] = 0$ $\cos \theta = 0.587401$ $\theta_{\text{max/crit}}$ = 54°

Solve for
$$
\theta
$$
 at $k_{\text{max}} = \infty$:

$$
F''_{\text{II-I}} = [(\infty^3 + 1)^{2/3} - \infty - \cos \theta] = 0
$$

$$
\cos \theta = 0
$$

$$
\theta_{\text{maxcrit}} = 90^\circ
$$

The above calculations state the maximum allowable values for the three-phase contact angles at each of the k_{min} and k_{max} values. Therefore.

• For k_{min}, which is 1, the critical/ maximum allowable value for θ , i.e. $\theta_{\text{max-crit}}$ was calculated to be 54° . This statement entails that an operation could only be feasible at k=l, when the three-phase contact angle (measured through the denser and

immiscible liquid on the solid surface in the presence of the less denser liquid) does not exceed 54°.

• For k_{max}, which is ∞ (infinity). θ_{max} was calculated to be 90° and implies that the critical/maximum allowable contact angle for feasible operation should not exceed 90° if the ratio of radii of agglomerates to solid particles strives toward infinity.

Finally, it could be concluded that as long as the actual value of k is within the range on I to ∞ , and the three-phase contact angle stipulated for each minimum and maximum k value is adhered to, the operation should be thermodynamically stable and, hence feasible.

Figure 3.4 shows the critical three-phase contact angle as a function of k, and it is clear that if θ strives to 90°, distribution and hence, separation into the oil phase, is only possible for large values of k, which confirms the above statement. Therefore. it is seen that for very large values of k the critical contact angle is always 90° (see Figure 3.3). which points out that the three-phase contact angle between the second immiscible liquid (indicated as 2 in Figure 3.1) and the solid (indicated as I in Figure 3.1) measured in the presence of the first liquid, has to be below or equal to 90° in order for the thermodynamic stability to be at its lowest, which is a negative value for $F''_{\text{II-1}}$.

Referring to the value of k, it is seen from all three graphs that this value only has a marked effect on the CGA process when \ery smalL e.g. below 10. whereas larger values favours the thermodynamic stability, but does not really effect it above the mentioned values for k. **In** fact, this means that the thermodynamic stability is fa"oured where the size of the agglomerates are much larger than that of the gold particles.

3.4.4 The effect of γ_{23} on the thermodynamic stability

As the interfacial tension (γ_{23}) was used to non-dimensionalise the free energy from F'_{II-1} (total free energy per unit area) to F''_{II-1} (non-dimensionalised total free energy), see
equations 3.6 to 3.7, the effect of this parameter was not investigated, as mentioned earlier in this section. The interfacial tension (y_{23}) [and three-phase contact angle (θ), which has been investigated], has a significant effect on the thermodynamic stability in two major instances (see equation 3.6):

- I. Firstly, when investigating a *specific process,* the choice of the agglomerant (oil) dictates the actual values of both θ and γ_{23} , which is the characteristic properties of a specific agglomerant/second immiscible liquid (oil). Therefore, the thermodynamic stability of a *specific process* could largely be affected by the agglomerant type selected, and hence the theoretical implication thereof. By measurement of γ_{23} and θ , it is possible to determine the most feasible agglomerant to use.
- 2. Secondly, when *different processes* of similar theoretical background are compared, this value becomes of major importance, whereas the magnitude γ_{23} might change the magnitude of the total free energy per unit area (F'_{II-1}) and hence. enhance or inhibit thermodynamic stability. Therefore. by direct measurement, the surface tension (γ_{23}) and three-phase contact angle (θ) can lead to an indication of which process is the more feasible operation in terms of theoretical stability.

As γ_{23} is by convention always a positive value (Glasstone et al., 1981), it does not effect the thermodynamic stability in the same way as k and θ . The numerical value of γ_{23} influences the actual numerical value of F'_{II-I} , which is why this va lable is of such importance. If equation 3.6 is modified slightly by replacing the part containing k and θ by F"II-I, as seen in the equation below, the above statement could be explained in a simpler manner.

$$
F'_{II-I} = \gamma_{23} [F''_{II-I}] \qquad \qquad \dots (3.8)
$$

where:
$$
F''_{H-1} = [(k^3 + 1)^{2/3} - k^2 - cos\theta]
$$
 ... (3.7)

By convention, the interfacial tension (γ_{23}) is always a positive value, as mentioned earlier, which implies that the actual magnitude of F_{II-1} is determined by the magnitude of γ_{23} . Although this is true, it is also seen that the magnitude of γ_{23} is not capable of ensuring thermodynamic stability, but could rather enhance or inhibit it. Therefore,

$$
F'_{II-I} = \gamma_{23} [-F''_{II-I}]
$$
 or
$$
F'_{II-I} = \gamma_{23} [+F''_{II-I}]
$$
Thermodynamically stable

The magnitude of γ_{23} either fascilitates thermodynamic stability by making F'_{II-1} more negative or inhibits it by making F'_{II-1} more positive, depending on the order of magnitude of γ_{23}

3.4.5 Conclusions

It could, therefore be concluded that k and θ is the determining factors dictating the actual "sign" of F'_{II-I}, and hence, the thermodynamical stability. whereas the order of magnitude of γ_{23} enhances or inhibits the effect these parameters have on the thermodynamic stability. Therefore. equation 3.7 is known as the thermodynamic stability model and is utilised to predict ideal conditions and the ideal range of k and θ in which the thermodynamic stability is most favoured.

The ideal conditions were for maximum thermodynamic stability found to be:

- when the three-phase contact angle strives to its lowest. $\theta = 0^{\circ}$ and when
- the ratio of agglomerate to solid particle radii strives to its maximum. $k \rightarrow \infty$

As the ideal conditions is a vague measure, the **ideal range** to ensure thermodynamic stability was stipulated as follows:

- For $k_{min} = 1$, the critical/maximum allowable three-phase contact angle, $\theta_{max/crit} = 54^{\circ}$,
- for k_{max} = ∞ , the critical/maximum allowable three-phase contact angle, $\theta_{\text{max/crit}} = 90^{\circ}$.

3.5 PARAMETER ESTIMATION

From the above, it was found that both equations 3.6 and 3.7 are valuable tools and has a dual purpose, where predictions as to the behaviour of the CGA process under certain theoretical conditions i.e. agglomerant type could be made. Furthermore, it also allows the CGA process to be compared to other processes, such a_s mercury amalgamation.

Although the total free energy (may it be F''_{II-I} or F'_{II-I}) and hence, the thermodynamic stability, of the CGA process is a defining factor, it is, however. not the only factor to be considered when dealing with optimum conditions for the recovery of gold. The percentage gold recoveries is amongst others influenced by more than one factor. as illustrated in Figure 3.5. As seen from the flowdiagram. the thennodynamic stability is one of three determining factors to be considered before any predictions as to the percentage gold recoveries are made and is illustrated in the equation to follow:

% Au recovery =
$$
f
$$
 (Thermodynamic stability; max. Au transfer;
separation efficiency) ... (3.9)

The above equation states that the percentage gold recovery is also a function of the maximum gold transfer and separation efficiency. This implies that the thermodynamic stability is not the only factor to be consulted when predictions are to be made or explanations of typical recoveries are seeked. Furthermore. it is seen from Figure 3.5 that each of the above factors are then, in turn, a function of some other operating parameters. as will be illustrated by the equations in the sub-heading to follow.

3.5.1 Thermodynamic stability

Equation (3.10) is in effect a simpler form of equation 3.6, and implies that the measure of the total free energy per unit surface area (F'_{II-1}) is a measure of the thermodynamic stability. (where γ_{23} = interfacial tension between oil and water, θ = three-phase contact angle and $k = r_{\text{agglomerates}}/r_{\text{gold particles}}$)

Thermodynamic stability (F′_{II-I})

\n
$$
= f(\theta; \gamma_{23}; k)
$$
\n...(3.10)

$$
= \gamma_{23} [(k^3 + 1)^{2/3} - k^2 - \cos\theta] \qquad \qquad \dots (3.6)
$$

$$
= \gamma_{23} [F''_{11}] \qquad \qquad \ldots (3.8)
$$

It is, however, found from the theoretical investigation in the previous sections (subheading 3.4) that the CGA process would be the most feasible operation and hence. yield higher gold recoveries where the conditions were most favourable for thermodynamic stability and hence, a lower value for F''_{II-1} (non-dimensionalised free energy) and requires the following to be true:

- The three-phase contact angle has to be within the range of 0 to 90° , which implies the use of oils which wets the surface of the gold in the presence of water. whereas the oils yielding three-phase contact angles closest to 0° would be the most suitable.
- Furthermore, it is found that the value of k had to be fairly large (at least above 10). which means that the size of the agglomerates has to be much larger than the size of the gold particles to be recovered by the agglomerates. Funhermore. it is seen that the value of k has no effect on the thermodynamic stability of the CGA process if these values strive to the largest possible value ($k \rightarrow \infty$), whereas the critical value for the three-phase contact angle in these circumstances had to be below 90° for the CGA process to be in its most stable configuration (see Figure 3.3). This implies that large agglomerates would perform the best if the three-phase contact angle between the oil and the gold surface in the presence of water is below 90° striving toward 0° (where $k_{max} = \infty$, $\theta_{max} = 90^{\circ}$. Moreover, the minimum allowable value, k_{min} , was chosen as

I, which states that the gold particles to be recovered should not be larger than the agglomerates and under these conditions the three-phase contact angle should not exceed 54° to ensure maximum stability.

• Finally, the value of the interfacial tension (γ_{23}) is of vital importance when comparing various oil types as agglomerant, but does not effect the thermodynamic stability (F'_{II-i}) in the same way as k and θ . As the numerical value of γ_{23} is always positive by convention, it could either cause an enhancement or inhibition of the thermodynamic stability, depending on the orientation of F''_{II-I} , which is defined by the actual values of k and θ .

3.5.2 Maximum Gold transfer

The maximum gold transfer is in effect the maximum amount of gold particles which has been transfered into the coal-oil agglomerate phase and is effected largely by operating parameters, such as shown in the equation below (see Figure 3.5):

Maximum Au transfer =
$$
f(C; T; S; M; Y)
$$
 ...(3.11a)

- where: $C = \text{Mode/motion of contact}$
	- $T = Period of contact (minutes)$
	- $M =$ Mineralogy of the ore
	- $S =$ Available surface area of agglomerates
	- $Y = Collectron addition$

where the available surface area of the agglomerates (S). in turn is a function of some other variables, as seen in the following equation:

$$
S = f(r_{\text{coal-ol age}lomerates}: N) \qquad ...(3.11b)
$$

where: N = The amount/number of agglomerates present The above equation $(3.11a)$ involves the following:

- The mode of contact (C) is the motion provided to assure that gold particles and coal-oil agglomerates are in sufficient contact, whereas the mode/means chosen could either increase the probability of the gold particles being attached/transfered to the agglomerates or retard it.
- The contact time (T) is the residence time which the coal-oil agglomerates and gold particles are allowed to be in contact, whereas a sufficient time of contact increases the probability of the agglomerates and gold particles being attached, causmg a poss: ble increase in the transfer of gold particles into the coal-oil phase.
- The **mineralogy of the ore (M)**, reflects the amount of *free/liberated* gold particles available in solution, whereas gold that is associated with other minerals have a lesser chance of being transfered into the coal-oil phase, as these minerals encapsulate the gold, rendering it less hydrophobic.
- The available surface area of the agglomerates (S) is in effect the total surface area of agglomerates that are available to gold particles to attach themselves to, whereas α larger total agglomerate surface area increases the probability of gold particles being attached successfully. It is. however seen that the total surface area of the agglomerates is affected greatly by two other parameters (see Equation 3.llb):
	- 1. $r_{\text{coll-oil agent}}$, which is a measure of the size of the agglomerates and hence, the available surface area of agglomerates for gold particles to contact themselws. providing that the radius of the agglomerates are larger than that of the gold particles ($r_{\text{coal-oul-}}$ agglomerates $> r_{\text{gold particles}}$) to be contacted. Smaller agglomerates are kept into suspension more readily and provides a larger surface area than large agglomerates, thereby increasing the probability of a gold particle being attached to a coal-oil agglomerate.
	- 2. The total amount of agglomerates influences the available surface area of agglomerates in the sense that more agglomerates in suspension provides more surface area for gold particles to contact themselves to, possibly increasing the pld transfer into the coal-oil phase

• By **collector addition,** the hydrophobicity of the hydrophobic components, i.e. coaloil agglomerates and gold is enhanced, increasing the hydrophobic attraction of gold particles by the coal-oil agglomerates and possibly increasing the gold transfer into the coal-oil phase.

3.5.3 Separation efficiency

The separation efficiency is a measure of the efficiency/completeness of separation and is influenced by certain operating parameters, as indicated in the equation below:

Separation efficiency =
$$
f(r_{\text{coal-ol agglomerates}}; X; Y)
$$
 ... (3.12)

Equation 3.12 implies that the efficiency of separation is largely determined by the size of the coal-oil agglomerates $(r_{coal-oil \ agelomerates})$ to be recovered. whereas it could either fascilitate or inhibit the efficiency of separation depending on the **means of separation** chosen. This implies that smaller agglomerates are separated easily by scraping them from the surface. whereas the larger agglomerates may require the use of a sieve. Whatever the case may be, it is vital to acknowledge that the effective separation of the bulk of the agglomerates as of major importance. as these are the "carriers" of the gold particles that are recovered. The **addition of a collector.** however, is of 'ital importance. as this chemical:

 (1) enhances the floatability of the floatable components, i.e. the coal-oil agglomerates. which fascilitates the separation thereof by scraping and (2) enhances the hydrophobicity of the hydrophobic components, i.e. coal, oil and gold, thereby increasing the hydrophobic attraction of gold into the coal-oil agglomerates as mentioned earlier.

3.5.4 CGA versus Merury Amalgamation

The model can be used to compare similar processes, such as coal gold agglomeration and mercury amalgamation on the basis of thermodynamic stability. The following conclusions were made:

- The CGA process and the mercury amalgamation prc cess is most likely to differ with respect to θ and γ_{23} , whereas these values could be met sured directly and by utilising Equation 3.6, provides a measure of the more stable process.
- As other factors, such as illustrated in Figure 3.5 and discussed in the previous subheadings play an important part in percentage gold recoveries, **it** is vital that these bc taken into account when comparing different processes, as the model can not fully predict which process would yield better recoveries.

k (rcoc.l-OJI agglorrerates : rgold)

Figure 3.2 - The non-dimensionalised Total Free Energy (F"_{II-I}) vs. k at selected values for the three-phase contact angle (θ) between 0° and 90° .

Figure 3.3 - The non-dimensionalised Total Free Energy (F"_{II-I}) vs. the three-phase contact angle (θ) for selected values of k ($r_{\text{coal-oul}}$ agglomerates: r_{gold}).

Figure 3.4 - Variation of the critical value of the Three-phase contact angle with the size parameter k.

Figure 3.5 - A simplified flowdiagram of all the determining factors influencing the percentage gold recoveries by the Coal Gold Agglomeration (CGA) process.

CHAPTER4

THE COAL GOLD AGGLOMERATION (CGA) PROCESS IN BATCH CONFIGURATION

4.1 MATERIALS USED

The experimental set-up used during this study is shown in a very simplified diagram in Figure 4.1. It is seen from the first step, which is the contacting of all the components, that the main materials used are coal, oil and a gold bearing ore. The characteristics of the materials used for this part of the study are as follows:

1. Coal

- A sample received form Athlone Powerstation in Cape Town
- Density = 833.846 kg/m^3
- Contains 14% ash and 25% volatile matter
- The BET surface area was measured as $\pm 4 \text{ m}^2/\text{g}$
- Calorific value = $25.5 28$ mJoule/kg
- Milled down to a particle size $\leq 90 \mu m$
- 2. Oil (chosen from chemical store, no preference was given to any specific oil)
	- Oil type: Ethane Oleate $(C_{17}H_{33}.COO.C_2H_5 = 310.52)$
	- Density = $869 874 \text{ kg/m}^3$ at 20 °C
- Acid value of not more than 0.5
- Iodine value of 75 84
- Assay (saponification) = $98 103\%$
- 3. Cald ore
	- A synthetic ore mixture of grade 7*g1t* was prepared from a silica sample having a particle size distribution in the range of 25 to $300 \mu m$ of which 40% was in the $106 \mu m$ range (see Figure 4.2). Therefore, 1 kg sample was used and weighed out as follows:
		- $*$ (-300 + 212) μ m: 150g
		- * $(-212 + 150)$ µm: 150g
		- * $(-150 + 106) \mu m$: $400g$
		- * $(-106 + 75) \,\text{µm} : 150 \text{g}$
		- * $(-75 + 25) \,\text{µm} : 150 \,\text{g}$ TOTAL 1000g
	- The gold powder used had a particle size of 44 μ m and according to trace analysis on an Induction Coupled Plasma, contained 60 ppm silver. 15 ppm iron, I ppm magnesium and the remainder was gold.
	- The synthetic mixture was prepared by mixing 0.007g of the gold powder with the 1kg silica sample in order to prepare the $7y/t$ ore sample.
- 4. Other materials
	- Distilled water was used to prepare the ore slurry.
	- A Collector, named potassium amyl xanthate (PAX) was used to enhance the floatability of the floatable components e.g. coal-oil agglomerates.

4.2 PRELIMINARY TESTS

Initially, it was attempted to understand the concept of the CGA process and from that develop an appropriate experimental program.

Trial-and-error tests were performed mainly to determine the sample sizes and amounts of coal, oil, water and gold ore needed and this was done within the following ratios: coal to oil; coal to ore and water to ore. These tests were done by adding different amounts of the various components while assuming the densities to be as follows: $\rho_{\text{cal}} =$ 834 kg/m³; $\rho_{\text{oil}} = 870 \text{ kg/m}^3$; $\rho_{\text{silica (sand)}} = 2500 \text{ kg/m}^3$ and $\rho_{\text{water}} = 1000 \text{ kg/m}^3$, whereas the symbol ρ denotes the density. The experimentation led to the following ratios:

- coal:oil 100:20 (by volume) and involves the addition of approximately 4.17 g coal to 1 ml of oil to justify this ratio.
- coal:ore 1:1 (by mass) i.e., exactly the same mass of ore and coal was used.
- water:ore 135 ml water/4g of gold ore to prepare an ore slurry.

The concentration and amount of collector i.e. PAX was initially selected to be 10 ml of a 19/100ml solution for sample sizes corresponding to the use of Iml oil.

4.3 THE BASIC EXPERIMENTAL PROCEDURE FOLLOWED TO PERFORM BATCH TESTS ON THE CGA PROCESS

All batch experiments were conducted in glass bottles of a 200 ml volume each having a screw on lid, therefore the sizes of the samples were kept manageable at this volume, facilitating the analysis thereof.

A single *standard* batch CGA experiment was then performed as follows (see Figure 4.2):

I. Representative samples of the "dry" components were weighed out according to the ratios determined earlier and the exact amounts used for a single batch test was: 4.17 g synthetic gold ore and 4.17 g coal, whereas the coal was weighed out directly into the 200 ml bottles.

- 2. The oil was added to the coal in the amount of I ml per 4.17 g coal used (based on the ratio coal:oil = 100:20 by volume, $\rho_{\text{coal}} = 834 \text{ kg/m}^3$ and mixed until agglomerates were formed. The coal-oil agglomerate formation could proceed either on dry basis (as above) or wet basis, whereby water was added and mixing proceeded. As the results are not effected, both methods were suitable for obtaining maximum recoveries.
- 3. The next step was to add the ore and remainder of the water (125 ml) together with 10 ml of the 100g/ml collector, i.e. PAX in our case, which added up to the water/liquid required i.e. \pm 135 ml per 4 g ore.
- 4. The lid was screwed on and the bottles placed inside the laboratory scale mechanical shaker and the contents contacted for 50 minutes.
- 5. After contacting the coal-oil phase and the slurry, the coal-oil-gold agglomerates were removed by firstly shaking the bottle so that the floatable agglomerates could be removed by scraping them from the surface of the aqueous medium. The lid was replaced three to four times and the procedure repeated to make sure that all the agglomerates had been removed. This was done approximately 5 minutes apart to ensure that the majority of the silica had settled out and the finer particles not removed together with the agglomerates.
- 6. The coal-oil-gold agglomerates were then dried overnight at 80 \degree C on a filter paper which was placed onto a watch glass, after which the dry and cooled off sample was then weighed out into a crucible. The crucible was then placed inside a muffle furnace where ashing proceeded at 700 \degree C for 7 hours. The crucibles containing the ash was allowed to cool, after which the weight of the ash was measured.
- 7. The remaining ore slurry was filtered in order to recover the remaining ore, which was dried and "ashed" in the exact same manner as the coal-oil phase. lt was attempted to ash all the "foreign" materials (even a percentage of coal remaining in the slurry) so that the effect of these on the analysis was eliminated.

8. Note that a set ore samples of the same representative sample was kept aside and analysed separately to determine the initial gold loading of the ore, so that the mass balance could be checked.

For each investigation, the above batch operation was repeated at least 3 (to 6) times in the same run so that the results mentioned later on are all the average of 3-6 identical experiments.

4.4 ANALYSIS FOR GOLD

It was essential to find an appropriate means to analyse for gold in both the ore and coaloil phases. According to thermodynamics, neither aurous *ar* auric cations arc stable in aqueous solution, but will be reduced by water to metallic gold. **In** order to stabilise these ions in aqueous solutions, it is necessary to introduce a complexing ligand and to employ a suitable oxidizing agent, since no reaction occurs unless both are present in solution. therefore both a complexing agent (such as HCl) and a suitable oxidising agent (such as $HNO₃$) need to be in solution for the gold to dissolve. The solution described above is called *aqua regia* and is generally used for the dissolution of gold for analytical purposes and consists of 82% hydrochloric acid (HCl) and 18% nitric acid (HNO₃) (Nicol *et al.*, 1987; Yannopoulos,1990).

All the "ashed" samples (coal and ore) were treated with *aqua regia* so that the gold present in each sample was in solution. The procedure followed in this study involved the use 5 ml of *aqua regia* for each sample (the weight of each sample to be analysed was rarely more than 5g) which was then put on a hot plate until the solution has "cooked" dry, this was repeated three times. After adding the solution to the sample for the third time it was not allowed to cook dry, but only until most of the vapours have escaped, as this procedure was done in a fume cupboard. The contents of each crucible was filtered thoroughly into a volumetric flask of appropriate volume. which contained enough

hydrocloric acid (HCI) to make up a 10% solution if distilled water was added. The HCI prevented the dissolved gold of precipitating out.

The contents of each volumetric flask was then analysed for gold on the Graphite Tube Atomiser (GTA), which allows the direct analysis of the acid solution (aqua regia). The GTA is generally used to analyse samples of very low concentrations, which explains why the samples are kept at such small amounts. A further advantage is that the use of smaller samples allows the analysis of the entire sample, whereas only a part of a larger sample (i.e. a representative sample of the larger sample) could be used for analysis and increases the probability of ending up with inaccurate or inconsistent results.

The GTA operates at the same principal as a furnace of wl ich a graphite tube (5 mm in diameter) serves as the furnace into which a very small volume (20 microlitres) of the sample solution is injected into a 1.5 mm transverse hole by using a microsyringe. During the analysis, the temperature inside the tube is raised, subjecting the sample to three temperature stages: drying, ashing and finally atomization, whereby free gold atoms are generated and the atomic adsorption measured, utilising a standard gold lamp. Gold standards of concentrations 0.01, 0.02 and 0.04 ppm was prepared and the atomic absorbance of each measured on the GTA. A calibration curve is drawn up, whereby the mass of gold (linked to the concentration of the standards) is plotted versus the absorbance for the standards. It is vital to note that the relationship between the mass of gold and the absorbance (as read on the GTA) is a straight line only below concentrations of \pm 0.06 ppm. For concentrations above this critical value, the calibration curve is more of a polynomial relationship than a straight line, which complicates the conversion of absorbance to mass gold present in the samples analysed on the GTA. Therefore. the actual samples to be analysed were diluted to such an extent that the maximum possible concentration of each does not exceed 0.04 ppm, which is still within a save range for accurate conversions. This explains why the CGA experiments are performed on such small scale, whereas minimum loss of sample takes place and accuracy is enhanced. The relationship for the straight line, as calculated from the calibration curve is then used to

convert the absorbance readings to the exact mass of gold present in each sample. Refer to Appendix A, SAMPLE ANALYSIS, for a more detailed outline of the GTA analysis.

By calculation it is possible to determine the exact weight of gold originally present in either the agglomerates and/or ore samples. The percentage gold recovery into the coaloil phase is calculated as a percentage, whereby the exact weight of gold present in this phase is expressed as a percentage ofthe exact weight of gold originally present in the ore sample before performing the CGA experiment.

The first official analysis (many trial-and-error analysis were done prior to this one for the familiarisation to the equipment), was performed on the synthetic gold ore to determine if the gold present in the representative samples corresponded to the theoretical amount of gold that was added, which is 7 g gold/ton ore.

4.5 RESULTS AND DISCUSSION

According to the results obtained after analysing the synthetic ore sample (Experiment 4.1, Appendix A) on the GTA, it was found that the actual gold loading on the specific representative samples taken averaged 5.93 g gold/ton ore. *Although* a representative ore sample was prepared and analysed for each sample utilised in a single batch CGA experiment, this reading was done purely to determine the consistency and accuracy of the equipment.

After performing the CGA test on the synthetic ore (theoretically $7g(t)$, it was found that an average of **38.4%** (analysis done on the GTA) of the gold originally in the ore phase had been recovered into the coal-oil phase (see Experiment 4.2, Appendix A). This result is the average of three identical experiments and is regarded as the result of the standard batch CGA experiment. All future experiments were based on the same experimental procedure as followed in this section.

Figure 4.1 - Particle size distribution of the silica sample used to prepare the synthetic gold ore.

Figure 4.2 - A Simplified flowdiagram of the Experimental Setup of the Coal Gold Agglomeration (CGA) process.

CHAPTER 5

THE EFFECT OF OPERATING PARA-METERS ON TH E PERFORMANCE OF THE CGA PROCESS

5.1 OPERATING PARAMETERS

It was needed to perform a sensitivity analysis on the CGA process in order to determine the conditions needed for optimal performance. The following parameters were investiga, ed to determine their effect ϕ n the efficiency of the process:

- The coal-oil phase and slurry were contacted at different periods of time.
- The mode of contacting these phases, whereas the following modes were investigated: shaking, stirring and the rolling bottle technique.
- The means of separating the coal-oil agglomerates from the slurry. whereas the following two methods were investigated: scraping and sieving.
- The amounts of coal, oil and water were altered within the ratios: c al:ore, coal:oil and Water:ore in order to determine the importance of each component to the efficiency of the CGA process.
- A preliminary investigations into the type of oil used.
- An investigation into the effect of mineralogy of the ore on the CGA process.

Before performing the sensitivity analysis on the CGA process, it is needed to stipulate the basic/standard experimental conditions, which were altered within each sensitivity parameter investigated at the time. The standard conditions were determined in Chapter 4 (Exp. 4.2, Appendix A) and are as follows:

- 4.17 g synthetic gold ore $(7g/t)$
- 4.17 g coal
- I m! oil (Ethane Oleate)
- 125 ml distilled water
- 10 ml of a ! g/IOO ml solution of collector. nl. potassium amyl xhantate (PAX)
- contact was done by shaking on a laboratory scale mechanical shaker for 50 minutes,
- separation of the coal-oil agglomerates from the ore slurry was done by scraping the floatable agglomerates from the surface of the aqueous medium.
- Samples were dried overnight at 80 $^{\circ}$ C and
- ashed at 700 \degree C for 7 hours.

5.2 EXPERIMENTAL PROCEDURES

5.2.1 Contact times

Batch experiments were performed under the same standard conditions, as determined in Chapter 4. whereas the periods of contacting the coal-oil agglomerates and ore slurries were altered in order to determine the effect of this parameter on the gold recoveries. Contact times such as 10 minutes, 20 minutes and 30 minutes were chosen for the 3 different batch experiments (Experiment 5.1, Appendix A) that were performed. As no variables other than the time of contacting the two phases were changed, it was possible to determine the minimum amount of time needed for effective and optimal operation.

5.2.2 Mode of Contact

As various methods exist whereby the coal-oil agglomerates and ore slurry could be contacted, it was decided to investigate the effect of each on the percentage gold recoveries. The various modes of contact selected for this study were: shaking, stirring and the rolling bottle technique. An advantage of this specific study was that each method was investigated by using the exact same bottles used for the standard procedures (Chapter 4), which meant that literally all other variables were kept constant.

52.2.1 *Shaking*

The use vf a laboratory scale mechanical shaker was not a new concept, as this was the same instrument used for contacting the two phases in the standard experiment. Therefore, this experiment was not repeated, but the results rather re-used for the purpose of comparison (Experiment. 4.2, Appendix A).

52.2.2 *Stirring*

The stirring action was provided by a magnetic stirrer of which the magnets were approximately 3.5 cm in length. As more than one experiment was set up at once, it was vital to use stirrers of the same brand, which was done in each case. All experiments were performed at maximum stirring speed, which ensured repeatability (see Experiment 5.2, Appendix A).

5.2.2.3 *Rolling bOllle technique*

The rolling bottle technique involved the use of circular moving shafts, one stationary and one moving at maximum allowable speed, onto which the bottles. containing the same sample masses as for the standard experiment. were placed. The rolling action of the one shaft caused the stationary shaft and, hence the bottles to move at the same speed. The two shafts were set at minimum width from each other to ensure that the bottles fit comfortably. The rollers were long enough for three bottles to fit perfectly when laid down in horizontal order, which ensured that three batch tests were performed under the exact same speed (see Experiment 5.3, Appendix A).

5.2.3 Means ofseparation

Experiments were devised to investigate means of separating the coal-oil agglomerates from the ore slurry to ensure maximum separation of the coal-oil agglomerates from the ore slurry to ensure maximum recovery of the gold from the slurry. The methods investigation were: scraping and sieving. Once again the results of Experiment 4.1 (Appendix A), the standard experiment, accounts for the lse of scraping to remove the floatable coal-oil agglomerates from the surface of the aqueous slurry.

A test was then conducted whereby the CGA process was performed under standard conditions and a sieve instead of scraping was used to separate the coal-oil phase. It was found, however, that the fine size of the agglomerates and the suspension of most of the fines in the slurry led to less successful separation of the agglomerates by utilising this method. Therefore, the samples were not analysed. as it was seen that a large percentagc of agglomerates were lost and those that were recovered contained a large amount of silica (synthetic ore mixture), which was attached to the agglomerates as the slurry was passed through the sieve. The analysis of the coal-oil agglomerates would. therefore. not be a true representation of the gold recovered into the coal-oil phase only. It is, however. important to note that sieving was not the most appropriate means of separating the coaloil agglomerates at this scale of operation. but with further adjustments and a larger scaleup it might be more successful!. Furthermore, depending on the size of the agglomerates (see Equation 3.12), the method of separation might change, as larger agglomerates will require the use of a sieve. For the purposes of this study. using a sieve was found to be the less effective choice of separation under the conditions specified earlier. but it must be kept in mind that alteration of other conditions. such as oil type etc. might alter the

size of the agglomerates, which could effect the size of the agglomerates and hence, the means of separation by requiring a sieve instead.

5.2.4 Alteration of the experimental ratios

It was decided to determine what effect each essential component i.e. coal, oil or water had on the efficiency of the CGA process by altering the amounts used within the experimental ratios determined in the previous chapter: coal:ore, coal:oil and water:ore.

5.2.4.1 *The coal.·ore ratio*

The coal: ore ratio was altered by increasing the amount of coal from a ratio of 1:1 to 1.5:1 (by mass). As the amount of coal was increased by half, it was essential that enough oil was added to maintain the coal:oil ratio (100:20, by volume). This change involved the use of 6.255g of coal and 1.5 m! of oil (ethane oleate) instead of 4.17 g coal and 1 mi oil. Furthermore, all other parameters were kept at the standard procedure followed in Chapter 4. (see Experiment. 5.4, Appendix A)

52.4.2 *The coal. oil ratio*

The coal:oil ratio was increased from 100:20 to 100:40 (by volume), therefore doubling the volume of oil used in each batch experiment from I ml/4. I7g coal to 2 ml/4. I7g coal and performing the remainder of the experiment under standard conditions. (see Experiment 5.5, Appendix A)

5.2.4.3 *The water:ore ratio*

It was attempted in this investigation to half the amount of water used. which was done by altering the water:ore ratio from 135 ml water: 4.17 g ore to 75 ml water: 4.17 g ore. The remainder of the experiment was, however, performed under the standard conditions. (see Experiment 5.6, Appendix A)

5,2.5 The addition of a collector

The significance of the collector addition to the CGA process was investigated by altering the volume and concentration of the collector in separate experiments. The collector is a chemical which is utilised to alter the surface properties of the components involved in the experiment by intensifying the hydrophobic properties of the hydrophobic components (coal-oil agglomerates and gold) and thereby increasing the hydrophobic attraction and the floatability of the final product (coal-oil-gold agglomerates). Therefore, the collector facilitates the separation of "cleaner/purer" coal-oil-gold agglomerates by floating them easier.

The collector used, potassium amyl xhantate (PAX) , was increased in two instances, i.e.:

- by increasing the volume of PAX from 10 ml to 20 ml per single batch experiment (see Experiment 5.7, Appendix A), or
- by increasing the concentration of PAX from $1g/100$ ml to $2g/100$ ml per single batch experiment (see Experiment 5.8, Appendix A).

The remainder of the variables and conditions were kept constant at the standard conditions specified earlier.

5.2.6 Oil type

It was in question whether the type of oil used in the experimental procedure might influence the percentage gold recoveries, hence an investigation was launched into the effect of oil type on gold recoveries. The oil used in the standard experiment was specified as Ethane Oleate, which was an oil chosen from the chemical store to start off the experimental programme. This oil was, therefore, not chosen for any other reason than for its availability. In order to complete the investigation, an industrial Caltex oil named Rando oil, HD 68, was used in the same amount and under the exact same standard conditions as for the standard experiment. (see Experiment 5.9, Appendix A)

5.2.7 The mineralogy of *the ore*

Other minerals within the ore structure can influence the liberation of gold.

- Firstly, gold particles could be associated with certain minerals, rendering them to be less hydrophobic and unreachable by the agglomera.es, caused by the fact that they are weaved into the matrix of these minerals. Therefoie, it is important to analyse an ore sample to determine which amount of gold is free/liberated or dissolved in order to decide what the feasibility would be of performing CGA on such an ore.
- Secondly, there is always the possibility of an ore sample containing hydrophobic minerals, other than gold, which could be recovered by the agglomerates as well. It was therefore aimed to investigate the possibility of such a phenomenon occurring and hence, determining the effect it has on the CGA process efficiency. Although mentioned in this chapter, this specific investigation was performed in the next chapter, whereby the CGA process was performed under optimised conditions on a real ore sample and XRD analysis performed on the untreated and pre-treated ore and coal samples to determine which minerals other than gold was recovered into the coal-vil phase (see Experiment 6.16, Appendix A).

As all previous CGA experiments were performed on the synthetic ore (7 g/t) , it was. therefore decided to repeat the standard experiment. utilising an industrial ore sample for the purpose of determining the effect of the mineralogy of the ore on the percentage gold recoveries. An ore sample containing \pm 4g gold/ton ore was received from the Western Area Gold Mine in South Africa and used for this investigation. (see Experiment 5.10a, Appendix A)

A further leaching experiment was performed inside a baffled reactor, whereby 10 ϱ of the industrial ore sample was leached with a 100 ppm free CN' (cyanide) solution as potassium cyanide (KCN). The purpose of this investigation was to ascertain the percentage dissolved gold present in the sample (Experiment 5. lOb, Appendix A). Samples of the KCN solution was removed at certain time intervals (30 minutes, 6 hours, 23 hours, 27 and 29 hours) and analysed on the GTA to detennine the amount of dissolved gold that has been removed from the ore sample. The aim was to find an equilibrium value of gold that has been removed from the ore by cyanide leaching, which accounts for the amount of dissolved gold present in the ore sample. By further treatment with *aqua regia* and GTA analysis of some additional ore samples, the total amount of gold present in the ore sample was measured, as *aqua regia* is most likely to dissolve both free and dissolved gold. The following equation was then used to calculate the quantity of free gold originally present in the industrial ore sample:

$$
[Au]_{total} = [Au]_{dissolved} + [Au]_{free}
$$
...(5.1)

where:
$$
[Au]_{total} = total gold loading on the gold ore (g/t)
$$

$$
[Au]_{dissolved} = dissolved gold concentration in the ore (g/t)
$$

$$
[Au]_{free} = free/liberated gold concentration in the ore (g/t)
$$

Finally the percentage of dissolved gold and free gold was calculated by the following equations:

$$
Percentage Free Gold = ([Au]_{free} / [Au]_{total}) \times 100 \qquad \qquad \dots (5.2)
$$

Percentage Dis solved Gold =
$$
([Au]_{dissolved} / [Au]_{total}) \times 100
$$
 ...(5.3)

The above investigation into the effect of mineralogy of the ore on the CGA process was strictly a preliminary investigation, which was completed and researched deeper in Chapter 6 (see section 6.4).

5.3 RESULTS AND DISCUSSION

Table 5.1 depicts a summary of the results obtained after performing the sensitivity analysis, which are arranged in order of increasing gold recovery. Following is the discussion of each result and possible causes of the recoveries found under the specified conditions. The sensitivity analysis was concluded by comparing each *new* result to that found in the standard experiment, as this was the basis on which the analysis was performed.

It was further attempted to apply the findings of the theoretical model (Equation 3.9, Section 3.5, Chapter 3) to the applicable results so as to ascertain possible theoretical explanations for each.

5.3.1 Overall application of the thermodynamic stability model

As mostly one oil type (Ethane Oleate) was utilised to perform the experiments and the investigation of oil type was strictly preliminary, it would be safe to state that the percentage gold recoveries with respect to the thennodynamical stability was mostly dependent on the value of k i.e. the ratio of coal-oil agglomerate to gold particle radii. Furthennore, **it** should be noted that the diameter of the gold particles used in the synthetic ore and hence, this study was $44 \mu m$, which gives an indication of the sizes of the agglomerates needed for feasible operation. It was, however, found that the sizes of the agglomerates fonned during experimentation ranged from a minimum diameter of approximately I mm to a maximum agglomerate diameter of approximately 4 mm. With the information at hand it was possible to determine what the *actual* minimum and maximum values of k was during experimentation, which could then be compared to the theoretical k_{min} and k_{max} in order to find if the CGA was performing in the allowable range for thermodynamic stability.

Calculation of the actual k_{min} and k_{max} values:

 \overline{k} = $\overline{r_{\text{coal-oul-gglomerates}}/r_{\text{gold particles}}}$

To find the actual value of k_{min} , the minimum diameter of the agglomerates (1 mm = 1×10^3 µm) was divided by the actual diameter of the gold particles used for the experiments:

$$
Actual k_{minimum} = 1 \times 10^3 / 44
$$

$$
= 22.7
$$

The actual value of k_{max} , was, however, calculated in the same manner, utilising 4.4 mm instead of 4 mm as the maximum size of the agglomerates in order to obtain a answer that was round off. As these are rough estimates of the actual diameters of the agglomerates, it would not make a difference:

$$
Actual k_{\text{maximum}} = 4.4 \times 10^3 / 44
$$

$$
= 100
$$

It is, however, seen from Chapter 3, section 3.5 that the ideal range for k to ensure thermodynamic stability was at $k_{min} = 1$ and $k_{max} = \infty$. Therefore, it was found that the actual experimental values of k was found to be well within range, as the actual k_{min} is not less than the theoretical value (22.7 > 1) and the actual k_{max} was very much below the theoretical value $(100 < \infty)$.

To confirm thermodynamic stability, it was, however, imperative that the actual threephase contact angle measured through the oil on the gold surface in the presence of water (θ), be below the maximum allowable three-phase contact angles(θ_{max}) for the actual k_{min} and k_{max} found from experimental findings. Therefore, θ_{maximum} was calculated for each of the k_{min} = 22.7 and k_{max} = 100 values. This was done by plotting F'_{II-1} versus θ for each consecutive actual minimum and maximum value of k. as seen from Figures 5.2 and 5.3. The θ_{max} was in effect the θ_{critical} value for each value of k and was calculated as the point where the F'_{II-1} line of each k intercepts with the 0-axis. From the calculations, it was found that the maximum allowable three-phase contact angle, θ_{maximum} for each of the actual minimum and maximum values of k to ensure thermodynamic stability was as follows:

- for the actual $k_{\text{minimum}} = 22.7$, the maximum three-phase contact angle. θ_{maximum} was calculated to be 88.3°
- and for the actual $k_{\text{maximum}} = 100$, the maximum three-phase contact angle. θ_{max} was calculated as 89.62°.

The above statements imply that the CGA process would, under standard conditions, be thermodynamically stable if the three-phase contact angle, θ , which is measured through the oil (Ethane Oleate) in the presence of water on the gold surface should not exceed

88.3° for the small agglomerates and should not exceed 89.62° for the larger agglomerates. Therefore, it was vital to measure the actual three-phase contact angle, θ in order to confirm thermodynamic stability, whereas the measurement was performed by utilising a moving microscope. A gold plate was put into a square see-through vial and topped up with water after which a microsyringe was used to carefully place an oil droplet (Ethane Oleate) of volume 30 μ l onto the gold surface. The dimensions of the drop was then measured and the three-phase contact angle calculated from the following equation:

$$
\tan \theta/2 = h/r \tag{5.1}
$$

The actual three-phase contact angle (θ) , measured through an Ethane Oleate oil droplet on the surface of gold in the presence of water was measured and calculated as 64.6° (sec Experiment 6.19, Appendix A). It is, therefore, seen that the actual value of θ for Ethane Oleate is well below the maximum allowable values as mentioned above. which confirms that the CGA process is indeed thermodynamically stable as performed in the experiments perfomed while utilising Ethane Oleate. as was all the experiments except for Exp 5.9.

Therefore, it could be concluded that the CGA process was found to be well within the thermodynamic stable range and based on this theoretical analysis. the rercentage gold recoveries should be promising. It was, however, stated in Chapter 3, section 3.5, that this is not the only factor to be considered when dealing with percentage gold recoveries. Furthermore, all experiments will be discussed in the light of the other factors (see Figure 3.5, Chapter 3) as having an effect on gold recoveries.

5.3.2 Contact times

Refer to Figure 5.1 for a plot of percentage gold recoveries versus *contact times* in minutes. It is seen that the percentage gold recoveries by the CGA process is at a maximum when the coal-oil agglomerates and ore is contacted for periods exceeding 50 minutes, whereas a contact time of 50 minutes is the minimum time needed for optimum recoveries. The contact time is a measure of the time needed for maximum contact between the agglomerates and gold particles. The larger contact time increases the probability of a gold particle "bumping" into the surface of the agglomerates and hence, increases the chance of being wetted by the oil, which explains the higher recoveries as more gold particles were attached to the agglomerates as seen from the better recoveries after increasing the contact time sporadically. (see Ex_1 . 5.1, Appendix A for the experimental conditions and raw data). The findings from the results confirm the importance of the *contact time* to maximise transfer of gold particles into the coal-oil phase (see Figure 3.5, Chapter 3).

5.3.3 Mode of Contact

The standard experiment in Table 5.1 represents the gold recoveries found after using the shaker as mode of contacting the coal-oil agglomerates with the ore slurry. whereas the use of a stirrer and/or rolling bottle technique is marked clearly in the summary of results in Table 5.1 and 5.2. It is clear form Table 5.2 that the higher gold recoveries were found after contacting the coal-oil phase and gold ore by means of the stirrer, whereas the contrary was found after utilising the rolling bottle technique and shaker.

Although the theoretical analysis has shown that larger agglomerates favours the thermodvnamic stability and supposedly the successful recovery of gold, it is hereby proven that a trade-off of other practical factors inhibits the theoretical tendency.

Table 5.2 - Results on the investigation of the mode of contacting the coal-oil agglomerates and ore

The lower gold recoveries are, therefore caused by other overbearing factors which is mentioned in Figure 3.5, Chapter 3, and the results obtained serve as confirmation of their influence. The most evident factors influencing maximum gold transfer into the coal-oil phase were, however, identified as the following (see Figure 3.5, Chapter 3):

- This specific mode of contact (rolling bottle technique) does not allow the necessary *motion* for optimal contact between the coal-oil agglomerate particles and gold particles, therefore the gold transfer into the coal-oil phase is inhibited to a certain extent, as shown in the results. This confirms that the mode/motion of contact is of imperative value for maximum gold transfer into the coal-oil phase and hence. gold recoveries, as seen in Figure 3.5, Chapter 3.
- Furthermore, the rolling action favours the formation of *larger agglomerates* and hence, provides the gold particles with a smaller available agglomerate surface area to attach themselves and confirms the importance of the coal-oil agglomerate size for maximum gold transfer into this phase.

By examination it was seen that the average size of the agglomerates yielded after performing the rolling bottle experiment were so large that they could not be removed by scraping only, as a sieve was utilised for completion of the separation procedure. Therefore, confirming the importance of selecting the appropriate means of separation required to separate the various *sizes of the coal-oil agglomerates* and hence, assuring efficient separation (see Figure 3.5, Chapter 3).

As for shaking, it was found that the *shaking action* was not sufficient for maximum gold transfer into the coal-oil phase, whereas the contrary was found after using a stirrer. The stirring action provided a dual advantage to the CGA process in that the motion was sufficient to keep both the agglomerate particles and gold particles in *suspension,* whilst providing a *constant stirring motion,* and thereby increasing the probability of a gold particle being attached to an agglomerate and hence, assuring maximum gold transfer into the coal-oil phase. The second advantage is that the stirring action kept the size of the agglomerates to a minimum and hence, providing the *maximum available surface area* at a maximum. (note that the minimum size of the agglomerates was still above or equal to the required size to yield a minimum of 22.4 for the value ofk). Furthermore, the *smaller agglomerates* favoured the efficiency of separation \ ia scraping. It is therefore, concluded that all three vital factors influencing percentage gold recoveries (see Figure 3.5) has shown favouratism toward stirring as mode of contact in some or other way. explaining why this means of contact yielded the better gold recoveries.

5.3.4 Alteration ofthe coal:ore ratio

It is seen from Table 5.1 that gold recoveries were increased when the coal:orc ratio was altered in such a way to increase the amount of coal by half, whereas an average increase of 3.55%, from 37.4 to 41.95 % was calculated.

As there exists a fixed relationship between coal and oil as well. it is in effect not only the amount of coal that was increased, but rather the amount of agglomerates that were increased by half. Coal is of great significance to the CGA process. as the presence of this inexpensive material facilitates the easiness with which the oil-pha5e and hence. the active ingredient/'gold carrier', is separated from the ore slurry. Furthermore, the use of the oil in the form of coal-oil agglomerates simplifies the separation of the gold particles, as both coal and oil is hydrocarbon materials which burns away to ash. leaving pure gold particles as the product.

As the importance of coal is understood, it is obvious that an increase in the amount of agglomerates used would have a positive influence on gold recoveries, as a *larger overall surface area* is available for gold particles to attach themselves, therefore, enhancing maximum gold transfer into the coal-oil phase to such an extent that an increase in recoveries were shown.

5.3.5 Alteration ofthe coal:oil ratio

It is seen from Table 5.1 that gold recoveries were decreased form 38.4% to 12.07%, with an increase in the amount of oil used. As the amount of oil was doubled, it meant that the agglomerates were prepared, using the same amount of coal and double the volume of oil. This lead to the formation of *unstable agglomerates.* w:.ich caused difficulty in the s eparation (efficiency) (see Figure 3.5, Chapter 3) of the oil phase, as the very unstable oil agglomerates were in more of a liquid than a stable solid form, with many coal particles and oil droplets drifting in suspension. Therefore, the importance of coal in the correct ratio was emphasised in this investigation. As separation was not as complete and effective and agglomerates were not as stable, it could be said that the low results were firstly caused by poor contact and secondly by poor separation of the oil phase.

5.3.6 Alteration of the water:ore ratio

According to Table 5.1 it is seen that the average percentage gold recoveries have increased form 38.4% to 49.45% after the amount of water used to prepare the ore slurry was decreased by approximately half.

By this time it is basic knowledge to the reader that the CGA process is based on the hydrophobicity of gold, coal and oil, which causes these main components to group together when in an aqueous medium. Therefore, water is imperative to the CGA process and besides being the driving force for the gold. coal and oil to agglomerate. it further serves as the medium in which the gold *travels* to reach the agglomerates. By decreasing

the amount of water, however, it was found that gold recoveries had increased by at least II%, which indicates that less water is in effect less distance for the gold particles to travel. Therefore, it could be said that the *motion of contact* was enhanced by decreasing the total amount of water in the system, as the same motion i.e. shaking was more effective, thereby causing a positive reaction to the maximum gold transfer into the coaloil phase and hence, the percentage gold recoveries.

5.3.7 The addition of a collector

Refer to Table 5.3, below for a summary of the results obtained after simultaneously doubling the volume and concentration of collector used it. the CGA experiments.

Table 5.3 - Results after altering the concentration and volume of collector (PAX)

The *addition ofa collector* (such as PAX) had a dual advantage to the CGA process and the better gold recoveries were as a result the positive influence of the both these advantages:

- \bullet Firstly, by enhancing the hydrophobicity of the gold and coal particles. It increased the probability of gold particles being entrapped into the agglomerates, thereby increasing possibly increasing the maximum Au transfer an i
- secondly, by enhancing the floatability of the coal-oil-gold agglomerates, it enables the effectiveness of separation and hence, gold recoveries are increased.
5.3.8 **Oil** type

As seen from Table 5.1, the preliminary investigation on a different type of oil has shown that the oil type has no significant effect on the percentage gold recoveries. As this was an early stage investigation, a further investigation was planned and is discussed in the next chapter (see Experiment 5.9, Appendix A). As the use of a different oil type alters the actual values of θ and γ_{23} , it should be kept in mind that these factors might influence percentage gold recoveries as a result of an alteration in the thermodvnamic stability of the system. See section 6.2, Chapter 6, for a detailed study into the effect of various oil types on the percentage gold recoveries.

5.3.9 The mineralogy of the ore

The effect of mineralogy of the ore on the CGA process was performed. which lead to a total decrease of 9% in average gold recoveries

when an industrial ore was used instead of the synthetic ore mixture. A summary of the results is shown in Table 5.4, below.

Table 5.4 - Preliminary results after performing the CGA experiment on an industrial ore as compared to that of the synthetic ore.

As the CGA process is most suitable for ores containing free/liberated gold particles, the industrial ore was subjected to cyanide leaching (Experimem 5.10b Appendix Ai to determine the amount of dissolved gold. The gold in the industrial ore could be characterised as follows (see Exp. 5.lOa and 5.1 Ob. Appendix A):

- 92.96 % was liberated/free gold and
- 7.04% was dissolved gold

From the above percentage free gold it is concluded that the industrial ore sample contained the sufficient amount of liberated gold to render it feasible for the CGA process. It was, however, found that gold recoveries for this ore was much lower than for the synthetic ore and other factors had to play a part. At this stage of the investigation it was speculated that the *mineralogy of the ore* could cause lower gold recoveries as a result of a decrease in the maximum gold transfer into the oil phase, cuased by either the entrapment of gold into the mineral matrix or by the fact that some minerals were transferred into the coal-oil phase, leaving "less space" for gold to attach. Therefore, further investigations into the effect of the mineralogy of LIe ore was conducted in the next chapter for completion of this study (see section 6.4, Chapter 6).

S.4 SUMMARY

lt is seen from the results on the sensitivity analysis (see Table 5.1) that the percentage gold recoveries by the CGA process was influenced positively by factors such as:

- An increased amount of coal
- An increased volume and concentration of PAX
- A decreased amount of water to prepare the slurry
- Contact by using the stirrer
- Contact times exceeding 50 minutes

Furthermore, it was found that factors such as an increased amount of oil and contact by the rolling bottle technique decreases percentage gold recoveries. As the investigations concerning the type of oil and mineralogy of the ore are preliminary studies, no conclusions were made on these subjects at such an early stage. Finally. the means of separation to be the most suitable for the abovementioned experimental conditions was

found to be scraping, whereas in certain cases the size of the agglomerates were to large and a sieve was used for thorough separation of the agglomerates.

Therefore, the conditions for optimal performance were found by lumping all the factors which caused gold recovery percentages to increase and are characterised by the following experimental ratios:

- the coal:oil ratio remained 100:20 by volume,
- \bullet the coal: ore ratio remained 1:1 by mass,
- the water:ore ratio was *halved* from 135 ml/4.17 g ore to 75 ml/4.17 g ore,
- the concentration of collector was doubled from $1g/100$ ml to $2g/100$ ml and finally
- \bullet the volume of collector was doubled from 10 to 20 ml of the above solution.

The optimal experimental conditions were therefore stipulated as follows:

- 4.17 g synthetic ore, \overline{a}
- 4.17 g coal, \overline{a}
- \overline{a} I ml Ethane Oleate as oil,
- 20 ml of a $2g/100$ ml solution of potassium amyl xanthate (PAX) as collector. ÷,
- 75 ml of distilled water, \overline{a}
- contact was done by stirring for 50 minutes, \overline{a}
- separation was done by either scraping or sieving. depending on the size of the \overline{a} agglomerates.
- all samples were dried overnight at 80 $^{\circ}$ and ashed for 7 hours at 70 $^{\circ}$.

All further tests were performed under the optimised conditions, which will be discussed in the next chapter.

Figure 5.1 - Percentage gold recoveries versus the contact times in minutes (Refer to Experiment 5.1, Appendix A)

The Three-phase contact angle (θ)

Figure 5.2 - F''_{II-I} vs. θ for actual $k_{min} = 22.7$, which states that the minimum value of $r_{\text{caol-oil agglomerates}} = 1 \text{mm}$, which was the minimum size of agglomerates yielded after performing the actual CGA experiments. $(\theta_{\text{critical}} = 88.3^{\circ})$

The Three-phase contact angle (θ)

Figure 5.3 - F"_{II-I} vs. θ for actual k_{max} = 100, which states that the maximum value of $r_{\text{coal-oil}}$ agglomerates = 4.4mm which was the maximum size of agglomerates yielded after performing the actual CGA experiments. ($\theta_{\text{cntical}} = 89.62$ °)

CHAPTER 6

EVALUATION OF THE CGA PROCESS UNDER OPTIMAL CONDITIONS

6.1 CGA UNDER OPTIMAL CONDITIONS

As mentioned in the previous chapter, the conditions for most efficient performance of the CGA process was a compilation of the parameters that favoured maximal gold recoveries. A summary of these are as follows: a contact period of at least 50 minutes. contact by stirring, the addition of a collector and a decreased amount of water was used to prepare the slurry. According to the results of this sensitivity analysis it implied that the following alterations had to be made to the conditions of the standard experiment: the amount of water was halved from 135 ml to 75 ml, the collector (PAX) concentration and volume was doubled to 20 ml of a $2g/100$ ml solution and finally all the components were contacted by stirring for 50 minutes.

A bottle with a 200 ml volume (the same as used in the standard experiment) was used to perform the experiments and all the materials were added in the amounts as stipulated under optimal conditions in the last section of the previous chapter (see Experiment 6.1. Appendix A). Analysis were performed by either the Graphite Tube Atomiser (GTA) or the Inductively Coupled Plasma (ICP) and are tabulated below. See Appendix A, SAMPLE ANALYSIS for more information on the OTA and/or ICP.

It is seen from Table 6.1 that the overall gold recoveries, as analysed by the GTA had increased from 38.4 to 44%. Although an improvement had been observed, it was still a very low result and for this reason it was sought to utilise an alternative instrument, i.e. the lCP, for analysis. Furthermore, another experiment was performed under optimal conditions, whereby the mass balance was checked by analysing the treated ore sample (C_1) as well, see Experiment 6.2, Appendix A. From these results the GTA gave much lower, but still consistent results (32.9%) and the mass balance was incomplete, which confirmed the need of utilising another instrument for analysis. The OTA used in these analyses is a very old instrument which was starting to be problematic for further analysis, as seen from the results of Experiment 6.2. All samples were treated in the exact same manner as before i.e. drying, ashing and dissolving them into *aqua regia,* the only difference being the instrumental analysis.

As seen from Table 6.1, the percentage gold recoveries by lCP analysis had increased to a promising 84.5% and by analysing the treated ore sample, it was seen that the mass balance had mostly been completed, as 12% gold remained in the treated ore. For a complete mass balance the total amount of gold in the system should equal that which had been recovered in the coal-oil phase, added to that which had remained in the treated ore and entails that the addition of the percentage gold of each phase should equal 100% ,

which is true in our case $(84.5\% + 15.75\% = 100.5\%)$, see Exp. 6.3, Appendix A. It could, therefore, be concluded that the low results in previous experiments were rather instrumental based than caused by human error. It is not the aim of this exercise to present an explanation as to why the ICP analysis yielded the better results, but rather to utilise the information to the advantage of this study. Needless to say, all subsequent analyses were performed on the ICP, as the results were fairly reproducible with an error margin of less than 5%. Although some of the results in this chapter was obtained by GTA analysis, these are mentioned for the sake of completeness, but emphasis is placed on the better results by ICP analysis.

As the sensitivity analysis had been completed and basic optimal conditions were specified and proven to result into gold recoveries abov< 80%, it was at this point possible to evaluate the CGA process. Subsequent investigations, therefore were aimed at evaluating the performance of the optimised CGA process under certain chosen conditions, as will be discussed within the bulk of this chapter.

6.2 THE OPTIMISED CGA PROCESS ON VARIOUS **OIL** TYPES

6.2.1 Experimental procedures and Results

The CGA process was performed on various industrial oil types to ascertain the effect of these on the percentage gold recoveries, as all past experiments were performed on ethane oleate, which was an oil taken from the chemical store to start off the experimental programme. The oils used for this investigation were supplied by Caltex and is known as base oils i.e. oils which has not undergone any extensive chemical treatment. Refer to Table 6.2 for the chemical analysis of these oils, as performed by Caltex. It is seen that these oils are very similar with respect to chemical composition. whereas each differs mainly with respect to viscosity and colour.

Table 6.2 - Chemical analysis of the oils supplied by Caltex

Table 6.3 - Gold recoveries by the CGA process found for different oil types, analysis by either ICP or GTA

The CGA experiment was performed under optimised conditions, each time using a different oil type. The average gold recoveries obtained by each oil type is shown in Figure 6.3. It is, however, seen that analysis were performed by both the ICP and GTA. The results from the GTA analysis were disregarded, as these were very low and mass balances were incomplete, hence the results from the ICP analysis were used in further discussions.

From the results it is seen that the CGA process yielded the best results when utilising ethane oleate as the agglomerant, followed by the 150 Solvent Neutral (SN) Caltex oil, 450 SN oil and finally the 100 SN Caltex oil.

6.2.2 Discussion ofthe Results

As illustrated in Figure 3.5, Chapter 3, the percentage gold recoveries were established/affected by the thermodvnamic stability, maximum gold transfer and the efficiency of separation. Therefore the results shown in Figure 6.3 were dictated by each of these factors to a certain extent. Each of these factors were, however. found to be influenced by a set of different variables/operating parameters. as scen in Figure 3.5. Therefore. a checklist was drawn up from the diagram (Fig 3.5) for each of the oils used in order to acknowledge the influence of these factors and variables on the percentage gold recoveries and hence, serve as possible explanation for the recoveries found.

6.2.2.1 *Predictions according to the thermodynamic stability model*

It has been mentioned at an earlier stage that the oil type influenced the thermodynamic stability of the CGA process in that the three-phase contact angle (θ) and interfacial tension (γ_{23}) for each varied. Therefore, it was vital to measure each for the purpose of comparing the oil types with iespect to the thermodynamic stability. The three-phase contact angles (θ) for each oil on the gold surface in the presence of water was measured in the same marmer as in Chapter 5. section 5.3.1. by utilising the moving microscope and

are recorded in Table 6.4 (see Experiment 6.19, Appendix A). Note that the following abbreviations were used: EO - Ethane Oleate, 100 SN - 100 Solvent Neutral Caltex oil, 150 SN - 150 Solvent Neutral Caltex oil and 450 SN - 450 Solvent Neutral Caltex oil.

The interfacial tension between each oil and water was calculated from the following equation (Glasstone *et al.,* 1981):

$$
\gamma_{AB} = \gamma_B - \gamma_A \qquad \qquad \dots (6.1)
$$

where: γ_{AB} YA Y8 = the interfacial tension between the oil and water (J/m^2) ⁼ the surface tension of water/oil *(l/m²)* = the surface tension of the oil, water (J/m^2)

Since surface (and interfacial) tensions are always positive, it follows that the interfacial tension is less than that of the larger of the two surface tensions. This is because the attraction across the interface, between the molecules of one liquid and those of the other. tends to reduce the inward pull of the molecules in the surface by those of the same kind. The greater the molecular attraction between the two liquids. the iower the interfacial tension. Before the calculation could be done. it was vital to determine the surface tensions of each oil and this was measured by utilising the *capillary rise* method.

The *capillary rise* method operates on the basis of comparing the capillary rise of the liquid in question with that of a standard liquid such as water, of known surface tension. If *h* is the rise of the liquid of surface tension γ and density ρ in a given capillary tube. and h_0 is the rise of a liquid in the same tube of a standard liquid. i.e. water, of density ρ_0 and surface tension γ_0 , it follows from equation (6.2) that:

$$
\gamma/\gamma_0 = h\rho/h_0\rho_0 \qquad \qquad \dots (6.2)
$$

Refer to Experiment 6.20, Appendix A, for all the measurements taken and calculations to obtain the oiVwater interfacial tension for each oil utilised in the experiments. Table 6.4, below depicts all the interfacial tensions, as were measured and calculated. Furthermore, it was seen that the sizes of the agglomerates varied with each different oil type used, whereas an estimation as to the average radii was made and is shown in Figure 6.4.

The agglomerates were, however divided into three "size classes" as follows:

- small agglomerates average diameter of 1.5mm
- medium agglomerates average diameter of 2 mm
- large agglomerates average diameter of 3 mm

As seen from Table 6.4, both the non-dimensionalised free energy (F''_{H-I}) and total free energy (F'_{II-1}) was calculated by utilising the equations that were derived earlier (Chapter 3) It is known that the oil yielding the lowest F''_{II-I} is thermodynamically the most stable and should therefore yield the highest gold recoveries, whereas it was then possible to predict which oil type would perform the best. According to the calculations of $F'_{H,I}$ the theoretical rating of the each oil is recorded in Table 6.4, thus predicting which oil type should yield the better gold recoveries. It was, however found that the actual performance of each oil with respect to actual gold recoveries were vastly different than predicted, as indicated in the last two rows of Table 6.4. Therefore, it was suspected that the other factors influencing gold recoveries had to be taken into consideration before any further prediction regarding the gold recoveries were to be made.

6.2.2.2 *Influence ofotherfactors on gold recoveries*

The *other factors* have been identified earlier as the maximum gold transfer of gold particles into the coal-oil phase and the separation efficiency of the agglomerates from the ore slurry, which are each individually influenced by certain variables. as seen in Tablc 6.5 and 6.6 (see Figure 3.5, Chapter 3). It is obvious from the results and predictions that the effect of these parameters were vital.

As all the experiments were performed under optimised conditions. it is seen from Tables 6.5 and 6.6 that the only variables influenced by the vary:ng the oil types. concerning both maximum gold transfer and separation efficiency, were the sizes of the agglomerates. According to the theoretical study performed in Chapter 3. section 3.5. both the maximum gold transfer and separation efficiency and hence, the gold recoveries were favoured by agglomerates of a small size:

• As the *smaller size agglomerates* provide a larger surface area for gold particles to attach, it is predicted that the maximum gold transfer into the coal-oil phase would take place under these conditions, thus causing increased gold recoveries.

• Furthermore, it was found that the separation efficiency was favoured by *smaller agglomerates* if separation was performed by scraping, as the *addition ofa collector* enhances the floatability of the agglomerates, whereas larger agglomerates were separated by utilising a sieve and the separation might be less efficient.

Table 6.5 - The checklist of variables **influencing the MAXIMUM GOLD TRANSFER of gold particles into the coal-oil phase.**

Table 6.6 - The checklist of variables **influencing the SEPARATION EFFICIENCY of coal-oil agglomerates from the ore slurry.**

By applying the above principles and utilising the information gained from performing the experiments, it was possible to predict which oil type would, according to the maximum gold transfer and separation efficiency, yield the better results.

It is seen from Tables 6.5 and 6.6 that the oil yielding the smallest agglomerates, i.e. ethane oleate should yield the best gold recoveries, followed by either the 150 SN or 450 SN Caltex oil, which both formed medium sized agglomerates and finally the 100 SN Caltex oil which formed very large agglomerates should yield the lowest gold recoveries.

All the predicted and actual ratings of which oil type should perform best were, however, summarised in Table 6.7, below:

Table 6.7 - The predicted and actual ratings of the performance of each oil type with respect to gold recoveries obtained by the CGA process.

It is seen from the above table that the actual performance of each oil type was closest to what was predicted by the maximum gold transfer and separation efficiency *models.* therefore, confirming that the influence of these factors on the percentage pold recoveries outweighed the influence of the thermodynamic stability. As both the 150 SN and 450 SN Caltex oils formed medium sized agglomerates. it was not possible to utilise the maximum Au transfer and separation efficiency *models* to predict the exact performance of each with respect to gold recoveries.

It could then be concluded that valuable predictions as to the performance of the CGA process could be made by utilising the thermodynamic stability model in conjunction with the maximum gold transfer and separation efficiency factors. Furthermore, it was vital to take the influence of each into account when explaining typical gold recoveries by the CGA process.

6.3. RECYCLING

6.3.1 Recycling **of**the ore phase

The coal-oil agglomerates (A_1) were contacted with and cre sample (C_1) after which it was separated and the ore contacted with a "fresh" coal-oil sample (A_2) of the same quantity as before (see Experiment 6.11, Appendix A). Both the coal-oil agglomerate (A_1) and Az) samples were analysed for gold. The first batch of agglomerates recovered and average of 37.9% of the gold from the ore phase and the second batch of agglomerates a further 4.25% of the gold. A further experiment was performed and all three samples: A_1 , A_2 and C_1 , were analysed in order to check the mass balance (see Experiment 6.12, Appendix A). From these results it was. however, found that 32.22% gold was recovered in the first batch of agglomerates and a further 4.27% in the second batch of agglomerates, which is very close to the previous results. Furthermore. it was found that a low 0.93% gold remained in the ore sample (C_i) , which was an unrealistic low value and was disregarded, as the GTA has proven to yield incomplete mass balances.

In conclusion, it is seen from the low results, that the analysis was done by the GTA and although these values are low, it could still be concluded that the coal-oil agglomerates are capable of recovering gold from very low grade ores.

6.3.2 Recycling of the coal-oil phase

The CGA process was performed as usual (utilising the 7 *glt* synthetic ore sample utilising ethane oleate and contacting with a magnetic stirrer for 50 minutes) and after separation, Step 1, the agglomerates (A_1) were contacted with a "fresh" ore sample (C_{02}) , Step 2. The objective of this experiment was to find if the coal-oil phase was capable of recovering gold after being recycled.

The sketch below illustrates the procedure:

Figure 6.1 - A schematic representation of the recycling procedure of the agglomerates.

See Experiment 6.13, Appendix A, for the experimental conditions and raw data. The analysis were done on the ICP, whereby gold values for C_{01} , C_{02} , C_1 , C_2 and A_1 were actually measured and hence, values of A_1 and A_2 were obtained by calculation (see $CALCULATIONS, Appendix A)$. A set of three experiments were performed and the results are recorded in Table 6.8.

The overall percentage gold recoveries were calculated from the actual measured values $(A₁, C₀₁$ and $C₀₂$), whereas the gold recovery percentages for before and after recycling the agglomerates (Step 1&2) are calculated from the calculated A_1 and A_2 values, see CALCULATIONS, Appendix A for the all the calculations done. The average overall gold recovery was found to be slightly above 100% (100.4). which is explained by the fact that the value of C_{01} , which equals C_{02} and represents the original gold loading of the ore, was a measured average of 6 different samples and this average value of 6.22 *glt* was used throughout all the calculations. All the samples that were taken, however were representative of those used during the experiments.

Table 6.8 - Results obtained after performing the CGA test and recycling the coal-oil agglomerates (see Experiment 6.13 , Appendix A).

From the tabulated results, it is seen that the agglomerates recovered an average of 83.03% of the gold after it was contacted with the ore the first time and a further average of 75.6% gold was recovered after recycling it. Therefore. it is found that the coal/oil agglomerates had the capacity of recovering an average of only 7.44% less gold after it had been recycled once. Furthermore, it was found that the gold loading onto the coal-oil agglomerates was increased from an average of ± 6 -7 g/ton [see Experiment 6.3 and Experiment 6.7, where gold loading on the coal samples $1-4$ (A₁) was roughly 6-7.5 g/t . Appendix A] to 12-14 g/ton [see Experiment 6.13, Appendix A, where the gold loading onto the coal samples 4.5 and 6 (At), where roughly 12-14 g/ton]. This confirms that the recycling procedure is an effective operation and hence, serves as an incentive to promote the CGA process in terms of the economical viability thereof. Experiments 6.3,6.7 and 6.13, which were quoted above were all performed under the exact same optimal conditions. whereas Exp. 6.13 was taken further by recycling the coal-oil phase.

It could, therefore, be concluded that the CGA process is capable of recovering gold particles into recycled coal-oil agglomerates with the capacity of recovering only $\pm 7\%$ gold less the second time around, thus almost doubling the gold loading onto the agglomerates.

6.4 CGA **ON AN INDUSTRIAL ORE**

It has been speculated from the preliminary investigation performed in Chapter 5 (see section 5.2.7 and 5.3.9) that the mineralogy of the ore caused lower gold recoveries due to the fact that the gold particles could be entrapped within the ore matrix or that minerals other than gold was *recovered* in the coal-oil phase. It was ahempted in this investigation to perform further experiments and apply the theoretical knowledge at hand to confirm the above statements or rather explain the lower gold recoveries.

As the experiment that was performed on the industrial ore in Chapter 5 was done under standard (see Experiment 5.1 Oa, Appendix A) and not optimised conditions and analysis performed on the GTA, the CGA experiment was repeated under optimised conditions and analysis performed on the ICP (see Experiment 6.14, Appendix A). Although the results of Experiment 5. lOa have shown percentage gold recoveries to be lower, the repeat was still carried out and the results are tabulated below in Table 6.9, only presenting the results of Exp. 5.1Oa for the record. It is, however. seen that the results for CGA under optimised conditions on a synthetic ore varied from 84.5 (Experiment 6.3) to 88.7% (Experiment 6.7), whereas the experimental conditions for these two experiments were exactly the same.

Furthermore, it is found from Table 6.9 that the percentage gold recoveries yielded by the CGA process, when performed on an industrial ore sample was 1.8 to 6% lower than for that performed on the synthetic ore. The exact same industrial ore that was used in

Experiments 5.lOa and b was utilised in this experiment (Exp. 6.14) and was found to contain 93% liberated gold particles (see Experiment 5.IOb), whereas the gold particles in the synthetic ore samples was 100% liberated. Therefore, the lower recoveries by the industrial ore was found to be caused by the fact that the gold in the sample was not 100% or fully liberated. As some gold was entrapped within the ore matrix because of the *mineralogy of the ore* it caused the maximum Au transfer of gold particles into the coal-oil phase to be less and hence, lower gold recoveries were found. As the experimental conditions were optimal, utilising ethane oleate as agglomerant, the thermodynamic stabilitv and separation efficiency were performed at its optimal and the lower gold recoveries were explained by the maximum Au transfer as done in the previous statement (see Figure 3.5, Chapter 3 for all the factors and variables influencing gold recoveries).

Table 6.9 - The percentage gold recoveries obtained by repeating the CGA experiment under optimised conditions on an industrial ore sample.

Conditions and ore types used	Instrumental Analysis	$%$ Au recoveries
CGA on synthetic ore under optimal conditions (Exp. 6.3 -6.7)	ICP	$84.5 - 88.7$
CGA on industrial or under optimal conditions (Exp. 6.14)	ICP	82.7
CGA on industrial or under standard conditions (Exp. 5.10a)	GTA	27.17

Therefore, the speculation that the entrapment of gold particles into the ore matrix caused lower gold recoveries was in fact true and is confirmed. The second speculation implies that other minerals were possibly recovered into ihe coal-oil agglomerates, leaving less "surface area" for gold particles to attach and hence, lower gold recoveries. It was then decided to perform the CGA experiment on the industrial ore under optimised conditions

and recycle the coal-oil phase to find if recoveries would be lower, refer to Table 6.10 for the results, as compared to that of utilising a synthetic ore sample.

Table 6.10 - Resulting % Au recoveries by the CGA process after recycling the coal-oil phase, using either the synthetic ore or Industrial ore.

As seen from the results in Table 6.10, the gold recoveries found after recycling the coaloil agglomerates were much lower when an industrial ore sample was used, whereas a decrease of 28.47% was found after using the same coal-oil samples for the second time. as compared to the 7.44% lower recoveries after recycling with the synthetic ore. Therefore, it was decided to perform a XRD analysis on the untreated and pre-treated coal and ore samples after performing the CGA process on an industrial ore, to determine if minerals other than gold was being recovered into the coal-oil phase which could cause the lower recoveries after recycling (see Experiment 6.16, Appendix A). The results on the XRD analysis is tabulated in Table 6.11

The X-ray Diffraction (XRD) analysis is a surface based analytic method, which does not quantify the minerals on the surface. but rather qualifies those present. Therefore. it is

seen that no gold particles were detected on the surface of the samples, which is no proof that they do not exist in the matrix of the coal-oil agglomerates, which seems to be the case. However, it is seen that the coal-oil agglomerates captured some other minerals, besides gold, as quartz, pyrophyllite and chlorite etc. were found in the treated coal-oil samples and that these results merely state that the presence of other minerals could have an effect on the performance of the Coal Gold Agglomeration (CGA) process (refer to Experiment 6.16, Appendix A).

Table 6.11 - The results on the X-Ray Diffraction (XRD) analysis, illustrating which minerals were present in the relevant samples (see Experiment 6.16, Appendix A for the conditions and diffractograms).

Therefore, it was assumed that the lower results obtained after recycling the coal-oil agglomerates were caused by the other minerals *clogging* the surface of *tr.* agglomerates so that all gold particles were not able to attach effectively. In the light of the theoretical study, the thermodynamic stability and separation efficiency was stable and at optimal performance, whereas the *mineralogy of the ore* affected the maximum gold transfer of gold particles into the coal-oil phase and, hence gold recoveries in two instances:

- Firstly, the entrapment of gold particles in an ore matrix, caused by the *mineralogy of the ore* had rendered the gold particles to be less hydrophobic, causing the maximum gold transfer into the coal-oil phase and, hence the gold recovery to be less, as seen with the lower results in Table 6.9.
- Secondly, the presence of other minerals, characteristic of the *mineralogy of the ore*, was found to be detrimental to gold recoveries as some of these minerals could be attached to the coal-oil agglomerates, clouding" the surface. Therefore, causing *less agglomerate surface.* in a certain sense, for gold particles to attach and hence, minimising the maximum gold transfer of gold particles into the coal-oil agglomerates. The above statement was concluded after recycling the coal-oil agglomerates and finding gold recoveries to be much lower than when using a synthetic ore mixture, refer to Table 6.10. Furthermore, a XRD analysis of the untreated and pre-treated ore and agglomerates have shown minerals other than gold to be "picked" up by the agglomerates, thus confirming the speculations made earlier.

6.5 CGA versus **MERCURY AMALGAMATION**

It was not only the purpose of this study to investigate the CGA process, but also to asses the potential of this "new" technology as a potential alternative to existing small scale mining operations, such as mercury amalgamation.

Trial and error tests were performed and a great deal of time and effort was spent on the safety aspects involved. The amalgamation test was run under the standard conditions as used at the Centre for Mineral Technology (CETEM). in Brazil, which are: 30% solids by weight; mercury/ore ratio: 1:20; Sodium hydroxide (NaOH)/ore ratio: 1/1000 and conditioning for two hours in a horizontal cylinder at 20 rpm (Costa *et al.. 1994).*

A synthetic gold ore $(7 \text{ g gold/ton ore})$ was prepared as follows: 44 μ m gold particles were thoroughly mixed into a I kg silica sample in the particle size distribution size of - 106 um. Pure mercury was obtained from the chemical store. Furthermore, sodium hydroxide (NaOH) was added to optimise the surface properties of the mercury with respect to the feed charge in the closed "vessel" (Hentschel *et al.*, 1992b).

Mercury is a dangerous substance, therefore the necessary safety precaution were important. If spilled, the metal devides into hundreds of "little balls", which is the effect impurities has on the pure metal. If not cleaned up properly, the metal vaporises with time and the toxic vapour is absorbed via the respiratory tract. Therefore, it was decided to perfonn the experiments inside a perspex container, specially designed to fit into a fume cupboard to account for both spillages and toxic vapours. Furthermore, sulphur (which reacts with mercury) was kept at hand as well as a vacuum pump fitted with a mercury trap, to account for spillages. Special cartridge masks. fitted with mercury cartridges, were worn during the experimentation. All mercury wastes were sent to Waste Tech for disposal.

The experiments were performed on the basis of 20 (Experiment 6.17) and 10 (Experiment 6.18) gram synthetic ore samples. From the above ratios, the 20 g ore samples was added to 50 ml of a 0.4 g NaOH/l solution into a 200 ml bottle, to which 1g of mercury was added and contacted for 2 hours on a roller at ± 200 rpm (the lowest setting on the rollers available for our use). The $10g$ samples were contacted with $0.5 g$ mercury and 25 mJ of the same NaOH solution. A separation funnel was used to separate the amalgam from the slurry. Both the ore samples as well as the amalgam was dissolved in *aqua regia* and the analysed on the ICP for gold. The amalgam sample was put inside a round bottom flask, fitted to a reflux condenser and heated by utilising a heating mantel inside the vacuum cupboard. The reflux condenser was used as an additional safety precaution to condense possible mercury vapours before they could reach the atmosphere. Refer to Experiments 6.17 and 6.18, Appendix A for the experimental conditions and raw data.

Representative ore samples (Samples 13-18, Exp. 6.17 and Samples 7-9, Exp 6.18) were analysed to determine the gold loading of the synthetic ore before any contact so that the mass balance could be checked. Unfortunately, the percentage gold recoveries were not calculated from the amalgam samples, as these measured very low gold values, thus concluding that the large amounts of mercury and/or acid had a negative affect on the instrumental analysis by ICP. Therefore, it was decided to calculate the percentage gold recoveries form the remaining ore samples. It was assumed, however, that the remaining gold was transferred to the "mercury phase". The results are tabulated in Table 6. I2.

Table 6.12 - Gold recovery percentages measured after performing mercury amalgamation as compared to that of Coal Gold Agglomeration.

The average gold recoveries obtained from the CGA process was recorded from Experiment 6.13 (83.03%) and Experiment 6.7 (88.75%) and represents the CGA process under optimised conditions, utilising ethane oleate and a synthetic ore mixture. It is, however, seen from Table 6.12 that percentage gold recoveries by the CGA process were approximately 12 to 18 % better than by the mercury amalgamation process.

6.5.1 Predictions according to the thermodynamic stability model

Both the mercury amalgamation and CGA processes were firstly assessed with respect to the thermodynamic stability.

As the oil type utilised in the CGA experiment was known (Exp. 6.7 and 6.13), it was possible to find the actual interfacial tensions, γ_{23} , for each process, whereas γ_{23} denotes the interfacial tension between ethane oleate and water $(\gamma_{\text{ol/water}})$ for the CGA process and the interfacial tension between mercury and water $(\gamma_{mercurv/water})$ for the mercury amalgamation process, whereas the following values were measured/calculated:

- $\gamma_{\text{oil/water}}$ for the CGA process, utilising ethane oleate = 2.06 $\times 10^{-2}$ J/m² (see Experiment 6.19, Appendix A)
- γ _{mercury/water} for the mercury amalgamation process was calculated from the surface tensions of water and mercury, whereas both are recorded in literature (Barrow, 1989):

$$
\gamma_{\text{mercury/water}} = \gamma_{\text{mercury}} - \gamma_{\text{water}}
$$

= 0.480 - 0.07275
= 4.07250×10⁻¹ J/m²

From the above calculations, it is, however, seen that the actual value of $\gamma_{\text{mercurv,water}}$ exceeded the actual value of $\gamma_{\text{oil/water}}$ by one order of magnitude and implies that the magnitude of the free energy per unit area $(F_{II}I)$ for the amalgamation process will always be one order of magnitude larger than F'_{II-1} for the CGA process, where F'_{II-1} = γ_{23} [F"_{II-1}] (see section 3.4.4, chapter 3). It was, however, stated that the actual "sign" in

front of F''_{II} was the determining factor when comparing processes, as a larger negative "sign" would identify the more stable process. Therefore, two scenario exists when comparing the mercury amalgamation process and CGA process:

- $F'_{\text{II-1}} = \gamma_{23} \left[+ F''_{\text{II-1}} \right] = + F'_{\text{II-1}}$, which is true for $\theta \ge 90^{\circ}$, for each process, as seen from Figure 6.2 and 6.3. When this is true, the fact that the order of magnitude of γ_{CGA} < $\gamma_{\text{Amalgamation}}$ is to the advantage of the CGA process, as the actual value of $F'_{\text{II-1}}$ for the CGA process will be one order of magnitude less than F'_{H-1} for the amalgamation process on the positive scale, rendering it to be the more stable process and implies better gold recoveries.
- $F'_{\text{II-I}} = \gamma_{23}$ [- $F''_{\text{II-I}}$] = $F'_{\text{II-I}}$, which is true for $0^{\circ} \le \theta \le 9.9^{\circ}$, as seen from Figures 6.2 and 6.3. When this is true, the fact that the order of magnitude of $\gamma_{\text{CGA}} < \gamma_{\text{Amalgamation}}$ is to the disadvantage of the CGA process, as the actual value of F_{U-1} for the CGA process will be one order of magnitude less than F_{UL} for the amalgamation process on the negative scale. This implies that the free energy per unit area for the amalgamation process is a larger negative value, rendering it to be the more stable and hence, the process to yield better gold recoveries.

For the above two scenarios to hold. it was, however. found that the three-phase contact angles for both processes had to be either above 90° (θ > 90°) or below 90° ($0^{\circ} \le \theta$ < 90°) at the same time. This is illustrated in Figures 6.2 and 6.3. which are plots of the free energy per unit area (F'_{II-I}) vs. k at various values for the three-phase contact angle (θ) for the mercury amalgamation process (Figure 6.2) and CGA process (Figure 6.3).

It was found that the measurement of the three-phase contact angle (θ) of mercury on a gold surface, in the presence of water required a more advanced technology, whereas mercury forms an alloy i.e. amalgam with gold as soon as it "touches" the surface, which

by measurement would then be θ for mercury on the amalgam surface instead of the pure gold surface in the presence of water. Therefore the mercury amalgamation process was not defined by a specific three-phase contact angle (θ) , but it was assumed that θ had to be less than 90° ($0^{\circ} \le \theta \le 0^{\circ}$) as gold recoveries were above 70%.

The gold recoveries by the CGA process, as recorded in Table 6.12, was for utilising ethane oleate as agglomerant (Exp. 6.7 and Exp. 6.13), which measured a three-phase contact angle (θ) of roughly 64.6°, which is below 90° and confirms that the value of $F'_{\text{II-1}}$ is a negative value, as seen from Figure 6.4. As various three-phase contact angles (θ) were measured for the different oils utilised in the CGA process (see Table 6.4 or Experiment 6.19, Appendix A for the measurement thereof), it would not be just to confine one specific three-phase contact angle to the overall CGA process. Therefore, it was seen from Figure 6.4, regardless the oil type used, the value of F'_{II} was always below 0, i.e. negative, as three-phase contact angles for the various oil types were all measured below 90°.

In conclusion, it was assumed that the three-phase contact angle for the amalgamation process was below 90 $^{\circ}$, and as θ for the CGA process was already measured to be below 90 $^{\circ}$, it implies that the free energy per unit area (F' $_{\text{II-I}}$) for both processes were in the negative scale. As the order of magnitude of $\gamma_{CGA} < \gamma_{Amalgamation}$, it is, therefore, further concluded that the free energy per unit area for the amalgamation process would yield a F'_{II-1} larger negative by one order of magnitude and hence, should yield the better gold recoveries. The opposite was, however, found when performing both e .periments, as gold recoveries by the CGA process exceeded that of the Amalgamation process (refer to Table 6.12). Therefore, the effect of other factors. such as the maximum Au transfer of gold particles into the coal-oil or mercury phase and/or the separation efficiencv of the coal-oil agglomerates and/ore amalgam also need to be considered.

6.5.2 Influence of other factors on gold recoveries

A checklist of **all** the variables possibly influencing the maximum gold transfer of gold into the coal-oil phase and/or mercury phase as well as the separation efficiency of the coal-oil agglomerates and/or amalgam, was drawn up in Figures 6.13 and 6.14 for each process. Although the CGA and mercury amalgamation differ with respect to the coaloil agglomerates and mercury droplets being the *gold carriers* the mechanism of attachment of gold particles to the agglomerates and/or mercury droplets are still assumed to be the same. The efficiency of attachment might, however, differ because of the physical differences of coal-oil agglomerates and mercury droplets, which might influence the maximum gold transfer and separation efficiency of each process. Therefore, it was aimed to determine which process should be the more effective according to these factors, which was dictated by the physical substance of the agglomerates and mercury droplets.

As it is seen from Figure 6.13, the contact periods, mineralogy of the ore, available surface area of agglomerates or mercury droplets and chemical additions were all stable and sufficient to ensure maximum gold transfer into the coal/oil and mercury phases. It was, however, found that the *mode/motion of contact* for the two processes differed. It is in effect not this variable as such which caused the difference in gold recoveries for the two processes, but rather the response of the *gold carriers* i.e. agglomerates and mercury droplets to each mode of contact selected for the specific process. In order to elucidate the above statement the following should be taken into account:

Mercury droplets are much denser and heavier than coal-oil agglomerates. which verifies why *stirring* was not selected as the mode of contact, whereas the mercury droplets would rather sink to the bottom than stay in suspension. Although the *rolling battle technique* is the most efficient mode of contact for the mercury amalgamation process. it does not entail that contact is as efficient as *stirring* is for the CGA process. However, due to the densiry of the mercury droplets. the rolling bottle technique might not provide the same

sufficiency of motion, as most of the mercury droplet/gold particle contact takes place at the side of the bottle, whereas the rolling action *forces* the matter in the bottle to assemble as the rolling action proceeds. It is, therefore, concluded that *stirring* provides a more sufficient contact for gold particles and agglomerates than does the *rolling bottle action* for the mercury droplets and *gold* particles, thus having a better chance of increased maximum gold transfer and hence, gold recoveries.

The same argument *holds* for the separation efficiency, whereas the agglomerate or mercury droplet stability, radii and chemical additions are sufficient for efficient separation, as depicted in Table 6.14. It was, however the response of the *gold carriers* (agglomerates and mercury droplets) to the means of separation, which caused the difference in gold recoveries. Therefore, it was assumed that separation via scraping for the CGA process was more efficient than was separation via a separational funnel for the amalgamation process. As mercury divides into small droplets, it was difficult to separate all the smaller droplets completely, leaving a larger percentage possible *amalgam* behind and explains why the separation efficiencv and hence. gold recoveries by amalgamation was less.

Table 6.13 - The checklist of variables **influencing the MAXIMUM GOLD TRANSFER of gold particles** *into the* **coal-oil and/or mercury phase.**

Table 6.14 - The checklist of variables **influencing the SEPARATION EFFICIENCY of coal-oil agglomerates and/or amalgam from the ore slurry.**

In conclusion the response of the coal-oil agglomerates to the *mode of contact* and *means of separation* chosen for the CGA process was more effective, than was the response of mercury droplets to those chosen for the amalgamation process. *This* explains why thc maximum gold transfer into the coal-oil phase and the separation efficiency thereof was better than for the amalgamation process, thus causing better gold recoveries.

Finally, the importance of the operating parameters to gold recoveries are confirmed. as these could explain the lower gold recoveries by amalgamation. as was not possible by utilising the thermodynamic stability model alone, whereas the affect of these on gold recoveries outweighed the predictions made by the model.

Although the CGA process does not outperform the amalgamation process in *strictu sensu,* it definitely is the better alternative based on the environmental friendliness and safety thereof. Furthermore, gold loadings can be increased on one batch of agglomerates by recycling, which makes the CGA even more attractive in terms of the economics, as would be compared to the amalgamation process.

Ratio of radii of coal-oil agglomerates to gold particles (k)

Figure 6.2 - The free energy per unit area (F'_{II-I}) vs. k at se ected values for the three-phase contact angle (θ) from 0° to 180° for the MERCURY AMALGAMATION process.

Ratio ofradii of coal-oil agglomerates to gold particles (k)

Figure 6.3 - The free energy per unit area(F'_{II-1}) vs. k at selected values for the three-phase contact angle (θ) from 0° to 180° for the COAL GOLD AGGLOMERATION process.

Figure 6.4 - The free energy per unit area (F'_{II-I}) vs. k for various oil types used while performing the Coal Gold Agglomeration process.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 CONCLUSIONS

- (a) The CGA process yielded promising results, which under optimal conditions were up to 85% gold recoveries. By performing a sensitivity analysis on the operating parameters, the optimal conditions were found to be:
	- Contacting the coal-oil agglomerates and ore slurry for periods exceeding 50 minutes.
	- Contacting the coal-oil agglomerates and ore slurry by stirring.
	- The addition of a collector. i.e. potassium amyl xhantate (PAX).
	- A decreased amount of water to prepare the ore slurry.
	- An increased amount of coal/agglomerates

Furthermore, it was found that factors such as an increased amount of oil and contact by the rolling bottle technique decreased percentage gold recoveries.

(b) A set of recycling experiments was performed. whereby the ore phase was recycled and it was found that the CGA process is suitable for recovering gold from very low grade ores. Further recycling of the coal-oil phase has proved that it is possible to increase the gold loading onto the coal-oil agglomerates, which will be to the advantage of the economical viability of the process.

- (c) When the CGA process was repeated on a real ore sample, whereby the effect of the *mineralogy of the ore* on the performance of the CGA process was investigated, lower gold recoveries than with the synthetic ore was found and the following conclusions were made:
	- Firstly, the *mineralogy of an ore* dictates the amount of free/liberated gold particles, as gold could be entrapped within the mineral matrix, causing less liberated gold particles to be available for recovery into the coal-oil agglomerates and hence, lower gold recoveries are found.
	- Secondly, a recycling test had revealed that gold recoveries by the second batch of agglomerates was much less than with a synthetic ore *mixture* and after performing XRD analysis, the *presence of other minerals* in the coal-oil phase was confirmed and concluded to cause the lower gold recoveries duc to the "clogging" of the agglomerates surface to gold particles.
- (d) A theoretical model was derived from first principles. whereby the thermodynamic stability of the CGA process was related to the total free energy per unit area, which *is* a *function* of the three-phase contact angle (8), interfacial tension (γ_{23}) and ratio of radii of agglomerates to gold particles (k) . The thermodynamic stability was concluded to be a measure of the feasibility of the process and hence, linked to the percentage gold recoveries and valuable predictions were made. It was, however, found that the thermodynamic stability was not the only determining factor when predictions as to geld recoveries were made, as the maximum gold transfer *into* the coal-oil phase and the separation efficiency of the agglomerates were factors found to be detrimental to gold recovery predictions. Furthermore, the last two factors mentioned in the previous

statement was each dependent on certain operating parameters. It was concluded, therefore, that percentage gold recoveries was affected by both theoretical factors (linked to the thermodynamic stability) and operating parameters (linked to the maximum gold transfer and separation efficiency), whereas both were to be acknowledged when gold recovery predictions were made. Finally, the application of the knowledge gained from the theoretical study was in aid of explaining certain tendencies found in the results after performing experiments.

- (e) By perfoming the CGA experiments on various oil types, it was concluded that the application of the theoretical knowledge was useful when utilising the thermodynamic stability model in conjunction with the maximum gold transfer and separation efficiency factors to make very ac urate predictions as to the percentage gold recovenes. It was, however, found that percentage gold recoveries were the highest when utilising ethane oleate as agglomerant. Although ethane oleate was found to yield one of the least thermodynamically stable configurations, the influence of the maximum gold transfer and separation efficiency factors outweighed the influence of the stability model, which explained the better gold recoveries.
- (t) A comparative study was conducted, whereby the CGA process was compared to mercury amalgamation in order to ascertain if it is in fact a valid alternative for small-scale gold mining. Laboratory scale batch tests were performed, whereby gold recoveries of 70% was found, which is less than for the CGA process. Although the amalgamation process yielded the thermodynamically more stable configuration, the influence of the other factors, i.e. maximum gold transfer and separation efficiency was found to outweighed the influence of the thermodynamic stability on gold recoveries and explains why' the CGA process performed better.
7.2 **RECOMMENDATIONS**

As the essence of this study is summarised in Figure 3.5, Chapter 3, it is recommended that any person/institution engaging in any future studies on the CGA process, utilise the meaningful information recorded in this diagram and the bulk ofthis script. However, it seemed plausible to recommend that the following preliminary investigations be carried out before engaging in any such investigations.

- Before the selection of an oil type, the three-phase contact angle (θ) of various oils measured on a pure gold surface in the presence of water, followed by the measurement of the interfacial tensions (γ_{23}) between water and the oils should be completed (see Experiments 6.19 and 6.20, Appendix A for the methods of measurements). The oil measuring the lowest three-phase contact angle (θ) below 90 \degree and the largest interfacial tension (γ_{23}), should then be selected, as a three-phase contact angle (θ) closest or equal to 0° ensures thermodynamic stability, which is then enhanced by a large interfacial tension. This type of configuration will ensure thermodynamic stability
- \bullet It is vital that the size of the agglomerates and gold particles be monitored, as the ratio of agglomerate to gold particle radii (k) should be as large as possible. implying that the size of the agglomerates should exceed the size of the gold particles to be recovered. Therefore, if very large gold particles are to be recovered, the size of the agglomerates could be enlarged by utilising larger coal particles to prepare the agglomerates.
- As the means of separation is dictated by the size of the agglomera es, it is vital to select the correct means of separation for complete separation e.g. larger agglomerates would require the use of a sieve rather than a floatation procedure.
- It is further recommended that the *mineralogy of an ore* be characterised, finding the exact amount of liberated gold particles in the gold ore and the actual minerals present.

• Finally, it is recommended that the stirring speed in a scale-up operation be investigated to find the optimal speed of particle rotation for optimal gold particle and agglomerate attachment.

NOMENCLATURE

REFERENCES

- 1. Adamson, WA; **Physical Chemistry of surfaces;** 5th Edition, John Wiley and sons, Inc., New York, 1990.
- 2. Alien, RW; Wheelock, TD; **Effects of pH and Ionic strength on the kinetics of oil agglomeration of fine coal;** *Minerals Engineering;* Volume 6, No I, 1993, pp. 87-97.
- 3. Barin, I; Barth, H; Yaman, A; **Electrochemical investigation of the kinetics of gold cementation from cyanide solutions;** *Erzmetall,* 33(7/8), 1980, pp. 399- 403.
- 4. Barrow, GM; Physical Chemistry; 4th Edition, McGraw-Hill Book Company. New York, 1989.
- 5. Bellamy, SR; House, Cl; Veal, CJ; **Recovery of fine gold from a placer ore by coal gold agglomeration;** *Gold forum on Technology and Practices* - *'World Gold* '89', Reno, Nevada, 5-8 November 1989.
- 6. Bonney, CF; **Coal Gold Agglomeration A novel approach to Gold recovery:** *Randol Gold Forum* 88: Scottsdale. Arizona, 23-24 January 1988.
- 7. Bonney, CF; **The BP-CGA and CARBAD processes- technical and cost differences;** *Innovations in Gold and Silver recol'eries:* Randol. Ch. 16. 1992. pp. 2320-2322.
- 8. Bosley, D; Recovery of gold from solution by Cementation; *The Extractive Metallurgy ofgold in South-Africa,* Stanley GG (Ed.), South African Institute of Mining and Metallurgy, Johannesburg, 1987, pp. 331-343.
- 9. Boss, CB and Fredeen, KJ; Concepts, Instrumentation and Techniques in Inductively Coupled Plasma Optical Emission Spectrometry; 2nd Edition. 1997.
- 10. Boucher, EA and Murrel, IN; Properties of liquids and solutions; John Wiley and sons limited; Chichester, 1982.
- 11. Branches, FJP; Erickson, TB; Aks, SE; Hryhorczuk, DO; The price of gold: Mercury exposure in the Amazon rain forest; *Clinical toxicology,* 31 (2), 1993, pp. 295-306.
- 12. Bretherick, L (Ed.); Hazards in the chemical laboratory; *The Royal society of chemistry;* London, 1987, pp. 390-391.
- 13. Brodie, KG; Doidge, P; Fravy, B; Mckenzie. T; Rowland, J; Analytical methods for Graphite Tube Atomisers; Rothery, E (Ed.), January 1982.
- 14. Bryson, PD: Comprehensive Review in toxicology: 2nd edition, Rockville, MD: Aspen, 1989, pp. 477-486.
- 15. Buckley, SA; House, CI and Townsend IG; A Techno-Economic Assessment of the Coal Gold Agglomeration Gold Recovery process; *The Canadian Mineral Processors conference,* Ottawa, January, 1989.
- 16. Cadzow, M and Lamb, R; CARBAD gold recovery; *World Gold* 1989; Edited by Bhappuaho, RB; Harden, RJ; AlME, Littleton, Colorado, USA, Chapter 45, 1989, pp. 375-379.
- 17. Calvez, JPS; Kim, MJ; Wong, PLM and Tran, T; Use of coal-oil agglomerates for particulate gold recovery; *Minerals Engineering;* Volume 11, No 9, 1998, pp. 803-812.
- 18. Câmara, VdeM; dSolva, AP; Maciel, MV; Pivetta, F; Perez, MA; Mercury Exposure and health effects among urban residents due to gold commercialisation in Pocone; *Technologia Ambiental*, MT, Brazil, 1997.
- 19. Cetem Centro de Technologia Mineral; Pocone project, Annual Report, Rio de Janeiro, 1989.
- 20. Cornejo. LM and Spottiswood, *DJ;* Fundamental aspects of the gold cyandidation process: A review; *Mineral Engineering Resources.* Colorado School of mines, 1984.
- 21. Costa, LSN; Lins. FF and Marciano. A; Utilization of coal-oil agglomerates to recover gold particles; *Minerals Engineering:* Vol. 7, No 11, 1994. pp. 1401- 1409.
- 22. Crowe, TB; Effect of oxygen upon the Precipitation of metals from cyanide solutions; *Bull. Am. Mining Eng..* 1918. pp. 1279-82.
- $23.$ Davidson, RJ; A review of the AARL Process for the elution of gold from Activated Carbon; *Lecture* 15. *SAlJfM "ClP" School;* Randburg. February 1986, pp. 23.
- 24. D'ltri, FM; The environmental mercury problem; *CRC Press;* Cleveland, Ohio, 1972, pp. 124.
- 25. Dreisbach, RH; Handbook of poisoning: prevention diagnosis and treatment; *Lange Medical Publications;* Los Altos, California, 1980, pp. 234-239.
- 26. Ellenhorn, MJ; Barceloux, DG, eds. Medical Toxicology: Diagnosis and treatment of Human Poisoning, $1st$ edition, New York: Elserivier, 1988, 1048-1053.
- 27. Emerson, T and Kepp, M; Brazils Amazon miners: risk mercury poisoning; *Newsweek,* 24 June 1991.
- 28. Finkelstein, NP; The chemistry of the extraction of gold from its ores; *Gold Metallurgy in South-Africa,* Adamson (Ed.), Chamber of mines of South-Africa. Johannesburg, 1972, pp. *305.*
- 29. Florentine, MJ and Sanfilippo, DJ; Elemental mercury poisoning; *Clin. Pharm.* 10; 1991, pp. 213-221.
- 30. Friberg, L and Vostal, J; Mercury in the environment: An epidemiological and toxicological Appraisal; *CRC Press. Inc..* Cleveland. 1972.
- 31. Gaidarjiev, St; Kireva, R; Stoev, St; Coal-gold agglomeration of alluvial gold; *Changing scopes in Mineral Processing;* Kernal, Arsian, Akar and Canpazojiv (eds.), 1996.
- 32. Glasstone, S and Lewis, D; Elements of Physical Chemistry; $2nd$ Edition. ISBN *0-333-038436, 1981.*
- 33. Goldfrank, IR and Lewis, R; Goldfrank's Toxicologic Emergencies; 3'd Edition, 1986, pp. 592.
- 34. Goldfrank, IR; Bresnitz, EA; Howland, MA; Mercury; *Toxicologic Emergencies*, 4th Edition, Goldfrank LF, Flomembaum, ME, Lewin, NA eds... Norwalk, CT: Appleton-Century-Crofts, 1990, pp. 641-648.
- 35. Habashi, F; One hundred years of cyanidation; *CfM Bull.,* 80(905): 108-14, 1987.
- 36. Hammelmann, CR; Lins, FF; Middea, A; Mon.e, MM; Amalgamation in small-scale gold operations alternatives and treatment of Hg-contaminated soils and effluents; *Extraction and Processing devision Congress* 1994; TMS 123rd annual meeting, Warren, GW (Ed.), TMS, 1994.
- 37. Helferrich, F; Ion-Exchange; *Mc Graw-Hill,* New York, 1962.
- 38. Henly, KJ; Gold ore mineralogy and its relation to metallurgical treatment: *Mineral Science and Engineering,* 17(4):289-312, 1975.
- 39. Henry, JD; Hovarongkura, AD; MaIcolm, TJ; Feasibility of separation processes in liquid-liquid solid systems: Free energy and stability analysis: *AICHEJournal.* Volume 25, No I. January 1979. pp. *160.*
- *40.* Hentschel, T and Priesler. M: Small-scale Gold mining. ISBN 3-528-02064-4. 1992a.
- 41. Hentschel, T and Priester, M; Mercury contamination in developing countries through gold amalgamation in small-scale mining: some process

alternatives; *National Resources and Development,* Volume 35, 1992b, pp. 67- 77.

- 42. Hocker, M; Cyanide Spring, Mineral Policy Centre, Environmental Impacts; *Internet: http://www.1872. org/cyanide. html*
- 43. Hoffman, R; Winning Gold; *American Scientist,* Volume 82, Jan-Feb 1994, pp. 15-17.
- 44. Homewood, B; Mercury poisoning confirmed among Amazon villagers; *New Scientist,* Rio de Janeiro, November 1991.
- 45. Hoover, HC and Hoover, LH; Agricola: De re Metallica (translated); *The Mining Magazine,* London, 1912, pp. 295-299, pp. 280-341.
- 46. House, CI; Townsend, IG; Veal, CJ; Separation Process; Pat. Int. Cl(4) C22B1/16, 1GB 87/00480, W 088/00248, 1988a.
- 47. House, CI; Townsend, IG; Veal, CJ; Coal Gold Agglomeration; *International Mining,* September 1988b.
- 48. House, CI: Townsend, IG and Veal. CJ: Coal Gold Agglomeration Pilot Scale retreatment of tailings; *Randol International Gold Conference,* Perth Australia November I988c.
- 49. Hughes-Narborough, C; Wall, NC; WilIey. G; Coal Gold Agglomeration: An innovative approach to the recovery of gold in environmentally sensitive areas; *IV International Minerals Processing.* Volume 2. October 1992. pp. 20- 22.
- 50. Huiatt, J; Kerrigan, J; Olson, F; Potter, 0; Workshop of Cyanide from mineral processing; Utah Mining and Mineral Resources Research Institute; College of Mines and Minerals Industries, Salt Lake city, February 1982.
- 51. Internet: http://www.dme.qld.gov.au/ed/ed-gold.htm
- 52. Internet: http://www.wantree.com.au/~menne/hgl.htm.
- 53. Internet: http://gurukul.ucc.american.edu/ted/BRAGOLD.HTM...
- 54. Internet: http://smi.minmet.mcgill.ca/SMI/volume-2/ghana.htm.; Small-scale gold mining: An evolving success story in Ghana.
- 55. Irukayama, K; Minamata Disease, Tsubaki, T; lrukayama, K (eds.); Kodansha, Tokyo, 1977, pp. 2-56.
- 56. Joselow, MM; Louria, DB; Browder. AA; Mercurialism Environmental and occupational aspects; *Ann. Intern. Med* 76; 1972. 119-130.
- 57. Keller, DV and Burry, W; An Investigation of a separation process involving Liquid-Water-Coal systems; *Colloids and Surfaces;* 22.]987, pp. 37-50.
- 58. Klein, SL and Sampaio. CH; Study of selective gold recovery from refractory slime flotation tailings by charcoal-oil agglomerates, *Comolex ores '97:* Bulawayo, Zimbabwe, 19-21 March 1997.
- 59. Kurland, LG; Faro, SN; Siedler, H; Minamata Disease, *World Neural 1*, 1960. pp. 370-390.
- 60. Labuschagne, *B1C;* Relationships between oil agglomeration and surface properties of coal: effect of pH and oil composition; *Coal Preparation;* Volume 3, Gorden and Breach Science Publishers SA, Printed in the United Kingdom, July 1986, pp. 1-13.
- 61. Labuschagne, *BC1;* Rex, V; Prinsloo, JJ; Influence of oil composition, pH and temperature on the selective agglomeration of coal; Proc. *5^h International Symposium on Agglomeration;* 1989, pp. 505-514.
- 62. Lein, C; Todoruk, D; Rajani, H; Accidental inhalation of mercury vapour: Respiratory and toxicologic consequences; *Can. Med. Assoc. J.* 129.; 1983, pp. 243-246.
- 63. Mainwaring, LM; Cadzow, M; Elkes, *lS;* Recovery of metal values from mineral ores by incorporation in coal/oil agglomerates; U.S. Patent no. 4,585,548; BP Australia Ltd., 19 April 1986.
- 64. Magos, L; Role of Catalase in the oxidation of mercury vapour; *BoichemPharmacol;* 1978, pp. 27: 1373-1377.
- 65. Magos, L; Physiology and Toxicology of Mercury; *Metal Ions in Biological systems;* Sigel, A and Sigel H (eds.), Volume 34, ISBN 0-8247-7828-7,1997, pp. 349-350.
- 66. Marcus, Y and Kertes, AS; Ion-Exchange and Solvent extraction of metal complexes; *Wiley-Interscience.* London, 1969.
- 67. McArthur, JC; Forrest, RW; Forrest, W; Process obtaining gold and silver from ores. Brit. patent 14174 (1887); U.S Patent 403,202 (1889); U.S Patent 418,137(1889). 1887- 1889.
- 68. McQuiston Jr., FW and Shoemaker, RS; Gold and Silver cyanidation plant practice; *S.ME* - *A.I.ME,* New York, Volume I, 1975.
- 69. Mehrotra, VP; Sastry, KVS; Morey, BW; Review of oil agglomeration techniques for processing of fine coals; *International Journal of Mineral Processing;* 11, 1983, pp. 175-201.
- 70. Nicol, MJ; Flemming, CA; Paul, RL; The Chemistry of the Extraction of Gold; *The Extractive Metallurgy of gold in South-Africa,* Stanley, GG (Ed.), South African Institute of Mining and Metallurg), Johannesburg, 1987, pp. 866- 867.
- 71. Nriagu, JO; Production and uses of Mercury; *The Biochemistry ofmercury in the Environment;* Elserivier/North Holland Biomedical Press, Amsterdam; 1979, pp. 23-40.
- 72. Nriagu, JO and Wong, HKT; Gold rushes and Mercury pollution from metal ions in Biological Systems; *Mercury and its effects on Environment and Biology* :*Volume* 34; Sigel, A; Sigel, H (Ed.).ISBN 0-8247-9828-7.1997.
- 73. Oak Ridge National Laboratory; Reviews of the environmental effect of Pollutants: Cyanide; 1978. pp. 139-45.
- 74. Penman, DW; Gravity concentration: *The extractive metallurgy of gold in South-Africa,* Volume 1, Stanley GG (Ed.). South African Institute of Mining and Metallurgy. Johannesburg. 1987. pp. 229-230.
- 75. Pfeiffer, WC and Lacerda. LD; Mercury imputs into the Amazon Region. *Brazil Environmental Technology letters. l'olume* 9: 1988. pp. 325-330.
- 76. Reynolds, JEF (Ed.) and Martindale; The Extra Pharmacopoeia, $13th$ edition, London, The Pharmaceutical press, 1993, pp. 1387-1388.
- 77. Rose, TK and Newman, WAC; The metallurgy of gold; *Philadelphia: JB Lippincott Co.;* 1937, pp. 168-87.
- 78. Schlettwein, NC and Petersen, FW; Comparative studies of the extractive performance in free cyanide removals by virgin and silver impregnated carbon; *Environmental and Innovation in Mining and Mineral technology,* Proceedings of the IV International conference on Clean Technologies for the Mining Industry; Santiago, Chile, May13-15, 1988.
- 79. Schrader, ME; High and medium energy surfaces: ultrahigh vacuum approach; *Modern approach to wettability* - *theory and applications.* Chapter 3, edited by Scrader, ME and Loeb, GI; Plenum Press, New York, 1992, pp. 53-71.
- 80. Sidall, M; Gold recovery process; V.S. Patent 4.597.791; Bateman Engineering International Ltd.. July 1986.
- 81. Smith, A and Mudder, T; The Chemistry and treatment of cyanidation wastes; *Mining Journal books limited;* London, ISBN 0-900-11751-6. 1991.
- 82. Souci Lu; Hydrophobic interaction in flocculation and flotation 3 Role of hydrophobic interaction in particle-bubble attachment; *Colloids and Surfaces.* 57, 1991, pp. 73-81.
- 83. StanJey, GG; Milling and Classification: *The extractive metallurgy of gold in South-Africa,* Volume 1; Stanley GG (Ed.). South African Institute for Mining and Metallurgy, Johannesburg. 1987.
- 84. Subasinghe, GKN and Maru, Y; The use of mercury in small-scale gold mining operations: effectiveness and pollution aspects; The Australian Institute of Mining and Metallurgy, Exploration and Mining conference; PNG Geology, 1994.
- 85. Szymocha, K; Pawlak, W and Ignaisak, B; Kinetics of coal at extended agitation times; *5th International Symposium on Agglomeration;* 1989, pp. 525- 536.
- 86. Tsubaki, T; Shirakawa, K; Hirda, K; Kondo, K; Sato, T; Kanbayashi; Minamata Disease; Tsubaki, T; lrukayama, K (ed .); Kodansha, Tokyo, 1977, pp. 56-78.
- 87. Veiga, MM and Meech, *lA;* Heuristic approach to mercury pollution in the Amazon; *Proc. ofthe International Symposium on Extraction and Processingfor the treatment and minimisation of wastes;* 123rd Congress of TMS, The Minerals, Metals and Materials Society, San Francisco, 27 Feb. - 3 March 1994, pp. 23-38.
- 88. Veiga, MM and Meech, *lA;* A brief history of amalgamation practices in the Americas; *Internet: http://www.mining.ubc.ca/faculty/meech/briefhi2.htm.*; April 1997.
- 89, Van der Merwe, PF; Fundamentals of the Elution of Gold Cyanide from Activated Carbon: Ph.D Thesis; University of Stellenbosch, June 1991, pp. 1-275.
- 90. Yannopoulos, JC; The Extractive Metallurgy of Gold, ISBN 0-442-31797-2, 1991.
- 91. Young, GJC; Cyanidation; *The Extractive Metallurgy of gold in South-Africa*, Stanley, GG (Ed.), South African Institute of Mining and Metallurgy, Johannesburg, 1987, pp. 291.
- 92. Zadra, JB; A process for the recovery of Gold from Activated Carbon by Leaching and Electrolysis; *U.S. Bureau of Mines Report of Investigations no.* 4672; 1950.
- 93. Zhongfu Dai and Souci Lu; Hydrophobic interaction in flocculation and flotation 2 - Interaction between non-polar oil drop and hydrophobic mineral particle; *Colloids and Surfaces,* 57, 1991, pp. 61-72.
- 94. Zisman, WA and Bemett, MK; Confirmation of spontaneous spreading by water on pure gold; *The Journal of Physical Chemistry,* 74; 1970, pp. 2309- 2312.

SAMPLE ANALYSIS

CALCULATIONS

EXPERIMENTAL CONDITIONS AND RAW DATA

SAMPLE ANALYSIS

The Coal Gold Agglomeration (CGA) process has been described in detail in this script and it will therefore be assumed that the reader is familiar with the sample methodology. For one single CGA test there are three different samples to be analysed nl.:

- the initial ore sample (C_0) , to determine the initial gold concentration in the ore,
- the coal-oil agglomerates (A_1) , which have been dried and ashed (see section 4.4) is analysed to determine the amount of gold which is recovered in this phase and
- the final ore sample (C_1) , to determine the gold loss from initial conditions.

Analysis to determine percentage gold recoveries was, however, performed by one of two instruments, nI. the Graphite Tube Analyser (GTA) or the Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), which will be referred to as the ICP in this script. The GTA was used to analyse the samples at the Cape Technikon, by the masters student herself, whereas more information as to the settings utilised during analysis is available. It is, however, not possible to provide the reader with the exact setting used during ICP anIysis, as these analyses were performed by an "outside" company, as will be discussed later in this section.

The Grapite Tube Analyser (GTA)

The temperature program and settings utilised for the analysis of gold on the GTA was as follows:

Table Al - Furnace operating parameters for the analysis of gold (Au) on the GTA, utilising a pyrolitic coated graphite tube as atomiser.

The function of the temperature raises are to generate a population of free gold atoms so that the atomic absorption could be measured. This is achieved in three temperature stages, which are identified as follows:

- 1. *Drying* step no. I to 3 this is the stage in which the solvent is removed from the sample furnace.
- *2. Ashing* step no. 4 to 8 at this stage the organic molecules or inorganic material is removed.
- *3. Atomization* step no 9 to 11 this is the stage in which free atoms are generated within a confined zone. The absorption signal produced in the atomization stage is a sharp peak, the height (or area) of which can be related to the amount of analyte element present and is being read at stages 9 and 10, as indicated in the table above.

The *atomic absorption* is measured via a techique called the *atomic absorption spetrometry* (AAS). In AAS light of a wavelength charact existic of the element of interest is shone through the atomic vapour. The source of light is provided by a '"gold lamp", which is inserted and wavelenght settings etc. are installed to ensure that the correct wavelength for gold is provided. Some of the light which is shone through the atomic vapour is then absorbed by the atoms of the element to be read, gold in our case. The amount of light that is absorbed by these atoms is then measured and used to determine the concentration of that element in the $20 \mu l$ which was injected by microsyringe (Brodie *et af.,* 1982; Boss *et al., 1997).*

The Inductively Coupled Plasma (ICP)

Analysis were performed by Mr Arrie van Deventer of the AGRICULTURAL RESEARCH COUNCIL at the Stellenbosch Institute for Fruit Technology, Infruitec, in Stellenbosch:

> *INFRUITEC, Private Bag X5013, Stellenbosch.* 7599, *South Africa. Tel: (021) 8093322, Fax: (021)* 8838669 *{International: -+-2721] E-mail: netmanager@infruit.agric.za, Web site: www.arc.agric.za*

The basic operation of an ICP is as follows: Argon gas is directed through a torch consisting of three concentric tubes made of quartz or some other suitable material. A copper coil, called the load coil, surrounds the top end of the torch and is connected to a radio frequency (RF) generator, which applies power to the load coil which causes an alternating current moving back and forth within the coil. This RF oscillation of the current in the coil causes RF magnetic and electric fields to be set up in the area at the top of the torch. With argon gas being swirled through the torch, a spark is applied to the gas causing some electrons to be stripped from their argon atoms, which are then caught up in the magnetic field and accelerated by them. Adding energy to the electrons by the use of a coil in this manner is known as *inductive coupling.* These high-energy electrons in turn collide with other argon atoms, stripping off still more electrons. This collision

ionization of the argon gas continues in a chain reaction, breaking down the gas into a plasma consisting of argon atoms, elecrons, and argon ions, forming what is known as an inductively coupled plasma (ICP) discharge. The ICP discharge is then sustained within the torch and load coil, as RF energy is continually transferred to it through the inductive coupling process. The ICP discharge appears as a very intense, brilliant white, teardropshaped discarge, which is toroidal, or "doughnut-shaped" at the base because the samplecarrying nebulizer flow literally punches a hole through the centre of the discharge. The body of the "doughnut" is called the induction region (IR) because this is the region in which the inductive energy transfer from the load coil to the plasma takes place. This is also the area from which most of the white light, called the argon continuum, is emitted. Allowing the sample to be introduced through the induction region and into the centre of the plasma gives the ICP many of its unique analytical capabilities.

The sample is introduced into the the ICP as a liquid that is nebilized into a *aerosol* (a very fine mist of sample droplets) which is carried into the centre of the plasma by the nebulizer. The high temperature plasma then *desolvates* (removes the solvent from the aerosol), leaving the sample as microscopic salt particles, which are then decomposed into a gas of individual molecules *(vapourisation)* that are then dissosiated into atoms *(atomization).* The process decribed in this paragraph is basically the same as used in the funace (GTA) tor atomic adsorption spectrometry. The next two functions of the lCP remaining is that of *excitation* and *ionization* where one of the electrons in the atom/ion is promoted to a higher energy level in order to be able to emit its characteristic radiation.

Obtaining *qualitative information,* i.e., what elements are present in the sample, involves identifying the presence of emission at the wavelength characteristic of the element of interest e.g. gold. Obtaining *quantitative information,* i.e., how much of an element is in the sample, can be accomplished using plots of emission intensity versus concentration called *calibration curves*. Solutions with known concentrations of the element of interest (gold in our case), i.e. *standard solutions,* are introduced to the lCP and the intensity of the characteristic emission for each element, or analyte, is measured. These intensities can then be plotted against the concentrations of the standards to form a calibration curve for each element. When the emission intensity from the analyte is measured, the intensity is checked against the element's calibration curve to determine the concentration ppm or mg/L) corresponding to the intensity. The software used with the lCP instrument represents these calibration curves mathematically within the computer's memory, thus it is not necessary for the analyst to construct these curves manually for quantitation of the elements in the sample (Boss *et aI.,* 1997). Therefore, the experimental raw data which was drawn from ICP analysis is directly in the form of ppm (mg/L).

CALCULATIONS

After *all* the samples have been treated with *aqua regia* and the analysis thereof had been completed by GTA analysis, the results were obtained after tedious calculations, as will be *seen* in this section. For each analysis a calibration curve was drawn by plotting the mass gold (mg), (linked to the concentrations of each standard) which is a known value by the standards chosen, versus the absorbance readings, as determined by analysis on the GTA. The calibration curve, being a straight line, provided an equation for converting the absorbance readings for each sample into the exact mass of gold [Mass Au (mg)l present in 20 μ 1 (injected by microsyringe) of the specific sample under consideration. The concentration of gold is calculated by deviding the mass gold by the volume, which is 0.00002 litres (20 μ) - [Conc. (ppm)]. From this the total mass of gold is calculated by multiplying the the concentration by the exact volume of the volumetric flask in which the sample was diluted - [Total mass of Au (mg)]. As the exact masses of ore and dry coal-oil agglomerates have been recorded, it is possible to determine the gold loading on each sample. Finally the % Au recoveries for each sample/run was determined by deviding the total mass of gold present in the coal-oil sample by the mass of gold originally found in the in the ore.

As the results by ICP analysis was provided in the form of $[Conc. (ppm)]$, all the calculation mentioned above up to this point was only necessary for samples that were analysed by lCP, as the software provided with the ICP analysis handled the mathematical conversions to this point. All further calculations, however, up to % Au recoveries were performed in the same manner as for the samples analysed by GTA.

Note that all numbers printed in *italics* were disregarded, as these values differed with to a large amount from the others read at the same time.

Mass Balance Check - Calculations

As some of the experiments in Chapter 6 were done to check if the mass balance was complete, it is vital to provide the reader with the calculations done to get to each result as will be discussed in this section.

The Coal Gold agglomeration process was performed, after which both ihe coal/oil and ore samples were analysed for gold. In the past the ore (C_0) was analysed for gold before performing the test after which the coal/oil samples $(A₁)$ were analysed and the assumption was made that the "lost" gold was still present in the remaining ore (C_1) . This procedure was followed because of the time consuming and :edious process of perfect separation of the coal/oil agglomerates and ore. However. it was decided to "check the mass balance" of the gold in the system by analysing the ore before (C_0) as well as after contact (C_1) . See Figure Al below for a simplified illustration of the samples.

Figure A1. A simple illustration to assist in the "mass balance" calculations.

From the above sketch: A_0 = The mass of coal/oil/agglomerates entering the system (g) A_1 = The mass of coal/oil/gold agglomerates after contact (g) C_0 = The mass of gold ore entering the system (g) C_1 = The mass of ore remaining after contact (g)

Overall Mass Balance:

$$
C_0 + A_0 = C_1 + A_1 \qquad \qquad \dots (1)
$$

Gold Balance:

$$
C_{o-Au} = C_{1-Au} + A_{1-Au} \qquad \qquad ...(2)
$$

Equation (2) states that no gold is present in the coal/oil agglomerates before contact and that the mass gold (mg) present in the ore before contacting the phases $(C_{o\text{-}Au})$ equals the sum of the masses of gold (mg) present in the ore (C_{1-Au}) and coal/oil agglomerates (A_{1-Au}) after contact.

The percentage gold recovery in the coal phase was calculated as follows:

% Au recovery in the coal/oil phase =
$$
(A_{1-Au})/(C_{o-Au}) * 100
$$
 ...(3)

The percentage gold remaining in the ore phase (C_1) was, however calculated as follows:

% Au unrecovered gold in the ore phase (C₁) =
$$
(C_{1-Au})/(C_{o-Au}) * 100
$$
 ...(4)

The mass balance was checked by adding the results of equations 3 and 4, whereas a complete mass balance implies that this addition should yield 100%.

Recycling of the coal/oil phase - Mass **balance checking - Calculations**

The CGA process was performed as usual and after separation, the agglomerates (A_1) were contacted with a "fresh" ore sample (C_{02}) . The objective of this experiment was to find if the coal/oil phase was capable of recovering gold after being recycled. The sketch below illustrates the procedure.

Figure A2. A schematic representation of the recycling procedure of the agglomerates.

The percentage gold recovery (% Au recovery) was determined for each step, where:

- Step $1 =$ Contacting the coal/oil agglomerates with an ore sample (50 minutes),
- Step $2 =$ contacting the recycled coal/oil agglomerates from Step 1 with a fresh, ore sample (50 minutes, stirring),
- A_1 = the mass of gold recovered from the ore after completion of Step I (mg),
- A_2 = the mass of gold recovered from the ore after completion of Step 2 (mg),
- A_t = the total mass of gold recovered in the coal/oil agglomerates after recycling it once $[A_1 + A_2]$ (mg),
- C_{01} = the mass of gold entering Step 1 (mg),
- C_{02} = the mass of gold entering Step 2 (mg),
- C_1 = the mass of gold remaining in the ore after completion of Step 1 (mg) and
- C_2 = the mass of gold remaining in the ore after completion of Step 2 (mg).

Overall gold balance:

(mass of gold) $_{\text{IN}}$ = (mass of gold) $_{\text{OUT}}$

$$
C_{01} + C_{02} + A_0 = A_1 + A_2 + C_1 + C_2
$$

$$
C_{01} + C_{02} = A_1 + C_1 + C_2
$$
...(5)

Gold balance over Step 1:

(mass of gold) $_{\text{IN}}$ = (mass of gold) $_{\text{OUT}}$ $C_{01} = A_1 + C_1$...(6)

Percentage gold recovery over Step I:

(mass of gold in coal/oil phase)/(mass of gold in original ore sample) $*$ 100

Therefore:

% Au recovery in coal/oil phase for Step 1 =
$$
(A_1/C_{01}) * 100
$$

= $(C_{01} - C_1)/C_{01} * 100$... (7)

The same was argued for Step 2, which gave rise to the following equation:

% Au recovery in coal/oil phase for Step 2 = $(C_{02} - C_2)/C_{02}$ * 100 ...(8)

The Overall gold recovery percentage was calculated as follows:

(total mass of gold in agglomerates)/(mass of gold in original ore sample) • 100

Overall percentage gold recovery in coal/oil phase = $[A_1/(C_{01} + C_{02})]$ * 100 ...(9)

The analyses were done on either the GTA or ICP, whereby values for C_{01} , C_{02} , C_1 , C_2 and A_t were actually measured and hence, values of A_1 and A_2 were obtained by calculation.

EXPERIMENTAL CONDITIONS AND RAW DATA

CHAPTER 4

Experiment 4.1 - Analysis of synthetic ore mixture on GTA

The exact amount of ore to be analysed on the GTA is calculated, as a straight lined calibration curve is only found below concentrations of 0.04 ppm (see section 4.4). Therefore, the exact sample masses and volumes in which these are to be prepared are vital for accurate analysis. As 0.04 ppm represents 0.04 mg Au per litre, it suggests that a 100ml volumetric flask should not contain more than 0.004 mg gold. Therefore, based on the theoretical gold load of 7 g/ton, it was calculated that a 0.571 g ore need to be weighed out in order for the maximum amount of gold present in the ore sample not to exceed 0.004 mg and samples are then, after dissolution in *aqua regia* and filtration diluted in 100 ml volumetric flasks. By use of a microsyringe, $20 \mu l$ or each sample is 'injected' into the stationary graphite tube in the GTA, after which analysis proceeded. All samples were representative to the entire sample mass. weighed out by spreading the sample mass out and deviding it into 4 blocks, of which the same mass (adding up to approximately 0.5 g ore) was taken from each "block".

Experiment 4.2 - CGA under Standard Conditions (GTA analysis)

The Standard Conditions are stipulated below and are based on the ratios determined in section 4.2: coal:oil = 100:20 (by volume); coal:ore = 1:1 (by mass) and water:ore = 135 ml water/4 g gold ore. A single batch experiment was performed under the following conditions:

- 4.17 g synthetic ore (7g/t)
- 4.17 g coal \overline{a}
- I ml Ethane Oleate
- 10 ml of a I g/I 00 m1 solution of Potassium Amyl Xhantate (PAX)
- 125 ml of distilled water
- Contact of the coal/oil phase and ore slurry was performed on a laboratory scale mechanical shaker for 50 minutes.
- Separation of the coal/oil phase was done by scraping the agglomerates from the surface of the aqueous solution.
- All samples (coal and ore) were dried overnight at 80 \degree C and weighed out into crucibles the following morning after which they were ashed for 7 hours at $700 °C$.
- Three batch experiments were performed at once. It was at first assumed that the mass balance had been completed, therefore. only using the coal-oil samples

(samples 1-3) and representative ore (samples 4-6, not exceeding 0.5 g. see Exp. 4.1) samples weighed out previously to determine the exact amount of gold originally present in the ore and the exact amount of gold that has been recovered into the coal phase.

- After dissolution into *aqua regia,* the coal samples (samples 1-3) were diluted into \blacksquare 250 ml flasks and the ore samples (samples 4-6) into 100 rnl flasks in order to ensure that the concentrations of each does not exceed 0.04 mg/l.
- Analysis were performed on the Graphite Tube analyser (GTA). $\overline{}$

EXPERIMENT 4.1

Determination of Au loading on synthetic ore of theoretical loading: 7 g/t.

CALIBRATION CURVE FOR GTA

Abs. $=$ slope $*$ mass Au $+$ intercept

RESULTS

Average gold loading on ore: 6.038926 g Au/t ore

 55

EXPERIMENT 4.2 - CGA under Standard Conditions

SAMPLE MASSES

CALIBRATION CURVE FOR GTA

Standard Absorbance readings Standard I 0.187 0.187 0.165 0.167 *0.177* 0.191 0.183 Standard 2 0.4 0.391 0.393 - - - - - -Standard ³ 0.737 0.712 *0.724* - - - -

 $Abs. = Slope*mass Au + Intercept$

Average Au loading on ore 6.509368 g Au/t ore

Mass of gold per single ore samp 0.027144 mg Au/ 4.17 g ore

EXPERIMENTAL CONDITIONS AND RAW DATA

CHAPTER 5

Experiment 5.1 - Contacting times (GTA analysis)

Three batch experiments (runs 1,2,3) were prepared in the exact same manner, after which each was subjected to different contact times:

- Run 1 10 minutes
- Run 2 20 minutes
- Run 3 30 minutes

The experimental conditions for each batch test were as follows:

- \overline{a} 4.17 g synthetic ore (7g/t),
- 4.17 g coal, \overline{a}
- 1 ml Ethane Oleate,
- 10 ml of a $1g/100$ ml PAX solution,
- 125 ml distilled water, \overline{a}
- contact was done by shaking,
- separation was done by scrapng the coal-oil phase from the surfacc,
- all samples were dried overnight at 80 \degree C and
- ashed at 700 $^{\circ}$ C for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1,2.3 (coal-oil agglomerates) - 250 ml flasks and samples 4,5.6 (ore samples) - lOO ml flasks.
- The ore samples indicated on the information sheet (4.5.6) were representative and analysed for the original gold loading, whereas the mass balance was assumed to be completed and the ore samples used in the actual experiment were disregarded, as the filtration procedure of the fine material was to time consuming without the use of a vacuum pump, which was not available at that stage.
- Analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.2 - Mode of Contact: Stirring (GTA analysis)

- 4.17 g synthetic ore (7g/t), \overline{a}
- 4.17 g coal, \overline{a}
- \overline{a} I ml Ethane Oleate,
- IO ml of a Ig/I OOml PAX solution,
- 125 m1 distilled water. \overline{a}
- contact was done by **stirring** for 50 minutes. using a magnetic stirrer of \overline{a} appropriate size, whereas the speed of the stirrer was set on maximum speed.
- separation was done by scrapng the coal-oil phase from the surface.
- all samples were dried overnight at 80 °C and $\ddot{}$
- ashed at 700 $^{\circ}$ C for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1,2,3 (coal-oil agglomerates) - 250 ml flasks and samples 4,5,6 (ore samples) - 100 ml flasks.
- The ore samples (4.5.6) were representative and analysed for the original gold loading, whereas the mass balance was assumed to be completed and the ore sample used in the actual experiment were disregarded, as the filtration procedure of the fine material was to time consuming without the use of a vacuum pump, which was not available at that stage.
- Analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.3 - Mode of Contact: Rolling bottle technique (GTA analysis)

- \overline{a} 4.17 g synthetic ore $(7g/t)$,
- 4.17 g coal, \overline{a}
- 1 ml Ethane Oleate, \overline{a}
- 10 ml ofa Ig/IOOml PAX solution,
- 125 ml distilled water,
- contact was done by the rolling bottle technique for 50 minutes, using a set of rollers, consisting of a stationary and a moving shaft, which provided the rolling action. The experiments was performed under maximum speed.
- separation was done by scrapng and sieving the coal-oil phase from the surface, as the size of the agglomerates were larger than usual and required the use of a sieve for effective and complete separation
- all samples were dried overnight at 80 \degree C and
- ashed at 700 \degree C for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1,2.3 (coal-oil agglomerates) - 250 ml flasks and samples 4,5,6 (ore samples) - 100 ml flasks.
- The ore samples (4.5.6) were representative and analysed for the original gold loading, whereas the mass balance was assumed to be completed and the ore sample used in the actual experiment were disregarded. as the filtration procedure of the fine material was to time consuming without the use of a vacuum pump, which was not available at that stage.
- Analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.4 - The coal:ore ratio (GTA analysis)

The coal:ore ratio was increased from 1:1 (coal:ore) to 1.5:1. using 6,255g coal together with 1.5 ml oil instead of 4.17 g coal and I ml oil.

- 4.17 g synthetic ore $(7g/t)$, **.**
- 6.255 g coal, \overline{a}
- **1.5 m)** Ethane Oleate, \overline{a}
- 10 ml ofa Ig/lOOml PAX solution, \blacksquare
- 125 ml distilled water, \overline{a}
- contact was done by shaking for 50 minutes, $\overline{}$
- separation was done by scrapng the coal-oil phase from the surface, \overline{a}
- all samples were dried overnight at 80 °C and \overline{a}
- ashed at 700 $\mathrm{^{\circ}C}$ for 7 hours,
- dilutions were done *in* the following volumetric flasks: samples 1-6 *(coal-oil* agglomerates) - *250* ml flasks and samples 7,8,9 (ore samples) - 100 ml flasks.
- The ore samples (7,8,9) were representative and analysed for the original gold loading.
- Analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.5 - The coal:oil ratio (GTA analysis)

Increasing the coal:oil ratio from 100:20 (by volume) to 100:40, therefore using 2 ml of Ethane Oleate instead of 1 ml.

- 4.17 g synthetic ore $(7g/t)$
- 4.17 g coal \mathbf{r}
- **2 ml Ethane Oleate**
- 10 ml of a $1g$. \vee 100ml solution of PAX
- 125 ml of distilled water, \sim
- contact was done by shaking for 50 minutes. \mathbf{r}
- separation was done by scrapng,
- All samples were dried overnight at 80 $^{\circ}$ C and
- ashed at 700 \degree C for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1.2,3 (coal-oil agglomerates) - 250 ml flasks and samples $4,5.6$ (ore samples) - 100 ml flasks.
- The ore samples (4,5,6) were representative and analysed for the original gold loading.
- Analysis were performed on theGraphite Tube analyser (GTA).

Experiment 5.6 - The Water:ore ratio (GTA analysis)

The amount of water was decreased by approximately half. from 125 ml to 75 ml.

- 4.1 7 g synthetic ore *Oglt)* L,
- 4.17 g coal \overline{a}
- I ml Ethane Oleate $\ddot{}$
- 10 ml of a I*gll* OOml PAX solution,
- 75 **ml distilled water,**
- contact was done by shaking for 50 minutes. \overline{a}
- separation was done by scrapng, \overline{a}
- All samples were dried overnight at 80 $^{\circ}$ C and
- ashed at 700°C for 7 hours, \blacksquare
- dilutions were done in the following volumetric flasks: samples 1.2,3 (coal-oil \blacksquare agglomerates) - 250 ml flasks and samples 4.5.6 (ore samples) - 100 ml flasks.
- The ore samples (4,5,6) were representative and analysed for the original gold $\overline{}$ loading.
- Analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.7 - Volume of PAX (GTA analysis)

Doubled the volume of PAX from 10 to 20 ml of $1g/100$ ml solution.

- 4.17 g synthetic ore *(7g1t)* $\bar{\mathbf{u}}$
- 4.l7gcoal \overline{a}
- I ml Ethane oleate \mathbf{r}
- 20 ml of a 1 g/100ml solution of PAX
- 125 ml of distilled water,
- contact was done by shaking for 50 minutes,
- separation was done by scraping, L.
- All samples were dried overnight at *80°C* and L
- ashed at *700°C* for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1.2,3 (coal-oil agglomerates) - 250 ml flasks and samples 4,5,6 (ore samples) - 100 ml flasks.
- The ore samples (4,5,6) were representative and analysed for the original gold loading.
- All analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.8 - Concentration of PAX (GTA analysis)

- Doubled the concentration of PAX from a $1g/100$ ml solution to $2g/100$ ml $\ddot{}$ solution, still using 10 m!.
- 4.17 g synthetic ore $(7g/t)$
- 4.17 g coal
- 1 ml Ethane oleate
- 10 ml ofa 2 g/lOOml solution of PAX
- 125 ml of distilled water,
- contact was done by shaking for 50 minutes.
- separation was done by scraping, \overline{a}
- All samples were dried overnight at *80°C* and $\ddot{}$
- ashed at *700°C* for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1.2,3 (coal-oil agglomerates) - 250 m! flasks and samples 4,5,6 (ore samples) - 100 m! flasks.
- The ore samples $(4,5,6)$ were representative and analysed for the original gold loading.
- All analysis were performed on the Graphite Tube Atomiser (GTA).

Experiment 5.9 - Type of oil: Rando oil, HD 68, Caltex (GTA analysis)

All the conditions were as for the standard experiment, whereas Rando oil was used instead of Ethane Oleate.

- 4. I7 g synthetic ore *(7g1t)* \overline{a}
- \overline{a} 4.17 g coal,
- 1 ml Rando oil, MD 68, Caltex, \overline{a}
- 10 ml of a 1 $g/100$ ml solution of PAX,
- contact was done by shaking for 50 minutes,
- separation was done by scraping,
- All samples were dried overnight at 80°C and \overline{a}
- ashed at 700°C for 7 hours,
- dilutions were done in the following volumetric flasks: samples 1,2,3 (coal-oil agglomerates) - 250 ml flasks and samples 4,5,6 (ore samples) - 100 ml flasks.
- The ore samples (4,5,6) were representative and malysed for the original gold loading.
- All analysis was performed on the Graphite Tube analyser (GTA).

Experiment $5.10a$ - The mineralogy of the ore (GTA analysis)

All conditions where kept as for the standard experiment. whereas a real ore sample was used instead of the synthetic ore mixture.

- 4.17 g ore, a sample received from the Western Area Goldmine containing a \overline{a} theoretical gold loading of 4.37 g/t, and by \overline{GTA} analysis was found to be approximately 2.037 g/t.
- 4.17 g coal,
- I ml Ethane Oleate, \overline{a}
- 10 ml ofa I *gllOO* ml solution of PAX, \overline{a}
- contact was done by shaking for 50 minutes, \overline{a}
- separation was done by scraping, \overline{a}
- All samples were dried overnight at 80 °C and
- ashed at 700°C for 7 hours, \overline{a}
- dilutions were done in the following volumetric flasks: samples 1.2,3 (coal-oil \overline{a} agglomerates) - 100 m1 flasks and samples 4,5,6 (ore samples) - lOO ml flasks.
- The ore samples $(4,5,6)$ were representative and analysed for the original gold loading.
- Analysis was performe on the Graphite Tube \hat{A} tomiser (GTA)

Experiment 5.1 Ob - Au leaching experiment (GTA analysis)

A I L perspex reactor with the following dimentions were used: Internal diameter of 11 cm, height of 15 cm with three evenly spaced baffles of width 1 cm each.

- A flat blade impeller of width 6 cm and height 5 cm, was used for agitation, which \overline{a} was driven by an electric motor at a constant speed of 360 rpm to ensure that the ore particles were kept in suspension.
- 10 g of the industrial ore used in Esp. 5.10a was used.
- The leachant was a 100 ppm CN⁻ solution of which a 1000 ml was prepared and added to the ore inside the reactor. Potassium cyanide (KCN) was used.
- The pH was kept at a constant value of **11,** using a 10 mlar solution of sodium hydroxide (NaOH) which is a 400 g/L solution.
- Agitation was continued and samples drawn at the following time intervals: 30 minutes, 6,23,27 and 29 hours. The adsorption of each samples was read and the equilibrium value used to delermine the concentration of dissolved gold present in the ore sample.
- All analysis was performed on the Graphite Tube Atomiser (GTA)

EXPERIMENT 5.1 - Contacting times

SAMPLE MASSES

Calibration curve data for ore samples $1,2,3$

Calibration curve data for coal samples $4,5,6$

Average gold 100lding on ore 7.416644 g Au/I ore

Mass gold per single ore sample 0.030927 mg Au/4.17 g ore

EXPERIMENT 5.2 - Mode of contact: Stirring

SAMPLE MASSES

CALIBRATION CURVE DATA

979738

Standard Absorbance readings

Abs. = $Slope*mass Au + Intercept$

Slope

Average Au loading on ore 5.336603 g Au/t ore

Mass of gold per single ore samp 0.022254 Ing Au/ 4.17 g ore

EXPERIMENT 5.3 - Mode of contact: Rolling bottle

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. = $Slope*mass Au + Intercept$

Average Au loading on ore 5.98721 g Au/t ore

Mass of gold per single ore samp 0.024967 mg *Ani* 4.17 gore

EXPERIMENT 5.4 - The coal:ore ratio

SAMPLE MASSES

Calibration eurve data for samples 1,2,3 and 7,8,9

Calibration curve data for samples 4.5,6

Average Au loading on ore 5.386517 g Au/t ore

Mass of gold per single ore samp 0.022462 mg Au/ 4.17 g ore

AVERAGE Au recovery % | 41.99821 %

EXPERIMENT 5.5 - The coal:oil ratio

SAMPLE MASSES

Calibration curve data for samples 1,2,3

Calibration curve data for samples 4,5,6

Average Au loading on ore 5.267063 g Ault ore

EXPERIMENT 5.6 - The water:ore ratio

SAMPLE MASSES

Calibration curve data for samples $1,2,3$

Calibration curve data for samples 4,5,6

Average Au loading on ore 5.634324 g *Ault* ore

Mass of gold per single ore samp 0.023495 mg Au/ 4.17 g ore

EXPERIMENT 5.7 - Volume of PAX

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. ⁼ Slope*mass Au + Intercept

Average Au loading on ore 5.311986 g Au/t ore

Mass of gold per single ore samp 0.022151 mg Au/ 4.17 g ore

EXPERIMENT 5.8 - Concentration of PAX

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. = $Slope*mass Au + Intercept$

Average Au loading on ore 5.328492 g Au/t ore

Mass of gold per single ore samp 0.02222 mg Au/4.17 g ore

EXPERIMENT 5.9 - Type of oil: Rando oil, Caltex

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. $=$ Slope*mass Au + Intercept

Average Au loading on ore 6.12137 gAu/tore

Mass of gold per single ore samp 0.025526 mg *Aul* 4.17 g ore

EXPERIMENT 5.10a - Mineralogy of the **ore**

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs.= Slope*mass Au + Intercept

Average Au loading on ore 2.031616 g Au/t ore

Mass of gold per single ore samp 0.008472 mg Au/ 4.17 g ore

$[Au]_{total} = [Au]_{dissolved} + [Au]_{free}$

% Dissolved Au 7.038733 %

EXPERIMENT 5.10b - Au Leaching experiment

CALIBRATION CURVE DATA

Abs. = $Slope*mass Au + Intercept$

RESULTS (Experiment 5.IOb)

Standards Absorbance readings

Standard 1 0.19 0.185 0.185 -
Standard 2 0.385 0.365 0.382 0.372

Standard 2 0.385 0.365 0.382
Standard 3 0.797 0.84 0.79 $|\text{Standard 3}|$ 0.797 0.84 0.79 -

EXPERIMENTAL CONDITIONS AND RAW DATA

CHAPTER 6

Experiment 6.1 - CGA on synthetic ore under optimal conditions (GTA analysis)

The Coal Gold Agglomeration (CGA) process was performed under optimised conditions, which are stipulated in section 5.4 and are as follows:

- 4.17 g synthetic gold ore (7g/t). \overline{a}
- 4.17 g coal, $\overline{}$
- I ml Ethane Oleate,
- 75 ml distilled water,
- 20 ml of a 2 g/100ml solution of collector, nl. PAX, \sim
- contact was done by stirring with the magnetic stirrer for 50 minutes, \mathbf{r}
- agglomerates were separated by scraping,
- all samples were dried overnight at 80 $^{\circ}$ C and ashed for 7 hours at 700 $^{\circ}$ C.
- Samples 1-9(coal-oil agglomerates) were diluted in 250 ml flasks and samples 10-18 (representative ore samples) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was assumed to be complete, only analysing the coal-oil agglomerates (A₁) and original ore samples (C₀), disregarding the gold content in the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA).

Experiment 6.2 - CGA on synthetic ore under optimal conditions: cheking the mass balance (GTA analysis)

The CGA experiment was performed under the exact same conditions as Experiment 6.1, whereas the ore samples (C_1) - dilutions were done in 100 ml flasks - used in the experiment was analysed together with the coal-oil agglomerate $(A₁)$ and original ore samples (C_0) in order to check the mass balance. The additional samples vere treated in the same manner as all the other samples, as discussed in the previous experiment.

Experiment 6.3 • CGA on synthetic ore under optimal conditions: cheking the mass balance (Induction Coupled Plasma analysis).

The CGA experiment was performed under the same conditions as Experiment 6.1 and 6.2, whereas analysis of all the samples: treated ore samples (C_1) , coal-oil agglomerates $(A₁)$ and original ore samples $(C₀)$ were all analysed on the Inductively Coupled Plasma (ICP). All samples were treated as before. After dissolution in *aqua regia* and filtration, the filtrate of each and every sample was diluted into 100 ml flasks, adding enough hydrochloric acid to total a concentration of 10% hydrocloric acid, which prevented that the gold in solution would not presipitate out.

Experiment 6.4 - Oil type: 100 Solvent Neutral, Caltex (GTA analysis)

The CGA was performed under optimal conditions, using a Caltex oil nl. 100 Solvent Neutral, instead of Ethane Oleate. Eight different experiments were done and the coal-oil agglomerates (A_1) and original ore samples (C_0) where analysed for gold. The experimental conditions were as follows:

- 4.17 g synthetic gold ore $(7g/t)$. \overline{a}
- 4.17 g coal, \overline{a}
- **1 mllOO Solvent Neutral Caltex oil**
- 75 ml distilled water,
- 20 ml of a 2 g/100ml solution of collector, nl. PAX. \overline{a}
- contact was done by stirring with the magnetic *stirrer* for 50 minutes, \overline{a}
- agglomerates were separated by scraping, \overline{a}
- all samples were dried overnight at 80 \degree C and ashed for 7 hours at 700 \degree C,
- Samples 1-8 (coal-oil agglomerates) were diluted in 250 ml flasks and samples 9-17 (representative ore samples) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was assumed to be complete, only analysing the coal-oil agglomerates (A_1) and original ore samples (C_0) , disregarding the gold content in the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA).

Experiment 6.5 - Oil type: 150 Solvent Neutral, Caltex (GTA analysis)

The CGA was perfonned under optimal *conditions,* using a Caltex oil nl. 150 Solvent Neutral, instead ofEthane Oleate. Eight *different* experiments were done and the coal-oil agglomerates (A_1) and original ore samples (C_0) where analysed for gold. The experimental conditions were as follows:

- 4.17 g synthetic gold ore $(7g/t)$.
- 4.17 g coal, \overline{a}
- **1 mllSO Solvent Neutral Caltex oil** \overline{a}
- 75 ml distilled water, \overline{a}
- 20 ml of a 2 $g/100$ ml solution of collector, nl. PAX, J.
- cvntaet was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates were separated by scraping,
- all samples were dried overnight at 80 \degree C and ashed for 7 hours at 700 \degree C. \overline{a}
- Samples 1-3 (coal-oil agglomerates) were diluted *in* 250 ml flasks and samples \overline{a} 4-6 (representative ore samples) in 100 ml flasks. Each sample was filtered

into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.

- The mass balance was assumed to be complete, only analysing the coal-oil \overline{a} agglomerates (A_1) and original ore samples (C_0) , disregarding the gold content in the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA). $\ddot{}$

Experiment 6.6 - Oil type: 450 Solvent Neutral, Caltex (GTA analysis)

The CGA was performed under optimal conditions, using a Caltex oil nl. 450 Solvent Neutral, instead of Ethane Oleate. Eight different experiments were done and the coal-oil agglomerates (A_1) and original ore samples (C_0) where analysed for gold. The experimental conditions were as follows:

- 4.17 g synthetic gold ore $(7g/t)$.
- 4.17 g coal, \overline{a}
- 1 ml450 Solvent Neutral Caltex oil
- 75 ml distilled water,
- 20 ml of a 2 $\varrho/100$ ml solution of collector, nl. PAX,
- contact was done by stirring with the magnetic stirrer for 50 minutes, \overline{a}
- agglomerates were separated by scraping, \overline{a}
- all samples were dried overnight at 80 °C and ashed for 7 hours at 700 °C.
- Samples 1-3 (coal-oil agglomerates) were diluted in 250 ml flasks and samples 4-6 (representative ore samples) in lOO ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was assumed to be complete. only analysing the coal-oil L. agglomerates (A_1) and original ore samples (C_0) , disregarding the gold content in the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA).

Experiment 6.7 - Oil type: Ethane Oleate (lCP analysis)

The exact same experimental conditions as used in Experiment 6.1, whereas analysis was performed by ICP instead of GTA was followed:

- 4.17 g synthetic gold ore $(7g/t)$. \overline{a}
- 4.17 g coal, \overline{a}
- **1 ml** Ethane Oleate, $\ddot{}$
- 75 m! distilled water, \mathbf{r}
- 20 ml of a 2 $g/100$ ml solution of collector, nl. PAX,
- contact was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates were separated by scraping.
- all samples were dried overnight at 80 °C and ashed for 7 hours at 700 °C.
- Samples 1-3 (coal-oil agglomerates, A_1) were diluted in 100 ml flasks and samples 4-6 (treated ore samples, C_1) 7-9 (representative ore samples, C_0) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates (A_1) and original ore samples (C_0) as well as the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (ICP).

Experiment 6.8 - Oil type: 100 Solvent Neutral, Caltex (lCP analysis)

The exact same experimental conditions as used in Experiment 6.4, whereas analysis was performed by lCP instead of GTA was followed:

- 4.17 g synthetic gold ore (7g/t).
- $\overline{}$ 4.17 g coal,
- 1 ml 100 Solvent Neutral, Caltex
- 75 ml distilled water, $\ddot{}$
- 20 ml of a 2 g/100ml solution of collector, nl. PAX, \overline{a}
- contact was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates were separated by scraping, \overline{a}
- all samples were dried overnight at 80 $^{\circ}$ C and ashed for 7 hours at 700 $^{\circ}$ C, \overline{a}
- Samples 1-3 (coal-oil agglomerates, A_1) were diluted in 100 ml flasks and samples 4-6 (treated ore samples, C_1) 7-9 (representative ore samples, C_0) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates (A_1) and \overline{a} original ore samples (C_0) as well as the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (lCP).

Experiment 6.9 - Oil type: 150 Solvent Neutral, Caltex (lCP analysis)

The exact same experimental conditions as used in Experiment 6.5. whereas analysis was performed by lCP instead of GTA was followed:

- 4.17 g synthetic gold ore $(7g/t)$.
- 4.17 g coal,
- 1 ml 150 Solvent Neutral, Caltex
- 75 ml distilled water,
- 20 ml ofa 2 g/IOOml solution of collector, nl. PAX.
- contact was done by stirring with the magnetic stirrer for 50 minutes. \overline{a}
- agglomerates were separated by scraping, \overline{a}
- all samples were dried overnight at 80 \degree C and ashed for 7 hours at 700 \degree C.
- Samples 1-3 (coal-oil agglomerates, A_1) were diluted in 100 ml flasks and samples 4-6 (treated ore samples, C_1) 7-9 (representative ore samples, C_0) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates $(A₁)$ and original ore samples (C_0) as well as the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (lCP).

Experiment 6.10 - Oil type: 450 Solvent Neutral, Caltex *(ICP* analysis)

The exact same experimental conditions as used in Experiment 6.6, whereas analysis was performed by ICP instead of GTA was followed:

- 4.17 g synthetic gold ore $(7g/t)$.
- 4.17 g coal,
- **1 ml450 Solvent Neutral,** Caltex
- 75 ml distilled water,
- 20 ml of a 2 g/lOOml solution of collector, n1. PAX,
- contact was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates were separated by scraping,
- all samples were dried overnight at 80°C and ashed for 7 hours at 700°C, \blacksquare
- Samples 1-3 (coal-oil agglomerates, A_1) were diluted in 100 ml flasks and samples 4-6 (treated ore samples, C_1) 7-9 (representative ore samples, C_0) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates (A_1) and \overline{a} original ore samples (C_0) as well as the actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (lCP).

Experiment 6.11 - Recycling the ore phase (GTA analysis)

The CGA experiment was performed under optimal conditions, as in Experiment 6.1, whereas the ore phase was separated and recycled. This implies the use of a "fresh" sample of agglomerates (A₂) on the already treated ore phase (C₁) after separating the first coal-oil phase (A_1) . This procedure was followed on 6 different runs of which the experimental conditions were as follows:

- 4.17 g synthetic gold ore $(7g/t)$,
- 4. I7 g coal, \overline{a}
- 1 ml Ethane Oleate,
- 75 ml distilled water, \overline{a}
- 20 ml of a 2 $g/100$ ml solution of collector, nl. PAX, \overline{a}
- contact was done by stirring with the magnetic stirrer for 50 minutes.
- agglomerates (A_1) , Samples 1-6 were separated by scraping and set aside to be dried, ashed and finally analysed.
- The ore was re-used by preparing an additional coal-oil agglomerate sample (A_2) from 4.17 g coal and I ml Ethane Oleate and contacting the two phases for 50 minutes by stirring. This second coal-oil phase (A_2) , Samples 13-18, was then separated from the slurry be scraping and set aside.
- All samples were dried overnight at 80 $^{\circ}$ C and ashed for 7 hours at 700 $^{\circ}$ C,
- Samples 1-6 (coal-oil agglomerates, A_1) and 13-18 (coal-oil agglomerates, A_2) L. were diluted in 250 ml flasks and samples $7-12$ (representative ore samples, C_0) in 100 ml flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was assumed to be complete, only analysing the coal-oil $\overline{}$ agglomerates (A_1 and A_2)and original ore samples (C_0), disregarding the gold content in the actual ore samples $(C₁)$ used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA).

Experiment 6.12 - Recycling the ore phase: Mass balance cneck (GTA analysis)

The CGA experiment were performed under the exact same conditions as Experiment 6.12, whereas the only difference was the analysis of the treated ore sample (C_1) in order to check the mass balance. Three runs were performed under the following conditions:

- 4.17 g synthetic gold ore $(7g/t)$,
- 4.17 g coal, \overline{a}
- I ml Ethane Oleate,
- 75 ml distilled water,
- 20 ml of a 2 $g/100$ ml solution of collector, nl. PAX,
- contact was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates (A_1) , Samples 1-3 were separated by scraping and set aside to be dried, ashed and finally analysed.
- The ore was re-used by preparing an additional coal-oil agglomerate sample (A*z)* from 4.17 g coal and 1 ml Ethane Oleate and contacting the two phases for 50 minutes by stirring. This second coal-oil phase (A_2) , Samples 4-6, was then separated from the slurry be scraping and set aside.
- All samples were dried overnight at 80 $^{\circ}$ C and ashed for 7 hours at 700 $^{\circ}$ C.
- Samples 1-3 (coal-oil agglomerates, A_1) and 4-6 (coal-oil agglomerates, A_2) \overline{a} were diluted in 250 ml flasks and samples 7-9 (representative ore samples, C_0) and 10-12 (treated ore samples, C_1)in 100 rai flasks. Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by only analysing the coal-oil agglomerates (A_1) and A_2) as well as the original ore samples (C₀) and actual ore samples (C₁) used during experimentation.
- Samples were analysed on the Graphite Tube Analyser (GTA).

Experiment 6.13 - Recycling the coal-oil phase: Mass balance check *(ICP* analysis)

The CGA experiment was performed under the exact same conditions as Experiment 6.1, whereas the coal-oil phase was recycled. This implies the contact of a "fresh" ore sample (C_2) with the agglomerates (A_1) that have already been used in a CGA experiment. Three runs were performed under the following conditions:

- 4.17 g synthetic gold ore $(7g/t)$, \overline{a}
- 4.17 g *coal,* \overline{a}
- I ml Ethane Oleate,
- 75 mI distilled water, \overline{a}
- 20 ml of a 2 g/100ml solution of collector, nl. PAX, \overline{a}
- contact was done by stirring with the magnetic stirrer for 50 minutes,
- agglomerates (A_1) , Samples 4-6 were separated by scraping and set aside to be \overline{a} contacted with a fresh ore sample.
- The second ore slurry was prepared from 4.17 g synthetic ore, 75 ml distilled water and the collector was added in the same volume and concentration (20 ml of a 2g/1 00 mI solution) as before and contacted with the *already used* coal-oil sample $(A₁)$ for 50 minutes by stirring.
- Once again separation of the coal-oil phase was done by scraping. The first batch \overline{a} of ore samples, Samples 1-3, C_1 , and the second batch of ore samples, Samples 7-9, C₂ together with the coal-oil agglomerates separated after recycling, Samples 4-6, A_t , was dried overnight dried overnight at 80 °C and ashed for 7 hours at 700 $^{\circ}C$
- Samples 4-6 (coal-oil agglomerates, A_t) and samples 10-12 (representative ore . samples, C_0) and 1-3 (first batch of treated ore samples, C_1) and finally samples 7-9 (second batch of treated ore samples, C_2) where then all filtered and diluted into 100 ml flasks. Furthermore, each sample was filtered into the flasks. adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates (At) as well \blacksquare as the original ore samples (C_0) and actual ore samples (C_1, C_2) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (ICP).

Experiment 6.14 - Optimised CGA on Industrial ore:checking the mass balance (ICP analysis)

The optimised CGA test was performed on an Industrial ore sample instead of using the synthetic ore sample, whereas the mass balance was checked as well. The industrial ore sample was the same ore as used in Experiment 5.10a and 5.10b. Three runs were performed under the following conditions:

4.17 g of an industrial gold ore as received from the Western Area Goldmine in \overline{a} South-Africa,

- 4.17 g coal, \overline{a}
- 1 ml Ethane Oleate, \overline{a}
- 75 ml distilled water, \overline{a}
- 20 ml of a 2 g/100ml solution of collector, nl. PAX, \overline{a}
- contact was done by stirring with the magnetic stirrer for 50 minutes, \overline{a}
- agglomerates were separated by scraping, \overline{a}
- all samples were dried overnight at 80 °C and ashed for 7 hours at 700 °C, ÷,
- Samples 1-3 (coal-oil agglomerates, A_1) were diluted in 100 ml flasks as well as \overline{a} samples 4-6 (representative ore samples, C_0) and samples 7-9 (treated ore samples, C_1). Each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.
- The mass balance was checked by analysing the coal-oil agglomerates $(A₁)$ and original ore samples (C_0) as well as actual ore samples (C_1) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (ICP).

Experiment 6.15 - CGA on Industrial ore: Recycling the coal-oil phase (ICP analysis)

The CGA experiment was performed under the exact same conditions as Experiment 6.14, whereas the coal-oil phase was recycled. This implies the contact of a "fresh" ore sample (C_2) with the agglomerates (A_1) that have already been used in a CGA experiment. Three runs were performed under the following conditions:

4.17 g Industrial gold ore,

- 4.17 g coal, $\ddot{}$
- I ml Ethane Oleate, \overline{a}
- 75 ml distilled water, $\ddot{}$
- 20 ml of a 2 $g/100$ ml solution of collector, nl. PAX,
- contact was done by stirring with the magnetic stirrer for 50 minutes, \overline{a}
- agglomerates (A_1) , Samples 4-6 were separated by scraping and set aside to be $\ddot{}$ contacted with a fresh ore sample.
- The second ore slurry was prepared from 4.17 g synthetic ore. 75 ml distilled water and the collector was added in the same volume and concentration (20 ml of a 2g/1 00 rnJ solution) as before and contacted with the *already used* coal-oil sample $(A₁)$ for 50 minutes by stirring.
- Once again separation of the coal-oil phase was done by scraping. The first batch $\ddot{}$ of ore samples, Samples 1-3, C_1 , and the second batch of ore samples, Samples 7-9, C_2 together with the coal-oil agglomerates separated after recycling. Samples 4-6, A_t , was dried overnight dried overnight at 80 °C and ashed for 7 hours at 700 $^{\circ}C$
- Samples 4-6 (coal-oil agglomerates, A_t) and samples 10-12 (representative ore $\ddot{}$ samples, C_0) and 1-3 (first batch of treated ore samples, C_1) and finally samples 7-

9 (second batch of treated ore samples, C_2) where then all filtered and diluted into 100 ml flasks. Furthermore, each sample was filtered into the flasks, adding enough hydrochloric acid (HCL) to ensure that the samples were diluted into a 10% solution of HCL.

- The mass balance was checked by analysing the coal-oil agglomerates (At) as well \overline{a} as the original ore samples (C_0) and actual ore samples (C_1, C_2) used during experimentation.
- Samples were analysed on the Inductively Coupled Plasma (lCP).

Experiment 6.16 - Analysis by MATTEK

These samples were sent to MATTEK (Materials Science and Technology) in Pretoria for XRD analysis to determine the surface characteristics of the ores and coal-oil agglomerates before and after performing CGA and were marked samples 1-12, consisting of the following "groups":

- Samples 1-3: **Ore** samples **before** being subjected to the CGA process. These samples were analysed to determine all the minerals present in the ore matrix. (C_0)
- Samples 4-6: **Ore** samples **after** being used in a CGA experiment. These samples were analysed to determine if any minerals were "lost" to the coal-oil phase during the contact procedure. (C_1)
- Samples 7-9: **Caol-oil** samples **after** being used in a CGA experiment. The qualitative analysis was done to determine if any of the minerals present in the ore phase was transfered to the surface of the coal-oil agglomerates during the contact procedure. (A_1)
- Samples 10-12: **Coal** samples that were taken **before** performing CGA. These samples were analysed to determine the minerals that were originally present on the coal so that a comparison could be made to samples 7-9, leading to answers about the minerals present before and after performing the CGA experiments. $(A₀)$

Three batch experiments were performed under the following optimised experimental conditions, leading to samples 4-6 and 7-9:

- 4.17 gore
- 4.17 g coal
- 75 ml water
- 20 ml ofa 2g/100 ml solution PAX
- I ml Ethane Oleate
- contact was done by stirring for 50 minutes \overline{a}
- separation was done by scraping the agglomerates from the surface of the aqeous medium
- all samples were dried overnight at 80 \degree C and
- ashed at 700 °C for 7 hours.
- samples 1-3 and 10-12 were weighed out before performing the CGA \overline{a} experiment and were not treated in any way, as these samples represent initial conditions.
- samples 4-6 and 7-9 were all dried and ashed, after which they were packed and sent to MATTEK for XRD analysis.

The raw data is the diffractograms of each sample, as marked and indicates which minerals and other materials were present at the surface of each sample. Note that the analysis is strictly qualitative in nature and only indicates which minerals are present in each phase before and after performing a Coal Gold Agglomeration experiment.

Experiment 6.17 - Mercury Amalgamation (lCP analysis)

For practical purposes, the following ratios and experimental conditions to perform an amalgamation experiment were taken from Costa *et al.* (1997), pg 1408 which are the standard conditions used at the Centre for Mineral Techrology (CETEM) in Brazil:

- 30% solids (by weight)
- A mercury: ore ratio of 1:20
- A sodium hydroxide (NaOH):ore ratio of 1:I000
- Conditioning at 2 hours in a horizontal cylinder ratating at 20 rpm.

A synthetic gold ore of grade 7 g/t was prepared as follows: $-44 \mu m$ gold particles (the same as used in the CGA experiments) were thoroughly mixed onto a 1 kg silica sample in the size of - $106 \mu m$. Pure mercury was obtained from the chemical store. The sodium hydroxide (NaOH) was added to optimise the surface properties of the mercury with respect to the feed charge in the closed "vessel".

The experiments were performed on the basis of 20 and 10 gram (Exp. 6.18) ore samples. From the above ratios, the experimental conditions for each run was as follows:

- 20 synthetic gold ore, as prepared for mercury amalgamation *Oglt).*
- 50 ml of distilled water, \overline{a}
- 0.02 g sodium hydroxide (NaOH), \overline{a}
- I g Mercury (Hg). $\ddot{}$
- The distilled water and NaOH was mixed to produce th 0.1 g N \cdot OH $/$ 250 ml water solution after which the synthetic ore and mercury were added to the solution into the 200 ml bottles (the same bottles were used for the CGA experiments), as 6 experiments were performed at once.
- The bottles were placed on the same roller as used during the rolling bottle CGA \overline{a} experiment and were contacted for 2 hours at approximately 200 rpm, which is the minimum speed of rotation for this piece of equipment.
- As the density of mercury is much larger than for the ore, it was possible to $\ddot{}$ separate the fine mercury (amalgam) droplets for each experiment by utilising a separation funnel, whereas the remaining ore was treated with *aqua regia* and

filtered into 100 ml flasks for further analysis. All the 100 ml flasks contained enough HCl to ensure that the filtrate was diluted into a 10% solution of HCl. The amalgam was treated with *aqua regia* as well. By utilising round bottom flasks, heating mantels and distillation columns in a vacuum cupboard and cartridge masks fitted with special mercury vapour cartridges, the risk of mercury vapour escaping and poisening the surrounding area and persons were limited. The vacuum cupboard was fitted with a perspex container into which mercury spillages could be caught up. As sulfur reacts with mercury, it was kept at hand to cover any mercury spillages and as a further precaution a vacuum pump, fitted with a mercury trap was kept available to suck up spillages, is any would occur.

- The amalgam was treated with 30 ml of *aqua regia,* which was heated until most ofthe solution had vapourised, after which another 30 ml was added into the round bottom flask containing the amalgam and cooked until about 5-10 ml of the liquid remained. This solution was then filtered into a 100 ml flask contianing 10% HCI and sent for analysis.
- The remaining ore samples were treated with 25 ml *aqua regia* and the mixture \overline{a} was boiled until dry. This procedure was repeated *twice* more, whereas the last addition was only boiled until the larger percentage of toxic fumes had been removed by the vacuum in the vacuum cupboard. The remainder of*the* sample was filtered into 100 ml flasks containing 10% HCI, which was sent for analysis.

Experiment 6.18 - Mercury Amalgamation (ICP analysis)

Another amalgamation experiment was performed on the basis of 10_g of synthetic ore instead of 20g, keeping all experimental conditions the same as in Experiment 6.17 and were as follows:

- 10 g synthetic gold ore *Og/t)*
- 25 ml of distilled water,
- 0.01 g NaOH, \overline{a}
- 0.5 g mercury

All the contact and separation procedures were the same as in Experiment 6.17, whereas half the amount of *aqua regia* was used to treat the ore and amalgam samples and utilising 100 ml volumetric flasks containing 10% HCl to dilute the filtrate of each sample.

Experiment 6.19 - Measurement of the three-phase contact angles (θ)

The three-phase contact angle (θ) is the angle measured through the oil onto the gold surface in the presence of water, as illustrated in the diagram (Figure A3) below. A thin gold plate, which fitter perfectly into the base of a square vial was used as the gold surface. After the gold was inserted into the vial, it was filled with water and a microsyringe was used to carfully place an oil droplet of volume 30 μ l onto the gold surface. A moving microscope was used to measure the dimensions of the drop i.e. h and d, as seen in Figure A3, below:

The measurements were done at the Polymer Science Department of the Stellenbosch University, where the masters student herself had performed the measurements. The three phase contact angle (θ) was then calculated from the following equation:

 $\tan \theta/2 = h/r$ where: θ three-phase contact angle $(°)$ $=$ h height of the oil drop (mm) \equiv radius of the oil drop = $d/2$ (mm) $r =$

The above procedure was followed for every oil type and the three-phase contact angle calculated from the dimensions, as recorder in the table below:

Experiment 6.20 - Measurement and calculation of the interfacial tensions (γ_{23})

The interfacial tension between each oil and water was calculated from the following equation (Glasstone *et al.,* 1981):

$$
\gamma_{AB} = \gamma_B - \gamma_A \qquad \qquad \dots (6.1)
$$

where: γ_{AB}/γ_{23} = the interfacial tension between the oil and water (J/m²) γ_A = the surface tension of water/oil (J/m²) y_B = the surface tension of the oil/water (J/m²)

Since surface (and interfacial) tensions are always positive, it follows that the interfacial tension is less than that of the larger of the two surface tensions. This is because the attraction across the interface, between the molecules of one liquid and those of the other, tends to reduce the inward pull of the molecules in the surface by those of the same kind. The greater the molecular attraction between the two liquids, the lower the interfacial tension. Before the calculation could be done, it was vital to determine the surface tensions of each oil and this was measured by utilising the *cappilary rise* method.

The *capillary rise* method operates on the basis of comparing the capillary rise of the liquid in question with that of a standard liquid such as water, of known surface tension. If *h* is the rise of the liquid of surface tension γ and density ρ in a given capillary tube, and h_{θ} is the rise of a liquid in the same tube of a standard liquid, i.e. water, of density ρ_{θ} and surface tension γ_0 , it follows from equation (6.2) that:

$$
\gamma/\gamma_0 = h\rho / h_0\rho_0 \qquad \qquad \ldots (6.2)
$$

Therefore, water was chosen as the standard liquid. As the density (ρ_0) and surface tension (y_0) of water is known, the capillary rise of the liquid had to be measured. In fact, the capillary rise of each oil type was measured as welL by utilising 4 capillary tubes of the exact same diameter.

Figure $A4 - A$ diagram illustrating the measurement of the capillary rise (h)

The same volume i.e. 10 ml of all 5 liquids (water and the 4 oils) were measured out into 5 vials. The capillary tubes were inserted and after each liquid had risen to its equilibrium height, a *vernier cal iper* was used to measure the height that each liquid had risen from the surface of the liquid in the vial, see Figure A4.

The capillary rise, density and surface tension for water was as follows:

The capillary rise of each oil (h) was measured and the surface tensions (γ) of each calculated from equation 6.2. By utilising the information, it was then possible to calculate the interfacial tension (γ_{23}) between the water and each oil by utilising equation 6.1. The results are stipulated in Table *A3,* below.

Table A3 - The results from the measurements and calculations done to determine the interfacial tension of each oil with respect to water.

Example of Calculations:

The surface tension for Ethane Oleate was calculated as follows:

$$
\gamma/\gamma_0 = h\rho / h_0 \rho_0
$$

\n
$$
\gamma_{E0} = \gamma_0[h\rho / h_0 \rho_0]
$$

\n
$$
\gamma_{E0} = 7.275 \times 10^{-2} [(1.188 \times 834)/(1.442 \times 1000)]
$$

\n
$$
= 5.214 \times 10^{-2} \text{ J/m}^2
$$

The interfacial tension (y_{23}) between Ethane Oleate and water:

$$
\gamma_{AB}/\gamma_{23} = \gamma_B - \gamma_A
$$

= (7.275×10⁻²) - (4.998×10⁻²)

$$
\gamma_{23} = 2.0606 \times 10^{-2} \text{ J/m}^2
$$

SAMPLE MASSES

GOLD RECOVERY PERCENTAGES (Refer to Results, Exp. 6.1)

Calibration curve data for samples 1,2,3 and 10,11,12 (Experiment 6.1)

Calibration curve data for samples 4,5,6 and 13,14,15 (Experiment 6.1)

Calibration curve data for samples 7,8,9 and 16,17,18 (Experiment 6.1)

RESULTS (Experiment 6.1)

Average Au loading on ore

4.815433 g Au/t ore

Mass of gold per single ore sample

0.02008 mg Au/ 4.17 g ore

EXPERIMENT 6.2 - Optimised CGA on synthetic ore: cheking the mass balance (GTA analysis)

SAMPLE MASSES

Calibration curve data for samples 1,2 and 6,7,8 and 11,12 (Experimcnt 6.2)

Calibration curve data for samples $3,4,$ ^{τ} and $9,10$ and $13,14,15$ (Experiment 6.2)

RESULTS (Experiment 6.2)

Average Au loading on ore

4.224989 g Au/t ore (Average of samples 6 to 10)

Mass of gold per single ore sample

0.017618 mg Au/ 4.17 g ore

PERCENTAGE GOLD RECOVERIES (Experiment 6.2)

Au recovery $% =$ [Total m_{Au} in coal sample/ m_{Au} in single ore sample]*100

Au recovery % = [Total m_{Au} in ore sample/ m_{Au} in single ore sample]*100

Mass balance check: 45.0346 %

EXPERIMENT 6.3 - Optimised CGA on synthetic ore: cheking the mass balance (ICP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.3 - lCP analysis)

PERCENTAGE GOLD RECOVERIES (Experiment 6.3)

Au recovery % = [Total m_{Au} in coal sample/ m_{Au} in single ore sample]*100

Au recovery % = [Total m_{Au} in ore sample/ m_{Au} in single ore sample]*100

Mass balance check: 100.2181 %

EXPERIMENT 6.4 - Oil type: 100 Solvent Neutral, Caltex (GTA analysis)

 $\Delta \sim 10^{11}$

SAMPLE MASSES

.
The Motor of English Process and Process Component Process (1999), the Component Component Component Component

 \mathcal{L}_G and the second second and the second contract of the second contract

والمتعارف والمستعين والمستحيل والمستحدث

 $\mathcal{L}^{\mathcal{L}}$ and $\mathcal{L}^{\mathcal{L}}$ are the set of the set of the set of the $\mathcal{L}^{\mathcal{L}}$

GOLD RECOVERY PERCENTAGES (Refer to Results, Exp. 6.4)

EXPERIMENT 6.4 - Oil type: 100 Solvent Neutral, Caltex (GTA analysis)

SAMPLE MASSES

GOLD RECOVERY PERCENTAGES (Refer to Results, Exp. 6.4)

Calibration curve data for samples 1,2,3 and 10,11,12 (Experiment 6.4)

Calibration curve data for samples 4,5,6 and 13,14,15 (Experiment 6.4)

Calibration curve data for samples 7,8,9 and 16,17,18 (Experiment 6.4)

RESULTS (Experiment 6.4)

Average Au loading on ore

5.131166 g Au/t ore

Mass of gold per single ore sample

0.021397 mg Au/ 4.17 g ore

EXPERIMENT 6.5 - Oil type: 150 Solvent Neutral, Caltex (GTA analysis)

SAMPLE MASSES

CALIBRATION CURVE DATA

-0.0085 872869

 $Abs. = Slope*mass Au + Intercept$

Intercept Slope

RESULTS (Experiment 6.5)

Average All loading on orc 4.476617 g Au/t ore

Mass of gold per single ore samp 0.018667 mg Au/ 4.17 g ore

EXPERIMENT 6.6 - Oil type: 450 Solvent Neutral, Caltex (GTA analysis)

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. = Slope'mass **Au** + Intercept

RESULTS (Experiment 6.6)

Average Au loading on ore 4.85062 g *Ault* ore

.

Mass of gold per single ore samp 0.020227 mg Au/ 4.17 g ore

EXPERIMENT 6.7- Oil type: Ethane Oleate (ICP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.7 -ICP analysis)

Average Au load on ore

7.498667 gAu/t ore

% GOLD RECOVERIES

EXPERIMENT 6.8- Oil type: 100 Solvent Neutral, Caltex (lCP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.8 - ICP analysis)

Average Au load on ore

7.498667 gAult ore

Mass Au per single ore sample 0.031269 mg/4.17g ore \blacksquare Mass balance check: 103.7754%

% GOLD RECOVERIES

EXPERIMENT 6.9 - Oil type: 150 Solvent Neutral, Caltex (lCP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.9 - ICP analysis)

Average Au load on ore

Mass Au per single ore sample

% **GOLD RECOVERIES**

EXPERIMENT 6.10 - Oil type: 450 Solvent Neutral, Caltex (lCP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.10 - ICP analysis) $\%$ GOLD RECOVERIES

Average Au load on ore

7.692406 gAu/t ore

Mass Au per single ore sample 0.032077 mg/4.17g ore

EXPERIMENT 6.11 - Recycling the ore phase (GTA analysis)

SAMPLE MASSES

Calibration eurve data for samples 1,2,3 and 7,8,9 and 13,14,15 (Experiment 6.11)

Calibration curve data for samples 4,5,6 and 10,11,12 and 16,17,18 (Experiment 6.11)

RESULTS (Experiment 6.11)

Average Au loading on ore

4.21367 g Au/t ore (Average of samples 7-12)

Mass of gold per single ore sample

0.017571 mg Au/ 4.17 g ore

PERCENTAGE GOLD RECOVERIES (Experiment 6.11)

Au recovery % = [Total m_{Au} in coal sample/ m_{Au} in single ore sample]*100

Au recovery % = [Total m_{Au} in coal sample/ m_{Au} in single ore sample]*100

Total Au recovery: 42.18709 %

EXPERIMENT 6.12 - Recycling the ore phase: Mass balance check (GTA analysis)

SAMPLE MASSES

CALIBRATION CURVE DATA

Abs. =Slope*mass Au + Intercept

RESULTS (Experiment 6.12)

Average Au loading on ore

4.824059 g Au/t ore (Average of samples 7-9)

Mass of gold per single ore sample

0.020116 mg Au/ 4.17 g ore

PERCENTAGE **GOLD** RECOVERIES (Experiment 6.12)

Au recovery $\% = [\text{Total } m_{Au} \text{ in coal sample/m}_{Au} \text{ in single ore sample}]$ *100

Au recovery % = [Total m_{Au} in coal sample/ m_{Au} in single ore sample]*100

 $\hat{\mathbf{z}}$

Au recovery % = [Total m_{Au} in remaining ore sample/ m_{Au} in single ore sample]*100

EXPERIMENT 6.13 - Recycling the coal-oil phase: Mass balance check (ICP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.13)

CALCULATIONS (Experiment 6.13)

PERCENTAGE GOLD RECOVERIES (Experiment 6.13)

EXPERIMENT 6.14 - Optimised CGA on industrial ore: cheking the mass balance (lCP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.14 - ICP analysis)

Average Au load on ore Mass Au per single ore sample 5.14811 gAu/t ore

% GOLD RECOVERIES

0.021468 mg Au/4.17g ore **IMass balance check:** 101.6259 %

EXPERIMENT 6.15 - CGA on industrial ore: Recycling the coal-oil phase (lCP analysis)

SAMPLE MASSES

RESULTS (Experiment 6.15)

Average Au loading 5.14811 g/Au ore

Per single ore sample 0.021468 mg/4.17g

CALCULATIONS (Experiment 6.15)

PERCENTAGE GOLD RECOVERIES (Experiment 6.15)

سأعتب والمتعارف والمتعارف

 $\ddot{\cdot}$

Carlo Carlos

Service

677

 $\ddot{}$

EXPERIMENT 6.16 - XRD analysis: Diffractogram for Sample 8 (agglomerates - after contact)

26-1077 C

 \mathbb{R}^*

235

 \mathbb{R}^*

 \mathcal{C}^{\bullet}

EXPERIMENT 6.17 - Mercury amalgamation (lCP analysis)

SAMPLE MASSES

RESULTS (Induction coupled plasma analysis)

RESULTS (continued for Experiment 6.17)

Average gold loading on ore Mass gold per single ore sample 5.061783 g *Ault* ore 0.101236 mg *Aul20* gore

PERCENTAGE GOLD RECOVERIES (Experiment 6.17)

EXPERIMENT 6.18 - Mercury amalgamation (lCP analysis)

SAMPLE MASSES

RESULTS (Induction coupled plasma analysis) 6 (SOLD RECOVERIES 1999) 6 (SOLD RECOVERIES

Average gold loading on ore 5.093144 g Au/t ore Mass gold per single ore sample 0.050931 mg Au/ 10 g ore

APPENDIX B

PUBLISHED PAPER

ENVIRONMENT & INNOVATION IN MINING AND MINERAL TECHNOLOGY (M.A. Sánchez, F. Vergara, and S.H. Castro, Eds.), University of Concepción-Chile, 1998.

THE EFFECT OF OPERATING PARAMETERS ON THE EFFICIENCY OF THE COAL GOLD AGGLOMERATION PROCESS

W. Kotze and F.W. Petersen

Department of Chemical Engineering Cape Technikon, PO Box 652, Cape Town, 8000 SOUTH AFRICA Tel: +27 21460-3159, Fax: +27 21460-3217, E-mail: fwp@norton.ctech.ac.za

ABSTRACT

Cyanidation and amalgamation, which are methods whereby gold is recovered, are very frequently a source of severe pollution due to the large scale use of hazardous chemicals such as cyanide and mercury. Coal gold agglomeration (CGA), a process developed some years ago for gold extraction, has the advantage in that it has little negative impact on the environment. The CGA process based is on the hydrophobic/oleophilic qualities of coal, gold and oil. Gold particles which are substantially free become attached to the coal-oil agglomerates during collision, and eventually penetrate into the agglomerates. The resulting agglomerates are recycled to increase the gold loading, separated from the slurry, burnt, ashed and smelted to produce gold bullion.

Batch tests were performed on a synthetic gold ore containing fine gold powder. The slurry was contacted with a mixture of coal and industrial charcoal, after which both the agglomerates and ore were analysed.

Operating parameters, such as the mode of contact between the coal-oil phase and the gold containing slurry. the contact time of slurry and the coal-oil phase, the type of oil, the means of separating the coal-oil gold agglomerates from the slurry, the coal to ore. coal to oil and water to ore ratios, the effect of collectors on the rate of settling and, hence the effectiveness of separating and the mineralogy of the ore were investigated.

W. Kotze and F.W. Petersen

Preliminary tests have shown that stirring the coal-oil phase and the slurry yielded higher gold loadings than shaking and the traditional rolling bottle technique. By increasing the time of contact between the coal-oil phase and the gold slurry, so the final gold loading in the agglomerates increases, until an equilibrium value is reached. Furthermore, a medium viscous oil has shown to form finer agglomerates, hence improving the floatability and gold extraction. An increase in the amount of coal and oil, together with a decrease in the amount of water used in the slurry, has proven to increase gold recoveries. Furthermore, by varying the concentration and volume of a collector such as potassium amyl xanthate (PAX) enhanced the settling rate and enabled the effectiveness of separation. However, it was necessary to recycle the coal-oil phase to increase the gold loading into the coal-oil agglomerates.

INTRODUCTION

Traditional gold mining, also known as small scale gold mining has a very negative impact on the environment. Up untill now small scale gold mining is mainly done through mercury amalgamation. Therefore, the British oil and minerals company, BP, has developed a novel gold recovery process in the late eighties, called coal gold agglomeration (CGA). Hydrophobic gold particles are selectively recovered from an ore slurry by incorporation into agglomerates of coal and oil. The agglomerates are recycled to increase their gold loading, then separated from the slurry, burnt and treated to recover the gold. Refer to Figure I for a schematic representation of the basic concepts involved in the CGA process.

Laboratory procedures have been devised for rapidly testing the suitability of ore samples for treatment by CGA. Many ores of differing types have been examined and high gold recoveries (>90%) have been achieved, provided that the gold is or can be liberated. Gold metal (gold in the $Au⁰$ form) is hydrophobic and so is oil and coal, and hence these three main components will group together when introduced into an aqueous medium. Gold bearing materials which are suitable for the CGA process include placers, deep leads, tailings and hard r0ck deposits where it is not associated with pyrite, arsenopyrite, or significant quantities of other sulphidic minerals. High gold recoveries are independent of particles size, which makes it easy to recovery gold grains ranging from 1 - 500 um with equal ease. "(House et al., 1988a: House et al., 1988b: Bellamy et aI., 1989; Sampaio et aI., 1997)"

It will be attempted in this study to investigate the effect of operating parameters on the CGA process with a view to optimize the process route for small scale mining.

 $\frac{1}{\epsilon}$

ENVIRONMENT & INNOVATION IN MINING AND MINERAL TECHNOLOGY

 $\ddot{\bullet}$

Figure 1. A Simplified flowdiagram of the Experimental Setup of the Coal Gold Agglomeration (CGA) process.

Figure 2. Stages I and II represent the state of the different components initially (I) and after contacting (II). [C/oil represents the coal-oil agglomerate]

W Kotze and FW Petersen

 \mathbf{r}

THEORY

The CGA process was divided into two theoretical stages, as shown in Figure 2. The first stage (stage I) represents the initial contact of the three main components (gold, water and the coaloil agglomerates whereas the second stage (stage II) is the representation of the coal-oil-gold agglomerate surrounded by a film of water. The system was viewed in terms of the free energy of each stage, where a stable state is found at the lowest free energy. The following equations were used:

$$
F_1 = A_1 \gamma_{13} + A_2 \gamma_{23} \qquad \qquad \dots (1)
$$

$$
F_{II} = A_1 \gamma_{12} + A_3 \gamma_{23} \qquad \qquad \dots (2)
$$

where: F_1 and F_H A_1 , A_2 and A_3 - the surface areas of each given component (m²) *Y*₁₂, *Y*₁₃, *Y*₂₃ - the free energy of each stage (J) - the surface tensions (J/m^2)

The above equations were combined with Young's equation (Adamson WA, Fifth Edition, 1990):

$$
\gamma_{12} - \gamma_{13} = \gamma_{23} \cos \theta \qquad \qquad \dots (3)
$$

which is a relationship between the surface tensions and the contact angle of the coal-oil phase to the gold surface. The resulting equation is as follows:

$$
F'_{II-I} = \gamma_{23} [(A_3 - A_2)/A_1 - \cos\theta]
$$
 ... (4)

where F'_{H-I} - Resulted free energy of the system per unit area (J/m^2)

Furthermore, the surface tension between the coal-oil agglomerates and water $(\gamma_{23})^*$ was chosen as one, so that a simple relationship between the contact angle, surface areas and total free energy (F'_{11-1}) was found. The aim was to find a relationship between the total free energy of the system and the size of the particles involved in the process together with the contact angle between the solid and liquid phases. Such a relationship could then serve as a useful tool to determine the total free energy of a given system by utilising the easier obtainable parameters. such as the radii of the ore, gold and agglomerates and the contact angle of the oil on the solid surface.

***The actual value of the surface tension (y) is measureable.**

The free energy can then be compared to that of another gold processing procedure, such as mercury amalgamation where the better process will be known by a lower free energy.

By utilising the well known equation for the volume of a sphere:

$$
V = \frac{4}{3}\pi r^3 \qquad \qquad \dots (5)
$$

it was possible to convert the areas in equation (4) to redius. A simple relationship between the radius, contact angle and total free energy was found as follows:

$$
F'_{II-I} = (k^3 + 1)^{2/3} - k^2 - cos\theta \qquad \qquad \dots (6)
$$

where k is the ratio of the radius of the coal-oil agglomerate to the radius of the gold particle.

EXPERIMENTAL

Experimental Setup

Industrial charcoal and oil was mixed to produce coal-oil agglomerates. These agglomerates were then contacted with a gold bearing ore, as seen in Figure 1. During this procedure the gold particles were then separated from the slurry into the coal-oil phase. The resulting coal-oil-gold agglomerates were then separated from the resulting, dried, ashed and analysed for gold. As indicated, these agglomerates can then in turn be recycled to increase their gold loading. Furthermore, ore samples were analysed for gold before and after performing the experiment so that the mass balance could be checked.

W. Kotze and F.W. Petersen

Standard Experiment

A synthetic gold ore (7 g/ton) was made up from a silica sample and fine gold powder $(\pm 44 \mu m)$. The sillica sample had a particle size distribution in the range of 25 to 300 μ m of which 40% was in the 106 μ m range. The coal used had a density of 833.85 kg/m³, consisted of 40% ash and was milled down to a particle size of ± 90 µm. This coal was obtained from the Athlone Powerstation in Cape Town, South Africa, and the oil used was ethane oleate.

It was then vital to decide on the amounts of coal, ore, oil and water to be used, therefore a series of trial and error tests were done, whereby the following ratios were determined: coal:oil -100:20 (by volume), coal:ore - 1:1 (by mass) and for every 4 g of ore 130 ml of distilled water was used to make up the slurry. Potassium Amyl Xanthate (PAX), was used as a collector at and was added at a concentration of 1g/100ml to enhance the floatability of the floatable components.

Furthermore, the experiments were performed in 150 ml bottles into which all the abovementioned components were added and shaked for 50 minutes, making use of a laboratory scale mechanical shaker. After contacting the two phases, the floating coal oil agglomerates, containing gold particles, was then separated from the ore slurry by scraping it from the surface of the aqueous medium.

After separation, the coal-oil agglomerates and ore samples were analysed for gold. Analyses were performed on a Graphite Tube Atomiser (GTA) after treating the samples with an acidic solution named *aqua regia* which consists of 82% hydrochloric acid (HC!) and 18% nitric acid $(HNO₃)$ by volume. Gold dissolves into the abovementioned solution, which makes it possible to determine the exact amount of gold present in a specific sample. A set of experiments were conducted under these conditions so that an average gold recovery of 37.6% was obtained.

Sensitivity Analysis

A sensitivity analysis was performed on selected parameters to determine their effects on the CGA process. These parameters were as follows:

The two phases were contacted at different periods of time.

ENVIRONMENT & INNOVATION IN MINING AND MINERAL TECHNOLOGY

- The mode of contacting these phases, i.e shaking, stirring and the rolling bottle technique.
- The means of separation, i.e scraping, sieving.
- The importance of coal, oil and water was investigated by changing their amounts in the following ratios: coal:ore, coal:oil, water:ore.
- The volume and concentration of the collector was changed so that it could be determined of which significance this component was to the process.
- Further experiments were then performed on a different type of oil.

After performing the sensitivity analysis, the stan lard experiment was then repeated on a real industrial ore.

RESULTS AND DISCUSSION

Refer to Table I below for a summary of the results obtained after performing the abovementioned sensitivity analysis. The influence that each separate parameter had on the effectiveness ofthe CGA process is indicated on the right hand side of the table.

Table J. Results obtained after performing a sensitivity analysis on the CGA process.

Preliminary tests have shown that the type of oil used had no relevant influence on the process, whereas a doubled amount of oil produces a percentage gold recovery as low as 8.78%. It was obvious that a larger amount of oil caused larger agglomerates to form, which in turn provided the gold particles with a much smaller contact area to attach themselves to. However, as

٠

W. Kotze and F.W. Petersen

seen with the next result, to much coal in turn had a negative effect on the process. After applying the rolling bottle technique of contact, it was obvious that the rolling action favoured the formation of larger agglomerates, which explains the lower gold recoveries. The addition of a collector enhances the floatability of the coal-oil agglomerates which enables the effectiveness of separation and hence the percentage gold recoveries are increased to 40.09% and 4 I.67% as seen in Table I. Furthermore, it was found that a stirring action kept the size of the agglomerates to a minimum and hence, the contact area for gold particles to attach themselves at a maximum, therefore an increase in the percentage gold recovery to 45%.

Finally, a halved amount of water enhanced the recovery of gold, which makes sense if kept in mind that the CGA process is based on tne hydrophobicity of gold, coal and oil. Furthermore, water serves as the medium in which gold particles travel to reach their final destination, which then explains the lower recoveries.

According to Figure. 3 gold recoveries are at a maximum when contacted for longer than 50 minutes. The last set of tests were conducted on a real industrial ore, which was a tailings sample obtained from the Western Area Gold mine in South Africa. Percentage gold recoveries dropped down to an average of 22.4%. The only explanation that can be offered is that the mineralogy of the ore, which introduces additional components into the system influences the recovery negatively.

Figure 3. Percentage gold recovery vs. Contact time (minutes)

ENVIRONMENT & INNOVATION IN MINING AND MINERAL TECHNOLOGY

CONCLUSIONS

It can be concluded that the CGA process yielded promising results and was positively affected by the following factors: contact times above 50 minutes, contacting the two phases by stirring, the addition of a collector and a decreased amount of water. However, the opposite effect was found after increasing the amounts of oil and coal used, and if the two phases were contacted by the traditional rolling bottle technique. Furthermore, preliminary results on a real ore sample indicated that the mineralogy of the ore does play a role in the effectiveness of the CGA process.

ACKNOWLEDGEMENTS

We would like to thank MINTEK for their financial contribution to this project.

REFERENCES

Adamson, W Arthur, The Physical Chemistry of Surfaces, Fifth Edition, 1990

Bellamy, SR; House, Cl; Veal, Cl, Recovery of fine gold by coal gold agglomeration, Gold Forum on Technology and Practices - "World Gold '89", 5-8 November 1988a.

House, CI; Townsend, IG; Veal, CJ, The coal gold agglomeration (CGA) process. *International Mining* (l988b).

House, CI; Townsend, IG; Veal, CJ, Coal Gold Agglomeration - Pilot scale retreatment of tailings, *Perth International Gold conference,* Perth, Western Australia, 28 October - 1November 1988.

Sampaio, CH; Klein, SL, Study of selective gold recovery from refractory slime flotation tailings by charcoal-oil agglomerates, *Complex Ores* '97, Bulawayo, Zimbabwe, 19-21 March 1997.

 $\ddot{}$

SUBMITTED PAPER

FREE GOLD RECOVERY BY COAL-OIL AGGLOMERAnON

W KOTZE and FW PETERSEN

Department a/Chemical Engineering, Cape Technikon, PO Box 652, *Cape Town, SOUTHAFRICA, 8000 Tel: +2721460-3159, Fax: +2721460-3217, E-mail: fwpiaJmpi.ctechac.za*

ABSTRACT

The gold mining industry has mainly relied upon the use of highly polluting chemicals, such as mercury and cyanide to recover gold from its ores. The Coal Gold Agglomeration (CGA) process was developed some years ago and has the advantage in that gold is recovered by a procedure which has little or no negative impact on the environment. A gold ore containing liberated gold particles is contacted with coal-oil agglorrerates, whereby the gold is recovered into the coal/oil phase.

Laboratory scale batch tests were performed on an artificial mixture gold slurry and gold recoveries of up to 85% were found under optimised conditions. By recycling the coal/oil phase, it was found that the gold loading onto the agglomerates was increased. Tests performed on an industrial ore vielded slightly lower gold recoveries, and X-ray Diffraction (XRD) analysis on the coal/oil phase showed that minerals other than gold was recovered into this phase.

A comparative study was conducted whereby the CGA process was compared to mercury amalgamation. Gold recoveries obtained through amalgamation were 15% lower than by the agglomeration process, which indicates that this process can be considered favourably as an alternative to amalgamation.

INTRODUCTION

Environmental protection has become the focus of world-wide research in the gold mining industry and favours the development of environmental sound processes, such as the Coal Gold Agglomeration (CGA) process, which is an alternative to existing gold processing methods, such as cyanidation and mercury amalgamation.

The CGA process was developed and patented by the British Petroleum (BP) research team in the early eighties and is based on the recovery of hydrophobic/oleophilic gold particles from ore slurries into agglomerates formed from coal and oil. The oil acts as the bridging liquid between the coal and gold particles where the coal is the carrier of the mineral and enables effective separation of the oil phase. The coal-oil agglomerates are recycled to increase their gold loading, after which they are separated and further treated to produce gold bullion [1-6]. Figure 1 shows a schematic representation of the basic concepts involved in the CGA process.

Gold bearing ores containing liberated/free gold particles, such as alluvial/free milling ores, gravity concentrates or gravity plant tailings was found to be suitable for the CGA process, as **associations with other minerals/metals, such as pyrites. arsenopyrirc etc. reduce the** oleophilicity of gold, and hence limits gold recoveries [2,3,6]. High gold recoveries, irrespective of the gold grain size (ranging in size from $1-500 \mu m$) were found, therefore providing an alternative to amalgamation or intensive cyanidation [3,4,6].

Although the CGA process is not a novel process, *strictu sensu,* and many further studies besides those done by BP have been conducted [7-11], it was attempted in this study to investigate and optimise the process route of the CGA process so as to provide a valid alternative to small scale **mining operations, such as mercury amalgamation.**

THEORY

A theoretical approach to the CGA process has produced a set of mathematical equations, whereby initial and final conditions were expressed in terms of free energy. Refer to Figure 2, where stage I is a representation of the initial contact of the three main components (gold, water and oil) and stage Π is a simplified illustration of the final condition in which of the components find themselves after the contact period. The following equations were used to express the free energy for each stage [12]:

$$
F_1 = A_1 \gamma_{13} + A_2 \gamma_{23} \qquad \qquad \dots (1)
$$

$$
F_{II} = A_1 \gamma_{12} + A_3 \gamma_{23} \qquad \qquad \dots (2)
$$

The above equations were combined with Young's equation [13]:

$$
\gamma_{12} - \gamma_{13} = \gamma_{23} \cos \theta \qquad \qquad \dots (3)
$$

which is a relationship between the various interfacial tensions and three phase contact angle (θ) measured through the oil phase on the gold surface in the presence of water. The resulting equation was simplified by utilising the equation for the volume of a sphere $[V = \frac{4}{3} \pi r^3]$ to convert the area terms into radii so that the final equation was found to be as follows:

$$
F_{H-1} = \gamma_{23} [(k^3 + 1)^{23} - k^2 - cos\theta] \qquad \qquad \dots (4)
$$

where: $F_{\text{II-1}}$

Y23 k

- total free energy difference for the system per unit area (J/m^2) - interfacial tension between the oil phase and water (J/m^2) *ratio* **of radii** of the agglomerates to gold particles ($r_{\text{agglomerate}}/r_{\text{Au particle}}$)

The total free energy difference of a system (F_{H-I}) is a measure of the stability of the system, whereas a lower value represents the more stable system [14]. Equation (4) serves as an empirical model which is a useful tool to evaluate the CGA process in terms of certain parameters such as radii of agglomerates and gold particles or the three phase contact angle. while F_{II-I} is non-dimensionalised by dividing both sides of the equation by γ_{23} . A sensitivity analysis on the dimensionless value of F_{II} has shown that the CGA process is favoured by a minimum contact angle (θ) , where this angle is a measure of the wettability of the gold by the oil phase and must range between 0 and 90 \degree which is classified as the perfect to non-wetting contact angle range [15]. Furthermore, the relationship between the dimensionless free energy and k was found to be an asymptotic function and has provided information about the size of the gold particles that is recoverable for certain size agglomerates. Furthermore, it was found that the agglomerates had to be larger than the gold particles if these were to be recovered successfully. For the purpose of this paper it was decided not to focus on the model and predictions made from it, but rather to provide the reader a brief background as to where it originated from and to identify possible applications thereof.

The model has a dual purpose in that it serves as a further tool to compare the CGA process to the amalgamation process on theoretical basis, whereby actual values for the free energy were calculated. Preliminary estimations have shown that the value of F_{U-1} is predominantly influenced by the value of *Y23,* which is the interfacial tension between oil and water and/or mercury and water for each process respectively. Actual measurements of θ and γ_{23} were, however done and by calculations, at constant values of k, it was found that the CGA process yielded lower values for the free energy, and hence is the more stable of the two processes. It is therefore seen that the CGA process is a viable alternative to the amalgamation process in terms of thermal stability.

EXPERIMENTAL

An experimental programme was commenced by determining the standard conditions for effective operation, followed by a sensitivity analysis on selected parameters and hence, lead to a data base providing sufficient information about the process to determine the conditions for optimal performance. The optimal conditions was used as the basis for further experimentation, including studies such as recycling the coal-oil and/or the ore phase and performing CGA process on an industrial ore. Finally, a comparative study was done, whereby the CGA process was compared to the mercury amalgamation process.

Experimental materials

A synthetic gold ore of grade 7g/t, was prepared from a silica sample $(d_{30} = 106 \mu m)$, ranging from $25 - 300$ μ m), and a fine gold powder of particle size 44 μ m was used for this purpose. The coal used had a density of 834 kg/m³, consisted of 40% ash and was milled down to a particle size of 90 μ m. The coal-oil agglomerates were prepared in a separate step, whereby the oil, i.e. ethane oleate, was added to the suspension of coal particles and agitated 'or a few seconds. A collector, i.e. potassium amyl xanthate (PAX) was added in a concentration of Ig/100 ml and its purpose was to increase the oleophilicity of the gold particles and/or enhance the floatability of the coal-oil agglomerates so as to enable effective separation. The batch experiments were conducted in 150 ml bottles, which kept the samples at manageable sizes and facilitated the analysis thereof.

Standard experimental conditions

The experimental conditions were compiled from the results of a series of trial and er'or tests to ascertain the exact quantities of the raw materials needed to successfully perform the Coal Gold Agglomeration process on laboratory scale. For the purpose of this study, the selected conditions

are referred to as the standard experimental conditions for the CGA process, which was the platform from which the sensitivity analysis was performed and is defined below.

The amounts of coal, oil, ore and water needed were determined and the following ratios were used: coal:oil - 100:20 (by volume), coal:ore - I: I (by mass) and the ore slurry was prepared by utilising 130 ml of distilled water for every 4 g of ore. The coal-oil phase and slurry was contacted for a 50 minute period using a laboratory scale mechanical shaker. The agglomerates were separated from the slurry by means of scraping it from the surface of the aqueous medium. Finally, the ore and agglomerate samples were dried, ashed and dissolved into *aqua regia,* and after further treatment analysed on a Graphite Tube Atomiser (GTA). A set of experiments were conducted under the above-mentioned conditions and a average gold recovery of 38.4% was obtained.

Sensitivity analysis

It was needed to perform a sensitivity analysis on the CGA process in order to determine the conditions needed for optimal performance. The following parameters were selected:

- The coal-oil phase and slurry were contacted for different periods of time,
- the mode of contacting these phases, i.e. shaking, stirring and the rolling bottle technique,
- the means of separating the coal-oil agglomerates from the slurry, i.e. scraping, sieving,
- the amounts of coal-oil and water were altered within the ratios: coal:ore, coal:oil and **water:ore,**
- and volume and concentration of PAX was changed to determine the significance of this additive.

The conditions for optimal performance were found by lumping all the factors which caused gold recovery percentages to increase. All further tests were done under the optimised conditions, which will be discussed under the next section heading.

Recycling

The optimised CGA process was conducted as normal, after which two consecutive recycling studies were done, the first one being the recycling of the ore phase and the second one being the recycling of the coal-oil agglomerates.

CCA on an Industrial ore

The CGA process was performed on a real ore to determine the effect ϵ mineralogy of the ore on the effectiveness of the process. The experimental study started by performing the process on an industrial ore sample, received from the Western Area Goldmine in South Africa, and recycling the coal-oil phase, after which coal-oil and ore samples were analysed for gold. A further analysis was then done whereby both the untreated and pre-treated coal-oil and ore samples were subjected to X-ray Diffraction analysis to determine which minerals were present in each, before and after contacting the agglomerates with the ore slurry.

Comparative study

A comparative study was conducted, whereby the CGA process was compared to the Amalgamation process. Mercury amalgamation is a process utilised predominantly by smallscale miners, whereby mercury is used to amalgamate the free gold particles in the ore. The amalgam is *roasted* on an open fire to get rid of the mercury, which vaporises at these high temperatures and the remaining reddish powder which mainly ccnsists of gold and is then sold.

Laboratory scale batch tests were performed, whereby the amalgamation process was simulated under the same conditions used at the Centre of Mineral Technology (CETEM) in Brazil and these were as follows: 30% solids by weight; mercury/ore ratio: l/20; Sodium hydroxide (NaOH)/ore ratio: 1/1000 and conditioning for two hours in a horizontal cylinder at 20 rpm [9]. The purpose of the sodium hydroxide was to optimise the surface properties of the mercury with respect to the feed charge in the closed vessel [16]. The ore used was a synthetic gold ore $(7g/t)$ and was prepared from a silica sample $(-106 \mu m)$ and gold powder (44 μ m).

After performing the amalgamation tests, the amalgam was separated from the slurry by means of a separation funnel and both ore and amalgam samples were treated with *aqua regia* to dissolve the gold present in each. The resulting solutions were analysed on the Induction Coupled Plasma (ICP), an instrument which is able to analyse for gold 'n the presence of other metals, such as mercury, which is dissolved by *aqua regia* as well.

RESULTS AND DISCUSSION

Sensitivity analysis

Table 1 depicts a summary of the results obtained after performing the sensitivity analysis. Note that they are arranged in order of increased gold recovery.

CONDITIONS	% GOLD RECOVERIES
Standard	38.4
Coal:oil (Oil amount was doubled)	12.07
Rolling bottle technique	37.00
Concentration of PAX (doubled)	41.02
Volume of PAX (doubled)	41.36
Coal:ore (1,5 times the amount of coal)	41.95
Water:ore (water amount was halved)	43.35
Stirring	49.45

Table I. Results obtained after performing a sensitivity analysis on the CGA process.

It can be seen that factors such as an increased amount of oil and COnlact by the rolling bottle technique negatively influenced the CGA process in terms of the percentage gold recoveries. The increased amount of oil gave rise to the formation of unstable agglomerates which could not be separated easily and hence, the recovery process was not brought to its full potential. Furthermore, the rolling bottle and shaking techniques for contacting the coal-oil phase and agglomerates were not as effective as stirring the two phases, where stirring was carried out by a magnetic stirrer. Further factors such as the addition of PAX, an increased amount of agglomerates and a decreased amount of water had caused the percentage gold recoveries to increase. Focussing on the most efficient means of separation, it was found that scraping, as compared to sieving, was the more feasible method for effective separation. Finally, the contacting procedure was repeated for different periods of time, ranging from 10 to 50 minutes

÷

and it was clearly found that the percentage gold recoveries were at a maximum for contact times above 50 minutes.

Optimised CGA

As mentioned earlier, the conditions for most efficient performance of the CGA process was a compilation of the parameters that favoured maximal gold recoveries. A summary of these are as follows: a contact period of at least 50 minutes, contact by stirring, the addition of a collector and a decreased amount of water was used to prepare the ore slurry. The CGA process was repeated under the specified conditions and analysis was done by either the Graphite Tube Atomiser (GTA) or the Induction Coupled Plasma (ICP). These results are tabulated below:

Table 2. The gold recoveries found from the CGA process as performed under optimal conditions

It is seen that overall gold recoveries as analysed by the GTA has increased from 38.4 to 44 % (Refer to Table I). Although an improvement was observed, it was still a very low result and for this reason it was sought to utilise an alternative instrument, i.e the lCP for analysis. All samples were treated in the exact same manner as before i.e by drying, ashing and dissolving them into *aqua regia,* the only difference being the instrumental analysis.

As seen from Table 2, the percentage gold recoveries had increased to a promising 85% and it could be concluded that the low results in previous experiments were rather instrumental based than caused by human error. It is not the aim of this excercise to present an explanation as to why the ICP analysis was the better, which is in fact a study of its own, but rather to utilise the information to our advantage. Needless to say, all further analyses were performed on the ICP. The results of all tests were fairly reproducible with an error margin of less than 5%.

Recycling

Recycling of the ore phase

The coal-oil agglomerates were contacted with an ore sample after which it was separated and the ore contacted with a "fresh" coal-oil sample of the same quantity as before. Both the coal-oil agglomerate samples were analysed for gold.. The first batch of agglomerates recovered an average of 37.9% of the gold from the ore phase and the second batch of agglomerates a further 4.25% of the gold that was originally in the ore sample before any contact. As seen from the low results, the analysis was done by the GTA and although these values are low, it could still be concluded from the results that the coal-oil agglomerates are capable of recovering gold from very low grade ores.

Recycling of the coal-oil phase

The CGA process was performed under optimised conditions and the coal/oil phase was separated as normal, after which these agglomerates were then contacted with a "fresh" ore sample. It was the objective of this study to determine what the possibility was of increasing the gold loading onto the coal-oil phase. The analyses were done on the ICP, whereby all the untreated and pre-treated ore and coal-oil samples were analysed. Table 3, below, stipulates the results:

From the tabulated results it is seen that the agglomerates recovered an average of 83.03% of the gold after it was contacted with the ore the first time and a further average of 75.6% gold was recovered after recycling the same coal-oil phase. Therefore, it is found that the coal-oil agglomerates had the capacity of recovering an average of only 7.44% less gold after it had been recycled once. Furthermore, it was found that the gold loading onto the coal-oil agglomerates was increased from and average of ± 6 to 12 g/ton, which confirms that the recycling procedure is an effective operation and hence, serves as an incentive to promote the CGA process in terms of the economical viability thereof.

CGA on an Industrial ore

As the CGA process was repeated on a real ore sample whereby the coal-oil phase was recycled afterwards, a XRD analysis was done on the untreated and pre-treated coal and ore samples to determine if minerals other than gold was recovered by the coal-oil phase. The results of the XRD analysis are depicted in Table 4.

Table 4. The results on XRD analysis

As seen from Table 4, no gold was detected in any of the samples. The X-ray Diffraction analysis is a surface based analytic method, which does not quantify the minerals on a surface. but rather qualifies those present. Therefore, it is clear that no gold particles were detected on

the surface of any of the samples, which is no proof that they do not exist in the matrix of the sample. However, it is seen that the coal-oil agglomerates *picked up* some other minerals, besides gold and these results merely states that the presence of other minerals could have an effect on the performance of the Coal Gold Agglomeration process. Through ICP analysis it was possible to determine what the effect of the different minerals was on the recoveries by the CGA process. The results are tabulated below.

Refer to Tables 3 and 5 for a comparison of the results. By carefully studying and comparing the results, it is seen that less gold is recovered into the coal-oil phase after performing the CGA process on a real ore sample (a decrease from 83.03 $\sqrt{0.4\%}$). Furthermore, less gold is recovered into the coal-oil phase after recycling with a real ore sample than with the synthetic ore sample (a decrease from 75.6 to 43%). It could then be concluded that the CGA process efficiency is inhibited when performed on a real ore sample, where the gold particles are in competition with minerals such as: quarts, pyrophyllite, pyrite and chlorite (Table 4) to attach themselves to the coal-oil agglomerates.

Comparative study

It is not only the purpose of this study to investigate the CGA process, but also to assess the potential of this new technology as an potential alternative to existing small scale mining operations, such as mercury amalgamation. Environmental legislation has become more stringent and techniques such as amalgamation has been researched in the finest detail so as to come up with ways to improve the environmental safety of this ancient technique. The gold mining operations in the underdeveloped areas, such as the Amazon region and North Africa depend on the amalgamation technique for recovering the gold from alluvial ores, where the miners are not highly skilled or educated which makes such a simple operation a very attractive means of income. Acute mercury poisoning is not only confined to the immediate surroundings of operation, but effects the lives of many people downstream, as fish *eat* the mercury and it is digested to methyl-mercury, which is even more dangerous than the vapours produced by roasting. Although it is not within the scope of this paper to discuss the *i*-etails of the dangers of mercury, it was however necessary to provide the reader some background as to why a valid **technical and economical alternative to mercury amalgamation is researched.**

After performing a batch of simulated mercury amalgamation experiments. the results were found to be very promising towards the CGA process, as seen in T*ab,e 6.*

Table 6. Gold recovery percentages measured after performing Mercury Amalgamation compared to that of Coal Gold Agglomeration

The analysis was performed by the ICP and the results are the average of 3 -5 batch experiments. As seen from the results, the agglomerates recovered an average of 15% more gold than by mercury. Although the CGA process does not outperform the amalgamation process in *strictu sensu,* it definitely is the better alternative based on the environmental friendliness and safety thereof. If referred to the THEORY section of this paper, it is seen that the CGA process is not only the better in terms of recoveries, but a free energy analysis has shown it to be the more stable process in theoretical terms.

CONCLUSIONS

The CGA process yielded promising results, which under optimal conditions was up to 85% gold recoveries. By performing a sensitivity analysis, the optimal conditions were found to be: contact periods above 50 minutes, contact by stirring, the addition of a collector and a decreased amount of water. Furthermore, factors such as an increased amount of oil and contact by the rolling bottle technique caused a decrease in gold recoveries.

A set of recycling experiments was performed, whereby the ore phase was recycled and it was found that the CGA process is suitable for recovering gold from very low grade ores. Further recycling of the coal-oil phase has proved that it is possible to increase the gold loading onto the coal-oil agglomerates, which will be to the advantage of the economical viability of the process.

The CGA process was repeated on a real ore sample, whereby the effect of other minerals was studied. The gold recoveries were less than with the synthetic ore and by performing XRD analysis on the coal-oil phase, it was found that the gold particles were in competition with minerals such as quarts, pyrophylite etc. which attached themselves to the agglomerates, hence the lower results.

Finally, a comparative study was conducted, whereby the CGA process was compared to mercury amalgamation. Laboratory scale amalgamation tests were performed, whereby gold recoveries of $\pm 70\%$ was found, which were less than for the CGA process

A **CKt"OWLEDGBl£:\T**

The authors wish to acknowledge Mintek for their financial support of this work.

REFERENCES

- 1. House, Cl., Townsend, IG and Veal, *CJ,* Separation Process. *Pat. 1nl.* Cl (4) C22B1/16, /OB87/00480, w088/00248 (1988).
- 2. Bonney, CF; Coal Gold Agglomeration A novel Approach to Gold Recovery; *Randol Gold Forum* 88, Scottsdale, Arizona, 23-24 January 1988.
- 3. House, Cl, Townsend, IG and Veal, *CJ;* Coal Gold Agglomeration Pilot Scale Retreatment of Tailings; *Randol International Gold Conference,* Perth, Australia, November 1988.
- 4. House, Cl, Townsend, IG and Veal, *CJ;* Coal Gold Agglomeration, *International Mining,* September 1988.
- 5. Buckley, SA, House, Cl, and Townsend, IG, A Techno-Economic Assessment of the Coal Gold Agglomeration Gold Recovery Process, *The Canadian Mineral Processors Conference,* Ottawa, January 1989.
- 6. Bellamy, SR, House, Cl and Veal, *CJ,* Recovery of fine gold from a placer ore by coal gold agglomerntion, *Gold Forum on Technology and Practices* - .*World Gold* '89 " Reno, Nevada, 5-8 November 1989.
- 7. Hughes-Narborough, C, Wall, NC, Willey, G, Coal Gold Agglomeration: An innovative approach to the recovery of gold in environmentally sensitive areas, *IV International Mineral Processing,* Vo!. 2, 20-22 October 1992.
- 8. Hammelmann, CR, Lins, FF, Middea, A and Monte, MM, Amalgamation in small gold operations alternatives and treatment of Hg - Contaminated soils and effluents, *Extraction and Processing Division Congress 1994* (TMS 123rd Annual Meeting), GW Warren (ed.), TMS, 1994.
- 9. Costa, LSN, Lins, FF and Marciano, A, Utilization of Coal-oil agglomerates to recover gold particles, *Minerals Engineering,* Vo!. 7, No. 11, pp. 1401-1409, 1994.
- 10. Gaidarjiev, St, Kireva, R and Stoev, St, Coal-gold agglomeration of alluvial gold, *Changing Scopes in Mineral Processing,* Kernal, Arsian, Akar & Canpazogiv (eds.), 1996.
- 11. Klein, SL and Sampaio. CH, Study of selective gold recovery from refractory slime flotation tailings by CharcoaVoil agglomerates, *Complex ores* '97. Bulawayo, Zimbabwe, 19-21 March 1997.
- 12. Barrow, GM, *Physical Chemistry*, 4th Edition, McGraw-Hill Book Company, New York
- 13. Adamson, WA, *Physical Chemistry of Surfaces*, 5th Edition, John Wiley & Sons. Inc., New York, 1990.

 $\ddot{\cdot}$

- 14. Henry, ID, Hovarongkura, AD and Malcolm, TJ, Feasibility of Separation Processes in Liquid-Liquid Solid Systems: Free Energy and Stability Analysis, *AIChE Journal,* Vo!. 25, No. I, pp. 160, January 1979.
- 15. Boucher, EA and Murrell, JN, *Properties of liquids and solutions*, John Wiley & Sons
limited, Chichester, 1982. Chichester, 1982.

 $\ddot{}$

 $\overline{}$

16. Priester, M, Hentschel, T, *Small-Scale Gold-Mining,* pp 59,1992.

A Simplified flowdiagram of the Experimental Setup of the Caol Gold Figure 1. Agglomeration (CGA) process.

Figure 2. Stages I and II represent the state of the different components initially (I) and after contacting (II), where the coal-oil agglomerates are represented by oil in the diagram.

 $\overline{}$

Au