# PRODUCTION OF ACTIVATED CARBON FROM PALM KERNEL SHELL FOR GOLD ADSORPTION USING LEACHATES FROM COCOA HUSK ASH (CRUDE POTASH) AS ACTIVATING AGENT

By

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# CERTIFICATION

I hereby declare that this submission is my own work towards the Master of Science in Chemical Engineering and that, to the best of my knowledge, it contains no material which has been accepted for the award of any other degree of the university or any other university, except where due acknowledgement has been made in the text.

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### ABSTRACT

Activated carbon, a porous amorphous industrial adsorbent used extensively in gold mine operations in Ghana was prepared from the agricultural waste, palm kernel shell using crude potash leached from another agricultural waste; cocoa husk ash as the chemical activating agent. The effect of impregnating ratios (I.R.), impregnating temperatures (room temperature and 85°C) and carbonisation/activation temperatures ( $600^{\circ}$ C -  $800^{\circ}$ C) on the quality of the produced granular activated carbon was investigated. The produced activated carbons of particle sizes between 1190µm and 2380µm recorded a mean fresh carbon activity of 91.67% for samples impregnated at 85°C and activated at 800°C at an I.R. of 1.0. An ash content of 2.95 - 6.68% was obtained for the produced GAC impregnated at both room temperature and 85°C. The apparent density of the samples ranged from 0.22 – 0.69g/mL. The quality of the activated carbon was not affected to any significant degree by impregnating temperatures.



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# LIST OF ABBREVIATIONS AND NORMENCLATURE

A-600-0.1	Sample impregnated at 85°C, Activated at 600°C, Impregnation Ratio 0.1
A-600-0.5	Sample impregnated at 85°C, Activated at 600°C, Impregnation Ratio 0.5
A-600-1.0	Sample impregnated at 85°C, Activated at 600°C, Impregnation Ratio 1.0
A-700-0.1	Sample impregnated at 85°C, Activated at 700°C, Impregnation Ratio 0.1
A-700-0.5	Sample impregnated at 85°C, Activated at 700°C, Impregnation Ratio 0.5
A-700-1.0	Sample impregnated at 85°C, Activated at 700°C, Impregnation Ratio 1.0
A-800-0.1	Sample impregnated at 85°C, Activated at 800°C, Impregnation Ratio 0.1
A-800-0.5	Sample impregnated at 85°C, Activated at 800°C, Impregnation Ratio 0.5
A-800-1.0	Sample impregnated at 85°C, Activated at 800°C, Impregnation Ratio 1.0
A-800-1.5	Sample impregnated at 85°C, Activated at 800°C, Impregnation Ratio 1.5
A-800-2.0	Sample impregnated at 85°C, Activated at 800°C, Impregnation Ratio 2.0
AARL	Anglo America Research Laboratory
AAS	Atomic Absorption Spectrophotometer
AC	Activated Carbon
ASTM	American Society for Testing and Materials
B.C.	Before Christ

- B-600-0.1 Sample impregnated at room temperature, Activated at 600°C, Impregnation Ratio 1.0
- B-600-0.5 Sample impregnated at room temperature, Activated at 600°C, Impregnation Ratio 1.0
- B-600-1.0 Sample impregnated at room temperature, Activated at 600°C, Impregnation Ratio 1.0

- B-700-0.1 Sample impregnated at room temperature, Activated at 600°C, Impregnation Ratio 0.1
- B-700-0.5 Sample impregnated at room temperature, Activated at 700°C, Impregnation Ratio 0.5
- B-700-1.0 Sample impregnated at room temperature, Activated at 700°C, Impregnation Ratio 1.0
- B-800-0.1 Sample impregnated at room temperature, Activated at 600°C, Impregnation Ratio 0.1
- B-800-0.5 Sample impregnated at room temperature, Activated at 800°C, Impregnation Ratio 0.5
- B-800-1.0 Sample impregnated at room temperature, Activated at 800°C, Impregnation Ratio 1.0
- B-800-1.5 Sample impregnated at room temperature, Activated at 800°C, Impregnation Ratio 1.5
- B-800-2.0 Sample impregnated at room temperature, Activated at 800°C, Impregnation Ratio 2.0
- BDH British Drug House
- C-600 Control Sample Activation at 600°C with pure Potassium Carbonate
- C-600-0.0 Control Sample, un-impregnated, Activated at 600°C
- C-600-1.0 Control Sample, impregnated at 85°C, Activated at 600°C, Impregnation Ratio of 1.0 with pure potassium carbonate
- C-700 Control Sample Activation at 700°C with pure Potassium Carbonate
- C-700-0.0 Control Sample, un-impregnated, Activated at 700°C

- C-700-1.0 Control Sample, impregnated at 85°C, Activated at 700°C, Impregnation Ratio of 1.0 with pure potassium carbonate
- C-800 Control Sample Activation at 800°C with pure Potassium Carbonate
- CIL Carbon In Leach
- CIP Carbon In Pulp
- CPH Cocoa Pod Husk
- GAC Granular Activated Carbon
- I.R. Impregnation Ratio
- IUPAC International Union of Pure and Applied Chemistry
- ND No Difference
- P-value Probability of obtaining a result beyond the test statistic on an appropriate distribution
- PET Poly Ethylene Terepthalate
- PKS Palm Kernel Shell
- r.t. Room temperature
- SD Significant Difference
- S-GAC Standard Granular Activated Carbon
- TB Thomas Baker

That was actually observed, assuming the null hypothesis is true

- V.M. Volatile Matter
- VOCs Volatile Organic Compounds
- α-value The level of significance and is the probability of rejecting the null hypothesis when the null hypothesis is true.

# **DEDICATION**

To my parents, Miss Mary Doku and Mr. Mathias Meteku and all my siblings.



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#### **CHAPTER I – INTRODUCTION**

#### 1.1 Background

The processing of agro-forest products generates substantial amounts of waste which can be harnessed using appropriate technologies that reduce, reuse and recycle waste to churn out useful products like activated carbon.<sup>[1],[2]</sup>

Activated carbon, a widely used industrial adsorbent, is mainly composed of carbonaceous material with high surface area and porous structure.<sup>[3]</sup> Onus to its relatively high adsorptive capacity and porous structure which allows it to preferentially adsorb organic materials and other non-polar compounds from a gas or liquid stream, it has been found to have significant application in many industrial processes. These include, gold mining operations, carbonated drink manufacturing processes, edible oil treatment operations and waste water treatment plants. These processing industries in Ghana however depend on supply of activated carbon from outside the country and this increases the cost of production. The most commonly used raw materials for the production of activated carbon are coal (anthracite, bituminous and lignite) and wastes of vegetable origin (such as coconut shell, palm kernel shell, corn cobs etc.), resources which are abundant in Ghana. The fixed carbon content of these vegetable waste materials is quite high and hence can be used as feedstock for the production of activated carbon.<sup>[4]</sup>

Activated carbon is produced by either the physical (steam) activation process or the chemical activation process. The physical activation process occurs in two stages: the carbonisation of the precursor in an inert atmosphere and subsequently followed by activation of the crude char in the presence of a reactive gas like carbon dioxide or steam at elevated temperatures of about 900°C. This method accounts for the majority

of activated carbon used in the world and the preferred raw material is coal. Chemical activation on the other hand is a one stage process involving the use of a chemical activating agent (an acid or a base) before carbonising. The chemical agent acts as a dehydrating agent thereby limiting the formation of tar during the carbonising step. <sup>[5]</sup> Chemical activation is also carried out at relatively lower temperatures which enhance the formation of porous structure. For both methods, the carbonising stage enriches the carbon content and creates an initial porosity whiles the activation stage, further develops the porosity in the final product. <sup>[6]</sup>

Although physical activation is mainly employed in the commercial production of activated carbon due to the relative high cost of chemicals (in case of chemical activation), the chemical activation mode has some outstanding advantages. The major advantages include - lower treatment temperatures, shorter treatment times and the exhibition of larger surface area.<sup>[4]</sup>

The choice of raw material for the production of activated carbon depends on the price, purity, potential extent of activation and the availability and stability of the supply. <sup>[7]</sup> Ghana is among the top 20 producers of palm oil, a commodity that generates a lot of waste. For every tonne of palm oil produced, 800kg of palm kernel shell is generated. <sup>[6]</sup> A total of 96,000 tonnes of palm kernel shells were generated from palm oil producing operations in the country in 2010. <sup>[8]</sup> Palm kernel shells are mainly used as fuel or fuel supplement in the small scale traditional oil processing industries. In large scale industries that use a boiler, only 5% of the shells generate heat for the boiler. <sup>[9]</sup> Other uses of palm kernel shell include filling of old wells and marshy lands. Small quantities of the shells are also used as a source of fuel by goldsmiths and blacksmiths. Palm kernel shell is therefore proposed as a starting material for the production of

activated carbon because of the availability of substantial amount of shell all year round, the cheap cost of the raw material, the relatively high fixed carbon content (18% w/w) and low ash content. <sup>[6]</sup>

As a leading producer of cocoa in the world, Ghana produced about 700,000 tonnes of cocoa in the 2011 cocoa season which yielded about 7,000,000 tonnes of wet husk.<sup>[8],[10]</sup>Cocoa pod husk, the major agricultural waste from the cocoa industry presents a number of challenges. It excessively increases soil alkalinity when left to rot on the farm thereby affecting crop yield. There are also environmental concerns in relation to the leachates from disposed husk which may pollute water bodies. <sup>[11]</sup> Potash can be leached from cocoa pod husk ash with water. <sup>[12]</sup> The leachate contains substantial amounts of potassium carbonate and can be used as a chemical activating agent for the production of activated carbon. The availability of raw material and the relatively low cost of technology for the production of potash, makes it (cocoa husk) a good candidate for use as an activating agent.

## **1.2 Problem Statement**

Most research conducted on activated carbon has centred on the physical method of production. Chemical activation method seems to attract little interest owing to the relative high cost of chemicals. The use of crude potash – an impure, less expensive form of Potassium Carbonate obtainable from cocoa husk ash leachates is yet to be explored.

Ghana, a major cocoa producing country has a lot of cocoa husk which hitherto considered a source of environmental pollution can be churned to a useful, less expensive product, a chemical activating agent for the production of activated carbon.

Palm kernel shell, another agro waste product, is under utilised as a resource. The associated disposal problems call for research into its potential use as a viable raw material to help alleviate some of the waste disposal problems facing the country.

## **1.3 Objectives**

The aim of this research is to produce activated carbon for the gold mining industry from agricultural wastes – palm kernel shell, using crude potash ( $K_2CO_3$ ) from cocoa husk ash as the chemical activating agent.

The specific objectives are:

- i. To produce potash alkali from cocoa husk ash.
- ii. To produce and characterise activated carbon from palm kernel shell using the prepared alkali as activating agent at various impregnation ratios and activating temperatures.
- iii. To evaluate the effect of impregnation temperature, impregnation ratio and activating temperature on the yield and quality parameters (activity, hardness, ash, bulk density) of the produced activated carbon.
- iv. Using the t-test to test whether the produced activated carbon meets the quality parameters of activated carbon used in gold mine operations.

# **1.4 Justification**

Gold mining is an indigenous industrial activity in Ghana over centuries. In modern gold mining operations, activated carbon, an important industrial adsorbent is used to recover gold from cyanide (CN) solution using carbon-in- pulp (CIP) or carbon-inleach (CIL) processes. Gold mining firms in Ghana depend on imported activated carbon for their operations. The huge demand for activated carbon following the increase in the number of mining companies makes a targeted production of activated carbon for the mining industry worth pursuing. <sup>[13]</sup> Other users of the commodity include the breweries and the soft drink industry. <sup>[9]</sup> Edible oil processing companies like Unilever Ghana Limited make use of activated carbon in the treatment processes of palm oil. Waste water treatment plants in some food and beverage industries like The Coca Cola Bottling Company of Ghana Limited employ activated carbon for the removal of coloured pigments and odour in waste water. These varied uses make it important to consider the production activated carbon locally to meet the market demand.

Problems associated with the disposal of agricultural waste, especially the volume they occupy (about 96,000 tonnes of palm kernel shell and 7,000,000 tonnes of wet cocoa pod husk in 2011 alone) makes it imperative to find a way of reducing the size (volume) before disposal or finding an alternate use for them. <sup>[2]</sup> Public conflict arising from site selection for landfills and non availability of land makes it important to find alternate ways of re-using waste. <sup>[11]</sup>

Environmental issues arising from leachates from cocoa pod husk left on farm lands or dump sites to rot, makes it vital to find alternate use of cocoa pod husk. The leachates, usually of high pH have a detrimental effect on the soil flora and fauna. The leachates sometimes end up in water bodies causing pollution and its related health problems.

The industrial demand, disposal problems and related environmental issues arising from the disposal of these agricultural wastes makes it imperative to turn such waste products into useful products (resources).

## **1.5 Scope of the Research**

In this work, crude potash, the leachate from cocoa husk ash was used as a chemical activating agent for the production of activated carbon.

The chemical method of producing activated carbon was employed using palm kernel shell as the raw material and crude Potassium Carbonate as the activating agent.

Five different impregnation ratios were employed at room temperature and at 85°C and the samples carbonised at between 600°C and 800°C. The effect of these varying operating parameters on yield and quality of activated carbon were analysed.

A standard test, fresh carbon activity of the produced activated carbon as per gold mine specifications were carried out in specialised mineral laboratory, Intertek Laboratory (formerly Transworld Laboratory), Tarkwa. Other tests such as wet attrition percent, particle size retention percent and apparent density were determined at the Chemical Engineering Laboratory, Kwame Nkrumah University of Science and Technology, Kumasi.

The quality parameters of the characterised activated carbon were compared to quality parameters of activated carbon used in the industry and a statistical analysis tool, the ttest was employed to determine the acceptability or otherwise of the produced activated carbon per gold mine operations.

# 1.6 Limitation of Research

Studies on the surface morphology of the produced activated carbon samples were not carried out because of lack of the requisite analytical tools such as the Scanning Electron Microscope.

## **1.7 Organisation of Chapters**

The thesis is divided into five chapters.

Chapter One: Covers the background information, the problem statement, objectives of the research and the justification.

Chapter Two is the literature review. It brings to the fore previous work in the field and throws more light on the subject matter. The theoretical concepts are well explored here.

Chapter Three describes the experimental work carried out. The various steps involved in the experiment and the standard procedures followed are outlined here.

Chapter four is the results and discussion. The results obtained from the experimental work is analysed and the possible scientific reasons behind the observed results are brought to the fore.

Chapter Five is the conclusion and recommendation: From the results discussed, a conclusion is made and recommendations are made for further studies.



#### **CHAPTER II – LITERATURE REVIEW**

# **2.1 Adsorbents**

Adsorption, the tendency of a molecule from an ambient fluid phase to adhere to the surface of a solid is an important principle employed in many purification and separation processes in the chemical processing industry. Adsorption may either take place physically or chemically. In physisorption (physical adsorption), the forces of attraction are relatively weak involving van der Waals interactions. Most applications of adsorption in separation and purification processes involve physisorption. Chemisorption, however involves the formation of chemical bonds (covalent) which are stronger. It is mainly encountered in catalysis.<sup>[5]</sup>

Adsorbents, solids that have the capacity to adsorb other substances (liquids and gases) are used extensively in adsorption processes. To qualify for usage as a commercial adsorbent, the adsorbent must have high selectivity for specific substance (adsorbate) and high adsorptive capacity. <sup>[14]</sup> The common adsorbents used commercially are outlined in table 2.1 below:

Adsorbent	Nature
Activated alumina	Hydrophilic, amorphous
Silica gel	Hydrophilic/hydrophobic, amorphous
Activated carbon	Hydrophobic, amorphous
Molecular-sieve carbon	Hydrophobic, amorphous
Molecular-sieve zeolites	Polar-hydrophilic, crystalline
Polymeric adsorbents	-

Table 2.1: The Six Major Types of Commercial Adsorbents

Source: Seader and Henley, 2007<sup>[15]</sup>

Activated carbon in particular has a broader spectrum of application and the raw materials for its manufacture are abundant in nature hence the considerable research conducted in recent years on the production and applications of activated carbon.<sup>[3],[6],[7]</sup>

# 2.1.1 Adsorption Isotherms

The process of adsorption is generally described or studied in terms of curves known as adsorption isotherms. <sup>[16]</sup> The Adsorption Isotherms, relate the amount of adsorbate on the solid adsorbent as a function of concentration or pressure (liquid or gas) at constant temperature and pH. <sup>[17]</sup> The adsorption of gold species in solution on granular activated carbon is a typical example of liquid- phase adsorption. The five most common isotherms for adsorption of aqueous solutions (such as Au(CN)<sup>2-</sup> ions in solution) onto activated carbon are illustrated in figures 2.1 to 2.4.

# 2.1.2 Adsorption Models

In liquid phase adsorption processes, the three most common models encountered are the Linear model, Langmuir model and the Freundlich model.

## 2.1.2.1 Linear Model

In the linear model, there is a linear relationship between the equilibrium loading onto the adsorbent and the equilibrium concentration.



Figure 2.1 Isotherm of the Linear Adsorption Model<sup>[18]</sup>

The linear model is mathematically stated as

 $\Gamma = k_1 C - \dots (1)$ 

Where

 $\Gamma$  = equilibrium loading onto adsorbent, mg/g

 $k_l = linear$  isotherm constant, L/mg

C = equilibrium concentration, mg/L

Long linear isotherms are not common in adsorption on carbon surfaces but are found at the initial part of isotherms on homogenous surfaces. The main advantage of the linear model is the fact that it gives accurate description of the adsorption process for very low adsorbate concentrations, a major limitation of the other models.

#### 2.1.2.2 Langmuir Model

The Langmuir model as shown in figure 2.2 is governed by the mathematical expression

Where

 $_{\Gamma}$  = equilibrium loading onto adsorbent, mg/g

 $_{\Gamma o}$  = maximum monolayer coverage capacity, mg/g

 $k_L = langmuir$  isotherm constant, L/mg

C = equilibrium concentration, mg/L

The graphical representation of the model has a plateau-like characteristic, an equilibrium saturation point where once a molecule occupies a site, no further adsorption takes place. At very low adsorbent concentrations however, the Langmuir isotherm is reduced to a linear form.



# 2.1.2.3 Freundlich Model

The Freundlich model of liquid phase adsorption as depicted in figure 2.3 below is expressed mathematically as

 $\Gamma = k_F C^{1/n} - \dots - (3)$ 

Where

 $\Gamma$  = equilibrium loading onto adsorbent, mg/g

 $k_F = linear$  isotherm constant, L/mg

C = equilibrium concentration, mg/L

n= adsorption intensity, a constant.



Figure 2.3 Isotherm of the Freundlich Model <sup>[18]</sup>

The Freundlich isotherm is a typical representation of adsorption on heterogeneous surfaces. It is the most common of all the liquid phase adsorption isotherms. The sole disadvantage of this model is the fact that it does not approach the ideal true linearity at low equilibrium concentrations.

# 2.1.2.4 Other Models

The high affinity and the sigmoid isotherms are however the rare liquid phase adsorption isotherms and therefore are not frequently encountered. Their isotherms are depicted in figure 2.4 below <sup>[15], [16], [18]</sup>



Figure 2.4 The High Affinity and Sigmoid Isotherms of liquid phase adsorption [18]

# 2.2 Activated Carbon

Activated Carbon is a general term used to describe a wide range of amorphous carbon based material prepared to exhibit a high degree of porosity and an extended interparticulate surface area. It is also a common terminology for a group of adsorbing substances of crystalline form, having large internal pore structure. <sup>[17]</sup> Owing to their versatility in usage and properties, various authors define activated carbon differently. In summary, activated carbon can be defined as a porous carbonaceous material with high adsorptive capacity and cannot be characterised by any distinctive formula.<sup>[6],[18],[19],[20]</sup>

#### 2.2.1 Historical Background of Activated Carbon in Metal Production

Activated carbon has long been used in hydrometallurgy. As at 3750 B.C., the Egyptians and Sumerians used wood char for the reduction copper and tin ores in the production of bronze.<sup>[21]</sup>

In 1880, W.N. Davis patented a process for gold recovery from chlorinated solution on wood charcoal. About fourteen years later, W.D. Johnson also patented his procedure for gold recovery from cyanide leach liquors but was not commercialised.

In 1901, R. Von Ostrejko, the father and inventor of modern day activated carbon patented his two revolutionary methods (basis for chemical and physical activation) of producing activated carbon.

About five decades into the twentieth century, Zadra and his team of researchers at the U.S. Bureau mines made a significant breakthrough in precious metal recovery when they developed the Carbon-In –Pulp (CIP) process and illustrated the commercial viability of activated carbon for precious metal (specifically gold and silver) recovery. To date, activated carbon continues to play a crucial role in silver and gold extraction. [21], [22],[23]

# 2.2.2 Types of Activated Carbon

Commercial activated carbon is classified into three main categories depending on the product dimension. These are Powdered Activated Carbon, Granular Activated Carbon and Pelletized Activated Carbon.

Powdered Activated Carbon has particle size less than 1.0mm and an average diameter between 0.15 and 0.25mm. They tend to have the largest surface area due to their small particle size. They are often applied in gas phase adsorption including flue gas treatment. Granular Activated Carbon has relatively larger particle size which ranges from 0.5mm to 4mm. They are mainly employed in liquid phase adsorption processes like water treatment and precious metal recovery. They can also be used in gas phase applications. Pelletized Activated Carbon is obtainable as cylindrical pellets. Their particle sizes are usually in the ranges of 0.8mm to 5mm. They are mostly employed in gas phase adsorption processes due to their low pressure drop and also their high mechanical strength. <sup>[17], [24]</sup>

## 2.2.3 Pore Structure

The pore structure of activated carbon is a parameter, apart from particle size, that is believed to contribute immensely towards the large surface area. Pores, voids, which are connected to the external surface of a solid are created during the thermal decomposition of organic precursors.<sup>[4]</sup>

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Pores are classified using several criteria. These include the origin, the state and the pore size. Based on the origin, there are two types namely the inter-particle and intraparticle pores. The inter-particle pores are based on how the primary particles stick together (aggregation or agglomeration) to form a secondary particle whiles the intraparticle pores are based on the crystal structure of the solid or whether it is intercalated with atoms, ions or molecules. Pores can also be classified by state as either open or closed and it is based on whether the pore will adsorb gas when exposed to an adsorbate gas or not. The dimension of the diameter or radius of the pore is the third mode of classification. Amongst these criteria, the pore size is the most widely and commonly used one. <sup>[21]</sup>

The International Union of Pure and Applied Chemistry (IUPAC) classify pores into three categories based on their pore diameter as shown in table 2.2

Classification	Pore Diameter, nm
Micro-pore	<2
Meso-pore	2-50
Macro-pore	>50

Table 2.2: IUPAC Classification of Pore Sizes in Activated Carbon

Source: Seader and Henley, 2007<sup>[15]</sup>

A pictorial depiction of the arrangement of the three types of pore sizes is shown in

figure 2.5.



Figure 2.5 Pore Size Distributions in Activated Carbon (Source: Strand, 2001)<sup>[25]</sup>

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# 2.3 Modes of Production

Activated carbon can be prepared from a number of raw materials ranging from forest and agricultural by- products to coals.<sup>[26]</sup>

There are two basic methods of preparing activated carbon; the physical activation method which is also termed 'two- step pyrolysis' and the chemical activation method also termed 'single step pyrolysis'. <sup>[17], [27]</sup>

#### 2.3.1 Physical Method

The physical activation process, also referred to as the thermal activation process, generally occurs in two stages; carbonisation (thermal decomposition) of the precursor and the activation (controlled gasification) of the crude char. <sup>[5]</sup> The thermal activation process is also referred to as a two step pyrolysis process because the carbonisation and the activation take place separately during the process.

Carbonisation, the conversion of organic matter to elemental carbon at high temperature in an inert atmosphere is the first stage in the physical activation process. This results in the elimination of elemental hydrogen and oxygen in the precursor to produce a carbon skeleton possessing a latent pore structure. <sup>[5],[17]</sup> Typical temperatures for the carbonisation step ranges from 500°C to 900°C. <sup>[24]</sup> For most lignocellulosic materials like Palm Kernel Shell, carbonisation starts above 170°C and is nearly completed at 600°C. The resulting adsorptive capacity of the char after this stage of carbonisation is small due to the deposition and blockade of pores by tarry substances. <sup>[4]</sup>

The carbonisation stage is subsequently followed by the activation process; the development of the precursor char to increase the adsorptive capacity. <sup>[28]</sup> The controlled gasification of the carbonised intermediate product is carried out by exposing the char to an oxidising atmosphere that greatly increases the pore volume through the elimination of the volatile pyrolysis products and from carbon burn-off. <sup>[5]</sup> The common activating agents used in the physical method include; steam, oxygen (air) and carbon dioxide (CO<sub>2</sub>). Typical temperature for the activation ranges from 800°C to 1000°C <sup>[24]</sup> The endothermic gasification reaction of carbon with steam and the corresponding heat of reaction ( $\Delta$ H<sub>r</sub>) are shown in the equation below;

$$C_{(S)} + H_2 O_{(g)} + \frac{117 \text{kJ}}{\text{gmol}} \rightarrow CO_{(g)} + H_{2(g)} \dots \dots \dots (4)$$

The associated side reaction of the reaction above is the production of carbon dioxide  $(CO_2)$  and hydrogen gas  $(H_2)$  as a result of the carbon surface catalysing the reaction of carbon monoxide (CO) and steam  $(H_2O)$ .

$$CO_{2(g)} + H_2O_{(g)} \to CO_{2(g)} + H_{2(g)} + \frac{42kJ}{gmol} \dots \dots \dots (5)$$

This side reaction retards the activation process since the hydrogen gas  $(H_2)$  becomes strongly adsorbed at the active sites of the carbon surface.

When carbon dioxide is used for the activation, the equation for the reaction and the resulting heat of reaction  $(\Delta H_r)$  is given as;

$$C_{(S)} + CO_{2(g)} + \frac{159 \text{ kJ}}{\text{gmol}} \rightarrow 2CO_{2(g)} \qquad \dots \dots \dots (6)$$

Although there is an inherent problem of side reaction when steam is used as activation gas, steam is still preferred to carbon dioxide because the steam has smaller van der Waals radius than carbon dioxide which enhance the rate of reaction and increases the diffusion in porous carbon matrix.<sup>[29]</sup>

Since the physical activation method of producing activated carbon is a two stage process, it allows the use of the by-product (char) from other pyrolysis activities aimed at producing oil to be used as a feedstock. A typical example is the use of the by-product (char) from the pyrolysis production of bio-oil from jute as the feedstock for the production of activated carbon. <sup>[30]</sup>

Although the physical activation method generally involves a two stage process, it can however also be carried out directly in a single stage without any prior production of a char before activation. <sup>[18]</sup>

#### **2.3.2 Chemical Method**

The chemical activation method of producing activated carbon is also referred to as the single step pyrolysis method. In this method, a precursor material, usually carbonaceous, is carbonised after the addition of a chemical reagent which restricts the formation of tar thereby resulting in an increased yield of activated carbon. The chemical reagent used is recovered for reuse after washing and also to free the pores after carbonisation. <sup>[31]</sup>

Chemical activation is employed mostly for the production of activated carbon from lignocellulosic materials. <sup>[4]</sup> Chemical activation is mainly directed toward the production of powdered activated carbon. However, to prepare granular activated carbon using the chemical activation method, the impregnation is done carefully to ensure intimate contact between the precursor and the chemical reagent. <sup>[18]</sup>

Typically, in the chemical activation process of the production of activated carbon, the carbonisation stage and the activation stage take place simultaneously. <sup>[6]</sup>

The chemical activation mode of production begins with the impregnation of the raw material with an appropriate chemical reagent. The common chemical reagents used for the impregnation include; phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), zinc chloride (ZnCl<sub>2</sub>), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium sulphide (KS), carbonates of alkali metal and metal chlorides <sup>[29],[32].</sup> These compounds are dehydrating agents and inhibit the formation of tar upon calcination. <sup>[4]</sup> The impregnation involves soaking the raw material in the chemical reagent (usually dissolved in distilled water) at a distinct temperature for a predetermined period without evaporation to facilitate the hydration of the precursor material in case of granular activated carbon. <sup>[18]</sup> The mixture is then dried in an oven to complete the impregnation stage. <sup>[33]</sup>

The impregnation is followed by carbonisation and activation which take place simultaneously at a temperature usually lower than that used in physical activation.<sup>[34]</sup> The carbonisation or activation duration and temperature for any raw material and chemical activating agent is determined experimentally to ascertain the optimum conditions. In the production of activated carbon from lignocellulosic raw materials using a chemical activating agent, the duration of activation is about 2 hours. This ensures a near complete expulsion of volatile content from the precursor. <sup>[35]</sup> Figure 2.6 below shows a representation of the level of pores created after activation.



Figure 2.6: Two-dimensional Representation of Carbon Activation (Source: Lehmann, 1998)<sup>[29]</sup>

After the thermo-chemical treatment, the char is allowed to cool and washed sequentially with hot distilled water until the pH of the washing effluent reasonably approaches 7 (neutral). <sup>[36]</sup> The washing ensures the removal and recovery of chemical reagent used for the impregnation and also makes available large amount of pores for adsorption. The washed product is then dried in an oven. <sup>[37]</sup>

The main advantage of chemical activation is the fact that, it involves the use of lower temperatures for pyrolysis and it can be done in just a single step. <sup>[24]</sup>

The physico-chemical method, a combination of both physical and chemical modes of production is considered by some as the third mode of production. <sup>[17]</sup> This is a special case of the two stage process in which the precursor char from the raw material is activated with a chemical reagent other than carbon dioxide or steam.

#### 2.4 Activated Carbon from Agro-waste

In recent years, a number of agro-based materials, including agro-waste have been identified as potential raw materials for the production of activated carbon. Depending on the targeted industry and the locality, some of the identified raw materials include; sugarcane bagasse (sugar industry), coconut shell (precious metal recovery), empty palm fruit bunches (heavy metal recovery- waste water treatment plants). <sup>[38], [39], [40]</sup> Palm kernel shell has been identified as having the potential for the production of activated carbon for various industries. <sup>[41]</sup>

## 2.4.1 Raw Material (Palm Kernel Shell)

Palm kernel shell, the endocarp that covers the endosperm of the palm fruit is a well known agricultural by-product. Although it has very high calorific value of about 3800Kcal/ kg, it is still under utilised as a source of fuel. <sup>[42]</sup> In Ghana, the major oil palm processing companies produce less than 20% of the country's oil palm and use less than a tenth of the kernel shells they generate as fuel. <sup>[43],[9]</sup> The palm kernel shells are usually discarded as a waste and left to rot on dump sites. Figure 2.7, shows a heap of palm kernel shells left to rot at a dump site near Len Clay Stadium, Obuasi.



Figure 2.7: Palm Kernel Shell Dump Site, Obuasi.

Palm kernel shell, has been identified as having the potential to be converted to activated carbon. A good raw material for activated carbon must be readily available in abundance and inexpensive. It must also have low inorganic content as the presence of inorganics reduces the adsorptive capacity. The inorganic component is usually measured as the ash component. The ease of activation and the possibility of generating activated carbon of high adsorptive capacity and hardness is another criterion used for determining the suitability of a raw material for the production of activated carbon. <sup>[21]</sup>

For lignocellulosic materials such as palm kernel shell, the raw material must also have a relatively high volatile content which is controlled carefully to produce the desired product. The precursor material must also have relatively high fixed carbon content. <sup>[18]</sup> The proximate analysis is usually carried out to determine these parameters – fixed carbon, volatile content, ash and moisture.

Previous research into the production of activated carbon from palm kernel shell depended on the intended application. A summary of some selected works on the production of activated carbon from palm kernel shell is shown in table 2.3.

Table 2.3: Summary of Selected Studies on the Production of Activated Carbon fromPalm Kernel Shell

Researchers	Year	Activation Method	Application
Guo and Luo <sup>[44]</sup>	2000	Physical	SO <sub>2</sub> Removal
Hu <i>et al</i> . <sup>[45]</sup>	2001	Chemical	Dye Adsorption
Guo and Lua <sup>[46]</sup>	2003	Chemical	NH <sub>3</sub> Adsorption
Daud and Ali <sup>[47]</sup>	2004	Physical	N <sub>2</sub> Adsorption
Nik et al. $[48]$	2006	Chemical	Heavy Metal Removal
Adinata <i>et al.</i> <sup>[33]</sup>	2007	Chemical	CO <sub>2</sub> Adsorption
Inyagba and Opete <sup>[49]</sup>	2008	Chemical	Cr and Pb Removal
Sumathi <i>et al</i> . <sup>[50]</sup>	2010	Chemical	SO <sub>2</sub> and NO Removal

# 2.4.2 Activating Agent

Depending on the intended usage of the activated carbon, the environmental issues involved, cost and availability, several chemical reagents have been effectively used as activating agents. Common examples include; phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), zinc chloride (ZnCl<sub>2</sub>), aluminium chloride (AlCl<sub>3</sub>), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium hydroxide (KOH), sodium hydroxide (NaOH) and potassium carbonate (K<sub>2</sub>CO<sub>3</sub>). Among these, the most commonly used, industrially are zinc chloride, phosphoric acid and potassium hydroxide. <sup>[18],[51],[52]</sup>

In recent years however, the use of potassium carbonate as activating agent is receiving considerable attention owing to the fact that it is non deleterious hence can be used to produce activated carbon for use in the food industry coupled with the fact that potassium carbonate can be produced from a number of agro wastes as well. <sup>[33]</sup>

Historically, potash refers to an impure form of potassium carbonate mixed with other potassium salts obtained by leaching wood ash with water. A number of indigenous technologies are available for the production of potash usually for soap production. The extract from most of these traditional technologies may be coloured brown due to the high level of impurities. <sup>[53]</sup>

A simple technology for the production of crude potash (impure potassium carbonate) from dried cocoa husk, involves the combustion of the dried husk in the open. The ash is soaked in distilled water for about 12 hours and leached out. <sup>[54]</sup> The ash sediment acts as a sieve (filter) through which the extract leaks. The extract can be concentrated by evaporation to obtain solid crystals.

The alkali extract from the cocoa husk is mainly carbonates of potassium and sodium. This is because, potassium and sodium, the main metals in significant quantities in plant materials are bound to the organic matrix and hence the excessive release of carbon dioxide in the combustion favours the production carbonates rather than oxides. The hydrolysis of the carbonates generates the hydroxides. Reported Experimental work conducted on cocoa pod husk shows that the yield of sodium carbonate and sodium hydroxide is negligible relative to potassium carbonate and potassium hydroxide. <sup>[53]</sup> The crude potash from cocoa pod husk therefore is made of potassium carbonate and potassium hydroxide.

### **2.5 Applications of Activated Carbon**

Activated carbon is used in a number of processes including gas phase applications, liquid phase applications and in catalytic processes.

Gas phase applications of activated carbon include; gas purification processes in which granular activated carbon (GAC) is used to reduce pollutant gases to very low
concentrations in order to reduce or minimise their harmful effects. Typical examples are in the removal of hydrogen sulphide ( $H_2S$ ) in sour natural gas and the removal of sulphur dioxide ( $SO_2$ ) and nitrous oxides ( $NO_x$ ) from flue gas (effluent gas purification).

Odour mitigation, the removal of foul smelling species like hydrogen sulphide and other organic mercaptans (thiols) fall into gas phase application. AC can also be used to remove volatile organic compounds (VOCs), from exhaust emissions of automotive engines. Another gas phase application of AC is in the purification stages in the production of natural gas from land fill sites and natural gas storage. <sup>[30], [56]</sup> The use of AC in the production of industrial gas mask and other chemical warfare protection apparels is also an important gas phase application. <sup>[18]</sup>

Liquid phase applications of AC are enormous. The major ones include; potable water treatment processes, specifically in the polishing stages to remove trihalomethanes, the product of chemical treatment to disinfect water. GAC may also be applied when there is persistent problem with odour and taste. The food and beverage industries also depend on AC to remove colour pigments and odour from products. Another popular liquid phase application is the use of AC in industrial waste water treatment, either to upgrade the waste water for reuse or to remove toxic contaminants before discharge. <sup>[18],[19]</sup> AC is also used extensively in the adsorption of dyes from waste water effluents in the textile industry.<sup>[56]</sup> In hydrometallurgy, AC is used in the recovery of precious metals like silver and gold from cyanide solutions. <sup>[39]</sup>

In catalytic processes, which accounts for the majority of chemical manufacturing processes in the world, AC is employed in some heterogeneous catalysis either as the main catalyst or as the catalyst support. An example of carbon as the main catalyst is

the production of carbonyl chloride by reacting carbon monoxide and chlorine over carbon catalyst. <sup>[18]</sup>

## 2.6 Activated Carbon in Gold Extraction

Gold adsorption is mostly carried out using granular activated carbon (GAC) because GAC has high selectivity towards gold relative to other base metals and ease of elution due to the large particle size. The ore however goes through preparatory stages before the application of the GAC. The gold bearing ore is finely ground up and mixed with water to form a slurry of about 40% solids. The pH is controlled by adding lime and is followed by dozing the solution with sodium cyanide to form a thick pulp. The inert gold is leached from the ore to form a gold cyanide complex.

The gold cyanide complex formed in the pre-treatment stages is adsorbed onto GAC of a particular size depending on the industry specification.

The next stage is the elution of the adsorbed gold from the GAC using an efficient eluant. The three main methods used industrially in the elution are; the Zadra process, the Anglo America Research Laboratory method (AARL) and the use of organic solvents such as methanol and acetonitrile. Although the Zadra process and AARL are effective in stripping gold from carbon, they consume a lot of energy and have long elution duration.

After the elution, the stripped carbon is made available for reuse by acid washing and thermal regeneration. The gold may be extracted from the concentrated aurocyanide solution by electrolysis.<sup>[57], [58]</sup>

#### 2.6.1. Factors Affecting Gold Cyanide Adsorption

The adsorption of gold cyanide onto carbon is influenced by the gold and carbon concentration, ionic strength, particle size, pH, temperature and pulp density.

Increasing the gold concentration in solution, imparts positively on the adsorption rate due to the increased diffusion gradient between the solution and the carbon surface. The carbon concentration in the adsorption vessel is usually maintained between 15 to 40g/L.<sup>[59]</sup> Also, an increase in the pH negatively affects aurocyanide adsorption as a result of competition for the active sites on the carbon by hydroxides and aurocyanide. Although there is substantial evidence that adsorption increases with decreasing pH, (optimum adsorption occurring at pH of about 5) this is not preferred industrially because low pH ranges favour the release of the toxic carcinogen, hydrogen cyanide gas.<sup>[22], [59]</sup>

Gold adsorption on carbon is also enhanced by the presence of cations such as  $Ca^{2+}$  whiles the presence of anions like  $Cu(CN)_3^-$  negatively affect the gold loading onto the carbon.<sup>[22]</sup>

A decrease in particle size for the same mass of carbon results in an increase in the adsorption rate due to the availability of large surface area. Increasing the pulp density on the other hand results in lower adsorption rate due to increased viscosity which makes mixing difficult and the partial blockage of the carbon pores with ore particles. <sup>[59]</sup>

#### 2.6.2. Gold Adsorption Mechanism

The mechanism of gold adsorption from aqueous solution is yet to be fully understood. Several mechanisms have been proposed in literature but a consensus is yet to be reached on a specific mechanism for gold adsorption. The differences arise mainly due to the fact that AC cannot be directly investigated using physical techniques like infrared spectroscopy and ultraviolet-visible spectroscopy.<sup>[22]</sup>

The rate of gold adsorption onto activated carbon consists of three major mechanisms namely; (i) film diffusion - mass transport of gold cyanide from the bulk solution through a hypothetical hydrodynamic boundary layer (film) around the carbon particle (ii) pore diffusion – mass transport of the dissolved species in the solution filling the pores of the carbon (iii) surface diffusion – the movement of the adsorbed molecules along the internal pore walls of the carbon.<sup>[60]</sup>

#### 2.7 Specifications of Activated Carbon for Gold Recovery

Generally, the specifications of activated carbon depend on the intended application or the end-use. For typical gold mining operations, the main parameters that are usually specified are gold adsorption activity, gold adsorption capacity, hardness (attrition) and particle size. Others like moisture, ash content, fixed carbon, apparent density and pH may also be specified.

#### 2.7.1. Carbon Activity

The adsorption rate of gold cyanide onto activated carbon is considered one of the most important criteria for selecting any GAC for use in CIP circuit in gold mining operations. Carbon activity determines the amount of carbon required and the contact time. With process parameters and carbon particle size held constant, higher rate of adsorption (activity) implies high stage efficiency and reduction in the loss of soluble gold. <sup>[60]</sup> Typical values for GAC used for gold adsorption are above 50% for particle sizes 8×16 and 6×16 on the U.S sieve series. Several methods are employed to determine the carbon activity. However, Carbon tetrachloride number and Iodine number which are traditional activity parameters for AC will not give a true estimate of

GAC used for gold mining. This is because, AC used for gold adsorption combines physical adsorption, surface reactions and solution equilibrium in the net adsorption process and not merely a simple case of vapour phase adsorption.<sup>[39]</sup>

Gold adsorption rate determination (activity) involves measuring the changes in the gold concentration of a standard solution upon the addition of a test carbon. The rate of change in the gold concentration with time expressed as a percentage gives the activity value. <sup>[60]</sup>

# 2.7.2. Carbon Capacity

The gold adsorption capacity of carbon (K value) is the measure of the equilibrium gold capacity. It is determined from an appropriate isotherm for the activated carbon in question. Although it seems to have been shadowed by carbon activity (R value), there is no link between the 'K' and 'R' values as they cannot be used interchangeably. It is considered however as of secondary importance when evaluating carbons for gold recovery because unlike activity, there is no correlation between the carbon capacity and the other necessary parameters like surface area for gold adsorption. <sup>[39]</sup>

#### 2.7.3. Hardness

Hardness, the measure of the wear resistance of carbon or resistance to attrition is one of the most important parameters of gold adsorbing carbon. The turbulence and transport of carbon in pulp with about 40% solids means the GAC is subjected to heavy wear. Not only does the high wear in GAC result in increased consumption of carbon, but also it leads to gold loss as the adsorbed gold onto the carbon fines are extremely difficult to elute. <sup>[57],[39]</sup>

The cause of degradation may be due to impact, crushing or attrition. Of these three, attrition or abrasion is the most common cause of degradation. The commonly used

methods of evaluating the hardness of GAC are the American Society of Testing and Materials (ASTM) Ball Pan Hardness (D 3802) and the Stirring Bar Abrasion Test. In recent years however, wet attrition test methods like the Marshall and Johns (1996) method and the Toles *et al.* (1999) method, are gaining grounds especially in laboratory scale experimental works. <sup>[31],[58,][61],[62]</sup>

#### 2.7.4. Particle Size

The particle size specification of GAC is usually based on the U.S. sieve series and this gives the range of carbon particles. Three main particle size divisions are used in gold mining operations. These are;  $6 \times 12$ ,  $6 \times 16$  and  $8 \times 16$ . A particle size specification of  $8 \times 16$  as used in this study, means the biggest allowable GAC will pass through the U.S. sieve 8 ( 2380 µm) and GAC particles smaller than (or that will pass) through U.S. sieve 16 (1190 µm) will be discarded. Generally, adsorption is higher with smaller particle size as there is a positive correlation between particle size and activity. However too small a particle size also leads to problems in the elution stage and also some dire consequences on pressure drop, which increases with decreasing particle size.<sup>[18][39]</sup> To contain all these successfully, there is always a trade off between particle size and pressure drop.

#### 2.7.5. Moisture Content

The moisture content is the measure of the water content of the GAC. In contrast to gaseous phase applications of AC such as in methane adsorption where higher moisture content of AC adversely affects the adsorption rate, there is no such clear effect of moisture content of GAC on the adsorption performance in gold recovery. It is however considered an important parameter to the consumer because; the cost of GAC is usually given in terms of mass (kg or tonne). GAC with high moisture content implies the consumer paying more for just water. Moisture content values not more than 4% are

considered good for gold adsorption operations <sup>[63]</sup>. The moisture content is usually determined using ASTM D 2867. <sup>[18]</sup>

## 2.7.6. Ash content

Ash content of AC is the residue that remains when the carbonaceous portion is burned off. The ash content increases in direct proportion to the degree of activation. It may be used to ascertain the source of raw material for the production of the AC. For instance the ash content of coconut based AC is 1-3% whiles that of coal based AC is 6-20%. The ash content is also used to assess the adsorptive properties of an AC. A small increase in the ash content decreases the adsorptive property of the AC gravely because ash, mainly inorganic residues are non porous hence their presence reduces adsorption. <sup>[4],[18],[21]</sup> The maximum ash content desired in gold adsorption operations is 4%. <sup>[63]</sup>

# 2.7.7. Apparent Density

The apparent density of AC is the measure of the mass of the material in a given volume (volume of skeletal solid, voids between particles and pores within particles) under specified conditions. <sup>[24][58]</sup> The value of the apparent density, is used in estimating the bed porosity (void volume), an important determinant of the flow characteristics of carbon bed. It is also used to estimate the quantity needed to fill a vessel. <sup>[18], [31]</sup> Apparent density values between 0.46-0.53g/ml are acceptable.<sup>[63]</sup>

#### 2.7.8 Fixed Carbon

This is the solid combustible residue remaining after the removal of moisture, ash and volatile materials from activated carbon. Generally, AC with high fixed carbon has large surface area and hence improved adsorption.<sup>[4]</sup> It is expressed as a percentage and obtained by subtracting the moisture, ash and volatile values from 100.<sup>[28]</sup>

# 2.7.9. pH

The hydrogen ion potential (pH) is another important parameter in gold adsorption processes. The rate of adsorption of gold is known to increase at low pH values(less than 5). This however comes with other health and safety concerns as the poisonous hydrogen cyanide gas (HCN) is released in this pH ranges. Onus to this, CIP plants usually operate in pH ranges above 9. <sup>[22],[59]</sup> Activated carbons commonly used in gold mine operations have pH values usually greater than 9.



# **CHAPTER III – METHODOLOGY**

# 3.1 Materials, Chemicals and Equipment

# 3.1.1. Raw Materials

The raw materials used in this study are shown in table 3.1

Table 3.1 R	aw Materials	and Their	Sources
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Raw Material	Source
Cocoa pod husk (CPH)	Akrokerri in the Adansi North District of Ashanti Region.
Palm Kernel Shell (PKS)	Obuasi in the Obuasi Municipality of Ashanti Region.

# **3.1.2.** Chemicals

The chemicals used for the experiment are presented in table 3.2

Table 3.2 Che	emicals used	d and Their	Sources
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Chemical	Source
Hydrochloric acid (37%)	British Drug House (BDH)
Sodium Acetate (purity 98%)	British Drug House (BDH)
Acetic Acid (99.8%)	British Drug House (BDH)
Potassium Carbonate (purity99.0%)	Thomas Baker (TB)
Sodium Hydroxide pellets	Sigma- Aldrich
Sodium Cyanide	Sigma -Aldrich

# 3.1.3. Equipment Used

The list of equipment used for the experiments are shown in table 3.3

Table 3.3 Apparatus Used and Their Sources

Equipment	Location
Gallenkamp hotbox oven	Chemical Engineering Laboratory
Nabertherm muffle furnace	Civil Engineering Laboratory
U.S. Sieve series	Civil Engineering Laboratory
Stuart hotplate stirrer SB 162-3	Chemical Engineering Laboratory
Burette	Chemical Engineering Laboratory
Pipette	Chemical Engineering Laboratory
Winchester bottle	Intertek Laboratory, Tarkwa
Bottle roller	Intertek Laboratory, Tarkwa
AAS Machine	Intertek Laboratory, Tarkwa
Closed reacting vessel	Ceramics Department
Ceramics Bowl with Lid	Obtained from Central Market, Kumasi

# 3.2. Raw Material Preparation and Characterisation

# 3.2.1. Cocoa Pod Husk (CPH)

The cocoa pod husk (CPH) was first washed in water to remove dirt. It was then sun dried on a clean platform for three weeks.

# 3.2.2. Palm Kernel Shell (PKS)

The obtained PKS was washed several times to remove sand particles and all other foreign materials. The washed PKS was then sun dried for about three weeks.

The dried PKS was crushed in a nut cracker machine to reduce the size. The crushed PKS was sieved to the required particle sizes -  $8 \times 16$ , using the 2380 µm sieve and the 1190 µm sieve.

The suitability of the PKS for the production of AC was ascertained by carrying out a proximate analysis of moisture content, ash content and volatile content on it.

#### 3.2.2.1 Proximate Analysis – Moisture Content

The moisture content of the raw material was determined using ASTM D 3173-03<sup>[64]</sup>. An empty crucible and its cover was heated at 110°C for about 15minutes and cooled in a desiccator. About 1g of pulverised PKS in the crucible was placed in the oven at 110°C for an hour and subsequently cooled in a desiccator. The moisture content was calculated using the relation;

Moisture Content (%) = 
$$[(A-B)/A] \times 100$$

Where

A = Sample used, g

B = Sample after heating, g

# 3.2.2.2 Proximate Analysis – Ash Content

The ash content of the PKS was determined using ASTM D  $3174 - 02^{[64]}$ . An empty crucible and its cover are preheated in a muffle furnace at 700°C for about 30 minutes and cooled in a desiccator. About 1g of dried pulverised PKS in crucible was put in the muffle furnace and heated gradually to a temperature of 500°C within the first hour and increased to about 700°C. It was maintained at 700°C for another 2 hours. It was then cooled in a desiccator and the ash content was evaluated using the relation;

Ash content (%) =  $[(D-E)/C] \times 100$ 

Where

D = weight of crucible, cover and ash residue, g E = weight of crucible and cover, g

C = weight of PKS sample used for analysis, g

#### 3.2.2.3 Proximate Analysis – Volatile Content

The volatile content of PKS was determined using ASTM D  $3175 - 02^{[64]}$ . The empty crucible and its lid was preheated at  $930^{\circ}$ C for about 10 minutes and cooled in a desiccator. 1g PKS is put in the crucible covered and placed in the muffle furnace at  $930^{\circ}$ C for 7 minutes. The crucible and its content are cooled in a desiccator and the volatile component determined using the relation;

Weight loss (%) =  $[(F-G)/F] \times 100$ 

Where

F = weight of sample used, g G = weight of sample after 930°C Volatile matter (%) = H- I

Where

H (%) = weight loss I (%) = moisture

The fixed carbon content of PKS is obtained from the relation;

Fixed carbon (%) = 100 - (moisture + ash + volatile).

#### **3.3 Production of Activated Carbon**

The production of activated carbon (AC) by the chemical activation method involves several stages. It begins with obtaining suitable activating agent, in this case the crude potash, impregnation of the PKS, carbonisation/activation, washing and drying. The flow diagram for the entire process is depicted in Figure 3.1 below:



Figure 3.1: A Block Diagram for the Production of Activated Carbon

#### 3.3.1 Production of Potash Alkali

The well dried cocoa pod husk was combusted to produce ash. Incompletely combusted particles (black particles) were removed from the ash. The procedure of Adewuyi *et al.* (2008) for alkali extraction as reported by Ogundiran *et al.* (2011) was adapted. <sup>[65]</sup> A known mass of the ash was placed in a 1.5L capacity polyethylene terepthalate (PET) bottle and an appropriate volume of distilled water was added. The bottle and its content were shaken vigorously and allowed to settle for about 12 hours, to ensure maximum dissolution of soluble mineral component of the ash in the distilled water. For each batch of ash, three different extraction set ups were made using different ratios of ash to water. Four pin holes were bored at the bottom of the bottle, the screw cap was removed and the extract leaked into the collecting beaker beneath it.

The pH of the clear extract solution was measured, after which double indicator titration method was employed to analyse the clear extract (solution), for the potassium carbonate and potassium hydroxide content.

#### **3.3.2. Impregnation of Precursor**

The Wang *et al.* (2010) method of impregnation of an agro-precursor was adapted in this work.<sup>[36]</sup> The prepared palm kernel shell of known weight was soaked in crude potash solution of known concentration. The impregnation ratios of 0.1, 0.5 and 1.0 were used. In the case of samples activated at 800°C, two additional impregnation ratios, 1.5 and 2.0 were investigated. The impregnation ratio (I.R.) was obtained using the relation;

I.R. = (mass of  $K_2CO_3$  in crude potash solution) / (mass of palm shell)

The impregnation was carried out at room temperature and at a predefined temperature of 85°C for 12 hours in ceramic bowls with well fitted lids to prevent evaporation. The

samples were then dried in the oven at 110°C to complete the impregnation. Samples for which impregnation was carried out at 85°C were labelled A whiles those carried out at room temperature were labelled B.

As a control, pure  $K_2CO_3$  was dissolved in distilled water and the solution was used in impregnating PKS at 85°C at similar conditions. In addition, raw PKS (un-impregnated PKS) was also used as another control.

# **3.3.3.** Carbonisation/ Activation

The carbonisation and activation which occur simultaneously was carried out in a muffle furnace. The procedure used by Adinata *et al.* (2005) for carbonisation/ activation was adapted. <sup>[33]</sup> The samples were placed in a closed clay vessel of dimensions  $26\text{mm} \times 17\text{mm} \times 11\text{mm}$  and put in the Nabertherm muffle furnace (Figure 3.2) for carbonisation. The furnace was heated at a rate of 8 °C/min. until the final temperature was achieved and the sample was maintained at this temperature for a further two hours. The chosen temperatures of carbonisation were varied from 600°C to 800°C. The samples were then allowed to cool in the furnace. The apparent yield (Y°), the yield obtained without washing to remove the residual chemical was then evaluated at this stage using the relation;

$$Y^{o}(\%) = [W_2/W_1] \times 100$$

Where

 $Y^{o}$  = apparent yield, %

 $W_1$  = weight of impregnated PKS before carbonisation, g

 $W_2$  = weight of PKS char, g



# Figure 3.2: Carbonisation / Activation Setup

# 3.3.4. Washing

The produced activated charcoal at this stage was then washed several times with hot distilled water and followed by cold distilled water until the pH was approximately 9. The washing helps to remove the excess chemical after activation.

# **3.3.5.** Drying

The activated carbon preparatory stage was completed by drying the washed samples in the Gallenkamp hotbox oven at  $110^{\circ}$ C for 24 hours (which was enough time for constant weight to be achieved). <sup>[50]</sup> The weight of the dried sample W<sub>3</sub> was noted and the yield (real yield) was evaluated using the relation;

 $Y(\%) = [W_3/W_1] \times 100$ 

Where

$$Y = Yield, (\%)$$

 $W_1$  = weight of impregnated PKS before carbonisation

 $W_3$  = weight of PKS char after washing and drying

# 3.4. Characterisation of AC

The characterisation of AC was limited to the core parameters used in gold mine operations.

#### **3.4.1.** Carbon Activity

The gold adsorption rate (activity) test on fresh carbon and the subsequent carbon activity determination was carried out at the mineral laboratory, Intertek Laboratory, Tarkwa. The prepared GAC samples of the required screened sizes (virgin carbon) were washed and dried at 60°C for about 8hrs. A 0.1% sodium cyanide solution was prepared and about 10ppm of gold standard solution was added to 900mls of the prepared solution and transferred into a Winchester bottle. A known mass of sample GAC (1-9g) was then added to the solution in the Winchester bottle and swirled gently on a mechanical roller for about an hour. The samples were then collected and transferred into test tube for analysis by an Atomic Absorption Spectrophotometer (AAS). The fresh carbon activity is evaluated using the relation

Activity% = [ 
$$(C_s - C_g) / C_s \times 100$$

Where

 $C_s$  = initial gold concentration in solution before bottle rolling, ppm

 $C_g$  = gold concentration left in solution after bottle rolling, ppm

# 3.4.2. Hardness (Attrition)

The Marshall and Johns (1996) method and the Toles *et al.* (2000) method, both wet attrition methods of evaluating hardness were adapted.<sup>[61],[62]</sup> A solution of 0.1M acetate buffer was prepared (0.07M sodium acetate and 0.03M acetic acid, p H 4.7). For each

of the activated carbon samples, 1.0g was placed in 150ml beaker containing 100mls of acetate buffer. The solution was stirred at 500rpm for 2hrs on a Stuart hotplate stirrer SB 162-3 using 1.5 inch stir bar at ambient temperature <sup>[61].</sup> The samples were poured on a 0.30mm screen and washed sequentially with 250mls of distilled water. The retained sample on the screen were transferred onto an aluminium pan and dried in the Gallenkamp hotbox oven at 110°C for 2hrs and cooled in a desiccator. The percent attrition or hardness is calculated using the relation;

Attrition % = 
$$(J-K)/J \times 100$$

Where

- J = Initial mass, g
- K = Final mass, g

The same procedure was used to determine the attrition of a sample granular activated carbon used in commercial gold adsorption process which was coded S-GAC. The attrition value for the S-GAC served as the benchmark with which to compare the attrition values of the produced AC.

## 3.4.3. Particle Size

As per the particle size chosen for the preparation of the AC, U.S. Sieve Series 8 x 16 (sieve opening 2380 $\mu$ m and 1190 $\mu$ m), manual shaking of sieves was used to determine the particle size retention on the sieve. A known mass of AC was placed on the 1190 $\mu$ m sieve and shaken vigorously for 10 minutes to eliminate finer particles (the elution of gold from finer carbons after adsorption is difficult).The retained AC particles on the 1190 $\mu$ m sieve were then expressed as a percentage of the initial quantity.

Particle Size  $\% = (M/L) \times 100$ 

Where

L = initial mass, g

M = final mass after shaking, g

# **3.4.4. Moisture Content**

The moisture content of the prepared activated carbon was determined using ASTM D 2867 – 04. A porcelain crucible and its lid were first dried in an oven and allowed to cool in a desiccator. About 5 g of the prepared AC was put in the crucible and covered with the lid and the mass recorded. The crucible was opened and the crucible and its content together with the lid were placed in the oven for 3 hours at a temperature of 150°C. The crucible was then covered with the lid again and cooled in a desiccator to ambient temperature and the mass of the dried sample was taken again. The moisture content was evaluated using the relation;

Moisture % =  $[(P-Q)/(P-N)] \times 100$ 

Where

N= mass of crucible with lid, g

P= mass of crucible with lid plus original sample, g

Q= mass of crucible with lid plus dried sample, g

## 3.4.5. Ash Content

The ash content of the AC is determined using ASTM D 2866 - 94 (Reapproved in 1999) procedure. A crucible was heated in the muffle furnace at  $630^{\circ}$ C for an hour and subsequently cooled in a desiccator to ambient temperature. A gram of the prepared AC

that has been dried at 150°C for 3hrs was weighed into the crucible. The sample was left in the furnace for 3 hours at 630°C. The crucible and its contents were cooled in a desiccator and weighed again. The ash content of the prepared AC was evaluated using the relation;

$$Ash\% = [(T-R)/(S-R)] \times 100$$

Where

- S = mass of crucible plus original sample, g
- T = mass of crucible plus ashed sample, g

# **3.4.6.** Apparent Density

The apparent density of the prepared AC was determined using Norazatul (2005) method as presented by Alam *et al.* (2008).<sup>[40]</sup> The prepared AC of known mass was wrapped in a plastic bag and dropped gently into 70mls of water in a 100ml capacity measuring cylinder. The difference in the level of water was measured and the density calculated as the mass of the AC divided by the volume of water displaced.

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Apparent Density g/ml = (X - U)/V

Where

U= mass of plastic rubber, g

X= mass of sample plus plastic bag, g

V= volume of water displaced, ml

#### **3.5 Statistical Analysis**

The Independent samples t-test was used to analyse samples prepared in the same manner (subjected to same I.R. and same activation temperature) except for the impregnation temperature. The analysis was carried out at 95% confidence interval and a decision was made as to whether there is significant difference in the quality parameters of samples impregnated at room temperature and those impregnated at  $85^{\circ}$ C. A p-value higher than the  $\alpha$ -value for the two tailed test means no significant difference exists between the samples.

One sample t- test was used to determine whether the quality parameters of the produced AC meets the quality parameters of samples used in the industry. For each parameter, the recorded value for the produced AC was compared to the accepted standard values. A decision was taken for the analysis at 95% confidence level taking cognisance of whether the test is one tailed or two tailed. When the p – value for the analysis is greater than the  $\alpha$  - value, then there is no significant difference between the particular quality parameter of the AC and the test value and vice versa.

A summary of typical quality parameter values of activated carbon used in gold mine operations are shown in table 3.4.

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Parameters	Standard Values	
Activity	>55%	
Attrition	≤7.80% *	
Particle Size Retention (1190µm)	>90%	
Moisture	≤4%	
Ash	≤4%	
Apparent Density	0.49g/ml	

Source: AngloGold Ashanti, 2000 and International Commerce Corporation Carbon, 2010<sup>[66], [63]</sup>

\* = not present in source material (experimentally determined using a wet method)



#### **CHAPTER 1V – RESULTS AND DISCUSSION**

#### 4.1 Proximate Analysis on Palm Kernel Shell

The summary of the results of proximate analysis on the PKS is contained in table 4.1. Details of the results of the individual parameters – moisture content, ash content, volatile matter content and fixed carbon content determination is found in tables A-1, A-2, A-3 and A-4 of Appendix A

PARAMETER	DETERMINATIONS	MEAN VALUE
	(%)	(%)
MOISTURE	7.051	
(ASTM D 3173 – 03)	6.9 <mark>58</mark>	6.994
	6.972	
ASH	1.388	
(ASTM D 3174 – 02)	1.397	1.361
	1.299	
VOLATILE MATTER	71.755	
(ASTM D 3174 – 02)	72.962	72.015
	71.329	
FIXED CARBON	19.806	X S
	18.683	19.630
	20.400	

Table 4.1: Summary of Proximate Analysis on PKS

The proximate analysis shows that palm kernel shell has a very low ash content of about 1.36% which according to Bansal *et al.*, (1988) as reported by Marsh and Rodriguez-Reinoso,(2006) makes it a desirable raw material for activated carbon production because raw materials with low ash content generate AC with low inorganic component which favours adsorption. The relatively high fixed carbon content of about 19.63% also makes palm kernel shell a suitable candidate for AC production.<sup>[18]</sup>

#### 4.2. Cocoa Pod Husk Leachate (Alkali) Analysis

The results of the alkali analysis are presented in Appendix B. The profile of the  $K_2CO_3$  of leachate is shown in figure 4.1 below.



# Figure 4.1: A plot of K<sub>2</sub>CO<sub>3</sub> Vrs Ash Concentration

Figure 4.1 shows that the  $K_2CO_3$  content of the leachate from each ash concentration used for leaching was appreciable and hence the crude  $K_2CO_3$  can be used as activating agent. Such a relatively high  $K_2CO_3$  content of the leachate was also observed by Afrane, (1992) as reported by Babayemi *et al.*, (2011). This may be due to the fact that clean cocoa pod husk was used and the ash was also further screened to remove all unburnt carbon particles before soaking. The effect was evident in the 'clear' colour of the extract solution (leachate) <sup>[12],[53],[54]</sup>

## 4.3 Results of Impregnation

The results of impregnation of the PKS with crude potash to obtain the impregnation ratios 0.1, 0.5, 1.0, 1.5 and 2.0 used in the experiment are in Appendix C. It was physically observed that as the impregnation ratio was increased, the degree to which the samples clung together after impregnation also increased.

#### 4.4 Activated Carbon Yield Analysis

# 4.4.1. Apparent Yield (Y°)

The results of the apparent yield Analysis is found in Appendix D and summarised in figures 4.2 and 4.3 below.



Figure 4.2: A plot of Apparent Yield Vrs I.R. at 85°C Impregnation

From figure 4.2, the apparent yield for the produced AC decreased with increasing activation temperature from 600°C to 800°C. Each carbonisation temperature showed an increase in apparent yield as the impregnation ratios increased from 0.1 to 1.0 and the trend continued when the impregnation ratio was increased to 1.5 and 2.0 for activation at 800°C. The increase in apparent yield as the impregnation ratio increased was probably due to the presence of residual chemicals and also due to the fact that impregnation with a chemical reagent restricts the formation of tar and also inhibits the contraction of particles resulting in relatively higher apparent yield values.<sup>[4],[18]</sup> Similar high apparent yields can be deduced from the works of Yeganeh *et at.*,(2006) and Soleimani and Keghazchi (2008).<sup>[24],[58]</sup>

The apparent yield values for the control samples C-600-1, C-700-1, C-800-1 did not differ significantly from samples A-600-1, A-700-1 and A-800-1 respectively as contained in Appendix D, hence in terms of apparent yield, crude potash was as effective as pure  $K_2CO_3$  in producing activated carbon.



Figure 4.3:A plot of Apparent Yield Vrs I.R. at Room Temperature Impregnation

Also from the graph in figures 4.3, it was evident that for impregnation at room temperature, the apparent yield decreased with increasing activation temperature as in the case of impregnating at 85°C. This may be due to higher reaction rates at higher activation temperatures resulting in an increase in devolatilisation and hence the weight loss.<sup>[18]</sup> In relation to the un-impregnated samples, the impregnated samples have higher apparent yield which implies impregnation with a chemical reagent imparts positively on the apparent yield.

# **4.4.2. Real Yield(Y)**

The results of the real yield analysis (the yield after the activated sample was washed and dried) is presented in Appendix E and shown in Figures 4.4 and 4.5.



Figure 4.4: A plot of Real Yield Vrs I.R. at 85°C Impregnation

The real yield of AC from PKS impregnated at 85°C as shown in figure 4.4 above decreased with increasing activation temperature for each impregnation ratio just like the apparent yield. This may be due to the increase in the release of volatile products as the carbonisation temperature increased. <sup>[33]</sup> In sharp contrast to the apparent yield however, the real yield for each activation temperature decreased with increasing impregnation ratio. This could be due to the removal of residual chemicals on the surface of the unwashed AC particles. The loss of disintegrated particles during the washing could also contribute to the low yield.

The observed real yield for samples impregnated at 85°C with crude potash was similar to the real yield recorded when pure potassium carbonate was used as presented in Appendix E. This demonstrates that crude potash was as good as pure potash in producing activated carbon.





The real yield of the produced granular activated carbon impregnated at room temperature like the samples impregnated at 85°C showed a decrease in yield as the activating temperature increased and the impregnation ratio also increased. A similar trend was also observed by Adinata *et al.*, (2007) <sup>[33]</sup>. The reasons for the observed trend could be due to the removal of residual chemicals and loss of disintegrated particles during the washing process as mentioned for impregnation at 85°C.

# 4.5 Activated Carbon Characterisation Results

# 4.5.1. Carbon Activity

Results of the fresh carbon activity test are presented in the table in Appendix F and shown in figures 4.6 and 4.7.



Figure 4.6: A plot of Fresh Carbon Activity Vrs I.R. at 85°C Impregnation

The fresh carbon activity of the produced activated carbon increased with an increase in activation temperature. The highest activity value was recorded for impregnation at 85°C with a value of 91.67 % for an I.R. of 1.0 and activation temperature of 800°C. The slightly higher activity values recorded for impregnation at 85°C as in figure 4.6 in comparison to impregnation at room temperature as in figure 4.7 may be due to the higher rate of penetration of the raw material by the alkali when impregnated at 85°C and subsequently led to the creation of more pores due to increased devolatilisation.<sup>[18]</sup> For activation at 800°C however, the activity values decreased after the impregnation ratio of 1.0 when extended impregnation ratios were used. This observation may possibly be due to the enlargement of micro-pores to meso-pores (widening of pores and destruction of some walls between the pores) after an I.R. of 1.0 as attested to by Foo and Lee (2010), and Adinata *et al.*(2007)<sup>[51],[33]</sup>

The control samples used (C-600-1.0, C-700-1.0 and C-800-1.0) in relation to the samples impregnated with crude potash as presented in Appendix F show that the crude potash was as effective as pure potassium carbonate.



Figure 4.7: A plot of Activity Vrs I.R. at Room Temperature Impregnation

The fresh carbon activity for samples activated at 600°C was relatively lower than those activated at 700°C and 800°C. This may probably be due to substantial devolatilisation occurring between 700°C and 800°C as a result of high reaction rates occurring at such high temperatures.<sup>[33],[51]</sup> The recorded activity for each impregnation temperature increased with increasing temperature but for activation at 800°C where the activity decreased after an impregnation ratio of 1.0. This observation may however be due to the collapse of micro-pore as the reaction rate increases as a result of the high temperature and high K<sub>2</sub>CO<sub>3</sub> concentrations. The fact that impregnation with a chemical reagent impacts positively on activity is highlighted as samples impregnated at room temperature in all instances had higher activity than the un-impregnated control samples.

#### **4.5.2. Hardness ( Percent Attrition)**

Figures 4.8 and 4.9 shows the results of the hardness test; the details of which are presented in Appendix G.



Figure 4.8: A plot of Percent Attrition Vrs I.R. at 85°C Impregnation

Generally, the percent attrition values recorded for the produced AC increased with increasing activation temperature as demonstrated in figure 4.8. This may be due to the fact that biomass tends to disintegrate when temperature of calcination increases. For each activation temperature, the recorded percent attrition increased as the impregnation ratio increased. Also, the attrition values recorded for samples impregnated at 85°C was slightly higher than those impregnated at room temperature as shown in figure 4.9. This could be that the impregnation at 85°C was much more effective than that at room temperature and hence the granules got weaker when the temperature of carbonisation increased.



Figure 4.9: A plot of Attrition Vrs I.R. at Room Temperature Impregnation

It was also observed that increasing the impregnation ratio resulted in higher attrition values which are detrimental to the use for gold adsorption. With the higher I.R., more alkali is likely to penetrate the precursor and this tends to increase the rate of dehydration and coupled with an increase in temperature, devolatilisation intensifies thereby increasing calcination and the granules get weaker.<sup>[33]</sup>

# 4.5.3. Particle Size Analysis

The experimental results obtained for the particle size analysis for the produced AC samples are presented in Appendix H and figures 4.10 and 4.11.

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Figure 4.10: A plot of Particle Size Retention Vrs I.R. at 85°C Impregnation

From figure 4.10, the particle size retention of the activated carbon reduced gradually when the activation temperature was increased from 600°C to 800°C. This could be due to the disintegration of some granules as the carbonisation temperature increased. Furthermore, the particle size retention values recorded showed an increasing trend with increasing impregnation ratio. The occupation of the interior of the PKS with K<sub>2</sub>CO<sub>3</sub> restricts the formation tar and also inhibits the shrinkage of the precursor. This effect is more pronounced at higher I.R. values<sup>[46]</sup>

The crude potash used as the activating agent was as effective as pure potash used in impregnation (Appendix H). For an impregnating ratio of 1.0, the particle size retention values recorded for crude potash used were slightly higher than the pure potassium carbonate used.



# **Figure 4.11:** Profile of Particle Size Retention Vrs I.R.at Room Temperature Impregnation

From figure 4.11, the trend for impregnation at room temperature was very similar to the impregnation at 85°C. In relation to the un-impregnated samples, it is evident that impregnation imparts positively the particle size retention. The least value of about 55% for the un-impregnated samples deviates sharply from the almost 80% retention values for impregnated samples.

# 4.5.4. Moisture Content Analysis

The results of the moisture analysis are displayed in Appendix I and also shown in figures 4.12 and 4.13



Figure 4.12: A profile of Moisture Content Vrs I.R. at 85°C Impregnation

The graph in figure 4.12 shows a somewhat constant moisture content analysis. This observation of constant moisture content for samples irrespective of the activation temperature and I.R. may probably be due to the fact that all the samples were subjected to the same drying procedure after washing.



**Figure 4.13: A Profile of Moisture Content Vrs I.R. at Room Temperature Impregnation** 

The samples impregnated at room temperature also exhibited similar trend in the moisture content as those impregnated at 85°C. The almost constant moisture content observed irrespective of the impregnation ratio was due to fact that the all the prepared samples were subjected to the same drying procedure.

#### 4.5.5. Ash Analysis

The results of the ash analysis on the produced AC are displayed in Appendix J. Figures 4.14 and 4.15 show the profiles of the ash content analysis for samples impregnated at 85°Cand room temperature respectively.



Figure 4.14: A Plot of Ash Content Vrs I.R. at 85°C Impregnation

The graph in figure 4.14 shows that, the ash content of the produced AC increases steadily when the activation temperature was increased. As the activation temperature increased, more volatile components were driven off and the inorganic constituents present with the carbon such as  $SiO_2$  residues are left over as ash.<sup>[67]</sup> Similar trend was observed by Yagsi (2004) in his chemical activation of apricot stones.<sup>[4]</sup>
Increasing the impregnation ratio for each carbonisation/ activation temperature also resulted in higher ash content values. This could be attributed to alkali residuals which may remain with the carbon as part of the ash as asserted by Yusufu *et al.*(2012). <sup>[52]</sup> The ash content of samples impregnated with pure potassium carbonate was similar to that of samples impregnated with crude potash.



**Figure 4.15:** A Plot of Ash Content Vrs I.R. at Room Temperature Impregnation Impregnation at room temperature as presented in figure 4.15 follows a similar pattern as impregnation at 85°C. The ash content values increased with activation temperature and impregnation ratio. The possibility that residual chemical remaining with the carbon could contribute to the higher ash content values as impregnation ratio increases was projected more clearly in figure 4.15. The un-impregnated samples for each activation temperature had ash content values of 2.76, 3.24 and 4.09 for 600°C, 700°C and 800°C respectively, values that were lower than recorded ash content values for the impregnated samples at the corresponding activation temperatures.

### 4.5.6. Apparent Density

Appendix K shows the results of apparent density analysis and the associated graphs are displayed in figures 4.16 and 4.17



Figure 4.16: A Plot Apparent Density Vrs I.R. at 85°C Impregnation

The apparent density, the mass per unit volume (including pores and voids between particles) is a parameter used to estimate the surface area and activity values of AC. Smaller apparent density values therefore implies the presence of large number of pores and for that reason higher surface area. The recorded apparent density for impregnation at room temperature as illustrated in figure 4.16 decreased with increasing carbonisation temperature and impregnation ratio. For samples activated at 800°C however, the apparent density decreased beyond an impregnation ratio of 1.0. The probable enlargement of micro-pores to meso-pores for impregnation ratios higher 1.0 may account for this observation.<sup>[33]</sup> These suggest an inverse relation between apparent density and surface area and for that matter fresh carbon activity.<sup>[67]</sup>

The apparent density values for the control (impregnation with pure  $K_2CO_3$ ) did not exhibit any significant deviation from samples impregnated with crude potash.



Figure 4.17: A Plot of Apparent Density Vrs I.R. at Room Temperature Impregnation.

The apparent density values for samples impregnated at room temperature generally showed similar trend as those impregnated at 85°C. An increase in the impregnation ratio results in the production of AC with lower apparent density. When higher I.R.s were used, the produced AC experienced a weight loss which might be due to the high loss of volatile products as the rate of dehydration intensifies.<sup>[66]</sup> This subsequently leads to the lower apparent density. The relatively higher apparent density values for the un-impregnated samples confirm this assertion.

### 4.6 Statistical Analysis of AC Characterisation Results

The results of the statistical analysis of the differences in quality parameters for impregnation at room temperature and  $85^{\circ}$ C and the appraisal of the characterisation results of the produced GAC for gold mine operations are contained in Appendices L and M. The statistical analysis of the results was carried out at 95% confidence interval and an  $\alpha$ -value, the probability of rejecting the null hypothesis when it is true of 5% was used.

Sample I.D.	Fresh Carbon	Percent Attrition	Particle Size	Moisture Content	Ash Content	Apparent Density
	Activity (%)	(%)	Retention (%)	(%)	(%)	(g/mL)
			KNIIS	T		
A-600-0.1	ND	ND	ND	ND	ND	ND
B-600-0.1						
A-600-0.5	ND	ND	ND	ND	ND	SD
B-600-0.5			NUM			
A-600-1.0	ND	ND	ND	ND	ND	ND
B-600-1.0						
A-700-0.1	ND	ND	ND	ND	ND	ND
B-700-0.1				1		
A-700-0.5	ND	ND	ND	ND	ND	ND
B-700-0.5		Te				
A-700-1.0	ND	ND	ND	ND	ND	ND
B-700-1.0			alots			
A-800-0.1	ND	ND	ND	ND	ND	ND
B-800-0.1			22			
A-800-0.5	ND	SD	ND	ND	ND	ND
B-800-0.5		15 AD		SAL SAL		
A-800-1.0	SD	ND	ND	ND	ND	ND
B-800-1.0		<	SANE NO	>		
A-800-1.5	SD	ND	ND	ND	ND	ND
B-800-1.5						
A-800-2.0	SD	ND	ND	ND	SD	ND
B-800-2.0						

Table 4.2: Summary of The Effect of Different Impregnation Temperatures (85°C and r.t.) on Quality Parameters

### 4.6.1 Carbon Activity Analysis

The results of the independent t-test analysis carried out in Appendix L, Table L-1, show that with the exception of samples A-800-1.0, A-800-1.5, A-800-2.0 and B-800-1.0, B-800-1.5, B-800-2.0, there was no significant difference between the carbon activity values obtained for samples impregnated at room temperature and those impregnated at 85°C. The p-values in the Levene's test column (sig.) for all the samples were greater than the  $\alpha$ -value (0.05) hence equal variances were assumed and the corresponding p-values in the main equality test of means column were all greater than the  $\alpha$ -value of 0.05. For the exceptions, samples activated at 800°C and having I.R. of 1.0 and above, there was a significant difference between the activity values for the two different modes of impregnation as the p-values of the main equality test column, 0.009, 0.000 and 0.000 were smaller than the  $\alpha$ -value of 0.05. This means that for high activation temperatures like 800°C and high I.R. of 1.0 and above, the impregnation temperature activity value.

The one sample t-test carried out in Table M-1 of Appendix M on the carbon activity results show that for samples activated at 600°C, there was significant difference between the activity of the prepared AC and the Standard value for AC used in gold adsorption processes because in all cases, the mean difference recorded was negative and the corresponding p-values (sig. 2-tailed value /2) were lower than the  $\alpha$ - value of 0.05. Hence all the samples activated at 600°C were not acceptable for gold adsorption. For samples activated at 700°C and 800°C, all but samples A-700-0.1, B-700-0.1, C-700-0.0 and C-800-0.0, had negative mean difference values. Their corresponding one tailed p-values (sig. 2-tailed value /2) were lower than the  $\alpha$ - value of 0.05, hence there was significant difference between their activity values and those used for gold adsorption. This means for samples activated at 700°C and 800°C, the un-impregnated

samples and samples with I.R. of 0.1 (700°C only) were not acceptable in terms of carbon activity for gold adsorption operations. All the other samples activated at 700°C and 800°C were acceptable for gold adsorption because, their recorded mean differences were positive.

### 4.6.2. Hardness (Attrition) Analysis

The statistical analysis results displayed in Table L-2 of Appendix L show that there was no significant difference between the attrition values of samples impregnated at room temperature and those impregnated at 85°C with the exception of samples A-800-0.5 and B-800-0.5. In almost all the cases, equal variances were assumed from the Levenes test column (p-value in Levenes column was higher than 0.05) and the p-value in the main test table was in almost all cases higher than the  $\alpha$ -value of 0.05. Impregnation temperature therefore has no effect on the percent attrition of produced granular activated carbon.

The attrition analysis for samples activated at 600°C, 700°C and 800°C as shown in Table M-2 of Appendix M reveal that, apart from samples A-600-0.1, B-600-0.1 and C-600-0.0 that had negative mean difference, all the other samples had positive mean difference. The one tailed p-value (sig. 2-tailed value /2) for samples with positive mean difference had p-values lower than the  $\alpha$ -value of 0.05 with the exception of samples B-700-0.1 and C-700-0.0 which recorded p-values of 0.076 and 0.425 respectively. This means that in relation to the attrition percent of the standard sample S-GAC, only the un-impregnated samples and those with I.R. of 0.1 for carbonisation at 600°C and 700°C were satisfactory for gold adsorption operations.

#### **4.6.3.** Particle Size Retention

The independent t-test analysis carried out in Table L-3 of Appendix L show that statistically, there was no significant difference between the particle size retention values recorded for samples impregnated at room temperature and those impregnated at 85°C. Equal variances were assumed for all the sample analysis because the p-value (sig.) in the Levenes column was higher than 0.05 but for sample A-600-1.0 and B-600-1.0. In all cases however, the p-values (sig. 2-tailed) in the main test column were higher than the  $\alpha$ -value and hence the conclusion above.

In terms of particle size retention for use in gold adsorption processes, only samples B-600-1.0 and A-800-2.0 had positive mean differences and therefore acceptable for gold adsorption. For all the samples that had negative mean difference, only samples A-600-1.0, C-600-1.0 and B-800-2.0 were satisfactory as their p-values of 0.197, 0.077 and 0.102 respectively were higher than the  $\alpha$ -value of 0.05. All the other samples failed to meet the standard for gold adsorption operations. The results of the analysis are displayed in Table M-3 of Appendix M.

#### 4.6.4. Moisture Content Analysis

The results of the statistical analysis in Appendix L shows that there is no significant difference in the moisture levels of the prepared AC's impregnated at 85°C and those impregnated at room temperature because in all cases, the p-values were greater than the  $\alpha$ -value of 0.05.

The moisture content of all the prepared samples meets the specification used for gold adsorption. In all cases, the mean difference value was negative and the p-values for the one tailed analysis (sig. 2-tailed/2) were less than the  $\alpha$ -value of 0.05 as illustrated in Table M-4 of Appendix M.

### 4.6.5. Ash Content Analysis

Generally, there was no significant difference between the ash content of samples impregnated at 85°C and those impregnated at room temperature except for samples activated at 800°C with an I.R. of 2.0 as shown in Table L-5 of Appendix L. In all cases, the p-values (sig. 2-tailed) were higher than the  $\alpha$ -value and hence the conclusion

Samples activated at 600°C were satisfactory in terms of ash content for gold adsorption operations. All but two samples had negative mean difference values. For the two exceptions, sample B-600-1.0 was also satisfactory as the p- value of 0.48 was higher than the  $\alpha$ -value of 5%. For samples activated at 700°C, the un-impregnated samples and those with I.R. of 0.1 and sample A-700-0.5 were satisfactory as they had negative mean difference values. For samples activated at 800°C, only sample C-800-0.0 recorded an ash content within the acceptable limits for gold adsorption operations as it had a p-value of 0.247 which was higher than the 0.05.

### 4.6.6. Apparent Density Analysis

Generally, the impregnation temperature did not cause any significant difference in the apparent density of the produced AC. As shown in Table L-6 of Appendix L, for all but samples carbonised at 600°C with I.R. of 0.5 which had the p-value (sig. 2-tailed) lower than 5%, all other samples had p-values that were higher than 5%  $\alpha$ -value for all the activation temperatures.

In terms of apparent density for gold adsorption purposes, samples B-600-0.1, B-600-0.5, C-600-0.0, A-700-0.5, A-700-1.0, B-700-0.5, B-700-1.0 and A-800-0.1 were acceptable as their p-values were greater than the  $\alpha$ -value of 5%.

#### **CHAPTER V – CONCLUSION AND RECOMMENDATION**

#### 5.1. Conclusion

The leachates from cocoa pod husk ash contained high levels of crude potash and was successfully used to impregnate palm kernel shell to produce activated carbon.

The apparent yield increased with increasing impregnation ratio whiles the real yield of the produced granular activated carbon was inversely related to the carbonisation temperature and chemical impregnation ratio.

The chemical activating agent used, crude potash impacted positively on some quality parameters for gold adsorption. The fresh carbon activity, the paramount quality parameter of AC in gold mine operations of samples with impregnation ratio of 0.5 and above activated at 700°C and 800°C were very high with the best activity value of 91.67% recorded for sample A-800-1.0. Impregnation ratios higher than 1.0, however resulted in lower fresh carbon activity values. Increasing impregnation ratio also positively influenced the particle size retention. The ash content and apparent density of samples activated at 600°C and 700°C were generally good in terms of gold adsorption. The moisture content of all the produced AC was as competitive as those for gold adsorption operations. The percent attrition of the produced GAC however does not meet specifications for gold adsorption operations.

The temperature of impregnation also affected the quality parameters. Although samples impregnated at 85°C had higher fresh carbon activity values and particle size retention, they were not statistically significantly different from samples impregnated at room temperature. Hence for the production of activated carbons of particle size 8x16, samples can be impregnated at room temperature.

#### 5.2. Recommendation

To fully comprehend the effect of impregnation temperature on parameters like fresh carbon activity and hardness of GAC prepared from PKS, a similar experiment should be carried out but with the particle size increased using other specifications for gold adsorption operations like '6x12' and '6x16' (3360µm by 1680µm and 3360µm by 1190µm respectively) and the sample size for each experiment should be increased whiles limiting the impregnation ratio to between 1 and 1.5.

The high attrition values recorded coupled with loses during the washing of the produced GAC due to the disintegrated particles makes the recovery of powdered activated carbon ( the disintegrated particles for other applications) alongside the main product GAC for gold adsorption worth pursuing.

A good carbon for gold adsorption must not only have high activity but also must have low attrition value, low ash content and a good apparent density value among others. The final choice may involve a trade off in the properties. It is therefore suggested for further studies to be carried with the intent of optimising the process.



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### APPENDICES

### APPENDIX A: PROXIMATE ANALYSIS ON PKS

Sample	1	2	3
Mass of Crucible + Lid (g)	53.948	35.900	38.155
Mass of Crucible + Lid + Sample Before Heating (g)	54.955	36.906	39.159
Mass of Crucible + lid + Sample After Heating and Cooling (g)	54.884	36.836	39.089
Sample Used, A (g)	1.007	1.006	1.004
Sample After Heating, B (g)	0.936	0.936	0.934
Moisture (%)	7.051	6.958	6.972
Mean Moisture (%)		6.994	

Table A-1: Moisture Content Determination on PKS (ASTM D 3173 – 03)

# Table A-2: Ash Content Determination on PKS (ASTM D 3174 – 02)

Sample	1	2	3
Mass of Crucible + Lid, B (g)	53.949	35.900	38.155
Mass of Crucible + Lid + Sample Before Ashing (g)	54.958	36.902	39.156
Mass of Crucible + lid + Sample After Ashing and Cooling, A (g)	53.963	35.914	38.168
Sample Used, C (g)	1.009	1.002	1.001
Ash (%)	1.388	1.397	1.299
Mean Ash (%)		1.361	

Sample	1	2	3
Mass of Crucible + Lid (g)	53.948	35.900	38.155
Mass of Crucible + Lid + Sample Before Volatilisation (g)	54.953	36.901	39.159
Mass of Crucible + lid + Sample After Volatilisation and Cooling (g)	54.161	36.101	38.373
Sample Used, A (g)	1.005	1.001	1.004
Sample After Volatilisation, B (g)	0.213	0.201	0.218
Weight Loss, C (%)	78.806	79.920	78.287
Moisture, D (%)	7.051	6.958	6.958
Volatile Matter Content (%)	71.755	72.962	71.329
Mean Volatile Matter Content (%)	42	72.015	I

Table A-3: Volatile Matter Determination on PKS (ASTM D 3175 – 02)

Table A-4: Fixed Carbon Determination

		1 1 1	
Sample		2	3
- wintpro	1		C
12000			
Moisture (%)	7.051	6.958	6.972
Ash (%)	1.388	1.397	1.299
Volatile Matter Content (%)	71.755	72.962	71.329
121		15	
	10.001	10.00	
Fixed Carbon (%)	19.806	18.683	20.40
S C	5	32	
Man Eined Carlson Contant (0/)		10 (20	I
Mean Fixed Carbon Content (%)	E NO	19.630	
SPIL			

1

### APPENDIX B: RESULTS OF ALKALI ANALYSIS

# **Procedure for Double Indicator Titration Calculation** Concentration of HCl = 0.6MVolume of Leachate used = 10mlMolar Mass of $K_2CO_3 = 138 \text{gmol}^{-1}$ Molar Mass of KOH = $56 \text{gmol}^{-1}$ Indicators = Phenolphthalein followed by Methyl Orange Mean Phenolphthalein End point value = $V_1$ Mean Methyl Orange End point value $HCl_{(aq)} + KOH_{(aq)} \rightarrow KCl_{(aq)} + H_2O_{(l)}$ .....(i) Volume for neutralizing KOH = $V_{1-}(V_2 - V_1)$ $HCl_{(aq)} + K_2CO_{3(aq)} \rightarrow KCl_{(aq)} + KHCO_{3(aq)} + H_2O_{(1)}$ ...(ii) $HCl_{(aq)} + KHCO_{3(aq)} \rightarrow KCl_{(aq)} + CO_{2(g)} + H_2O_{(1)}$ ....(iii) Volume for neutralizing $KHCO_3 = V_2 - V_1$ Combining equations (ii) and (iii)

 $2\text{HCl}_{(aq)} + \text{K}_2\text{CO}_{3(aq)} \rightarrow 2 \text{ KCl}_{(aq))} + \text{H}_2\text{O}_{(l)} + \text{CO}_{2(g)}$ 

Volume for totally neutralizing  $K_2CO_3 = 2(V_2 - V_1)$ 

### For K<sub>2</sub>CO<sub>3</sub>

Therefore, Molar Concentration of  $K_2CO_3 = [(0.6M \times 2(V_2 - V_1)ml] \div [2 \times 10ml]$ 

 $= 0.06(V_2-V_1) \text{ M or } 0.06(V_2-V_1) \text{ moldm}^{-3}$ 

Concentration of  $K_2CO_3$  in gdm<sup>-3</sup> = 0.06(V<sub>2</sub>-V<sub>1</sub>) moldm<sup>-3</sup> × 138gmol<sup>-1</sup> = 8.28(V<sub>2</sub> - V<sub>1</sub>) gdm<sup>-3</sup>

### For KOH

Molar Concentration of KOH =  $[0.6M \times (2V_1 - V_2) ml] \div [10ml]$ 

 $= 0.06(2V_1 - V_2) M \text{ or } 0.06(2V_1 - V_2) \text{ moldm}^{-3}$ 

Concentration of KOH in  $gdm^{-3} = 0.06(2V_1 - V_2) \text{ moldm}^{-3} \times 56 \text{ gmol}^{-1} = 3.36(2V_1 - V_2) gdm^{-3}$ 

### Summary Results of Alkali Analysis

Table B	3-1: I	Results	for	$1^{st}$	Batch
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Mass of	Vol. of	Mean	Phenol.	Met.Or.	K <sub>2</sub> CO <sub>3</sub>	KOH	K <sub>2</sub> CO <sub>3</sub>	KOH %
ash (g)	H <sub>2</sub> O	pН	End Pt.	End Pt.	in	in	% of	of
	$(dm^3)$		Value	Value	g/dm <sup>3</sup>	g/dm <sup>3</sup>	leachate	leachate
			$(V_1)$ ml	$(V_{2)}$ ml				
60.781	1.0	11.95	6.07	11.07	41.4	3.60	68.11	5.92
200.214	1.0	12.40	17.83	33.83	132.48	6.15	66.20	3.07
500.558	1.0	13.02	40.13	77.60	310.25	8.94	61.98	1.79
550.621	1.0	13.29	43.83	85.00	340.89	8.93	61.91	1.62
630.260	1.0	13.41	45.80	90.33	368.71	4.27	58.50	0.68

Table B-2: Results for 2<sup>nd</sup> Batch

Mass of	Vol. of	Mean	Phenol.	Met.Or.	K <sub>2</sub> CO <sub>3</sub>	KOH	K <sub>2</sub> CO <sub>3</sub>	KOH %
ash (g)	H <sub>2</sub> O	pН	End Pt.	End Pt.	in	in	% of	of
	$(dm^3)$		Value	Value	g/dm <sup>3</sup>	g/dm <sup>3</sup>	leachate	leachate
			$(V_1)$ ml	(V <sub>2)</sub> ml				
50.810	0.8	12.28	6.53	11.80	43.64	4.23	68.70	6.67
170.274	0.8	12.93	19.90	38.03	149.87	6.05	70.41	2.84
402.129	0.8	13.32	43.37	83.90	335.59	9.54	66.76	1.90
552.806	1.0	13. <mark>3</mark> 4	44.27	85.63	343.37	9.41	62.11	1.70
623.100	1.0	13.42	47.60	92.80	374.26	8.06	60.06	1.29

Table B-3: Results for 3<sup>rd</sup> Batch

Mass of	Vol. of	Mean	Phenol.	Met.Or.	K <sub>2</sub> CO <sub>3</sub>	KOH	K <sub>2</sub> CO <sub>3</sub>	KOH %
ash (g)	H <sub>2</sub> O	pН	End Pt.	End Pt.	in	in	% of	of
	$(dm^3)$		Value	Value	g/dm <sup>3</sup>	g/dm <sup>3</sup>	leachate	leachate
3	2		(V <sub>1</sub> )ml	(V <sub>2</sub> )ml	13			
65.367	1.0	12.01	6.70	11.87	42.81	5.14	65.488	7.87
213.825	1.0	12.88	19.10	36.83	147.14	4.47	68.81	2.09
504.567	1.0	13.13	42.27	82.43	332.61	7.01	65.14	1.38
550.642	1.0	13.37	42.20	85.10	338.65	11.09	61.50	2.01
640.081	1.0	13.46	48.37	95.13	387.26	5.38	60.50	0.84

Table B-4: Mean Values for Analysis

Mass of ash (g)	Vol. of $H_2O$ (dm <sup>3</sup> )	Mean pH	K <sub>2</sub> CO <sub>3</sub> in g/dm <sup>3</sup>	KOH in g/dm <sup>3</sup>	K <sub>2</sub> CO <sub>3</sub> % of leachate	KOH % of leachate
58.986	0.93	12.08	42.62	4.32	67.43	6.82
194.771	0.93	12.74	143.16	5.56	68.47	2.67
469.085	0.93	13.16	326.15	8.50	64.63	1.69
551.360	1.0	13.33	340.97	9.81	61.84	1.78
631.147	1.0	13.43	376.74	5.90	59.69	0.94

Parameter	Batch 1	Batch 2	Batch	Mean Value
Mass of Ash	60 781	50.810	65 367	58 986
(g)	200.214	170.274	213.825	194,771
(8)	500 558	402 129	504 567	469.085
	550.621	552.806	550.642	551 360
	630,260	623,100	640.081	631.147
Volume of	1.0	0.8	1.0	0.93
$H_2O$ (dm <sup>3</sup> )	1.0	0.8	1.0	0.93
2 ( )	1.0	0.8	1.0	0.93
	1.0	1.0	1.0	1.0
	1.0	1.0	1.0	1.0
Mean pH	11.95	12.28	12.01	12.08
1	12.40	12.93	12.88	12.74
	13.02	13.32	13.13	13.16
	13.29	13.34	13.37	13.33
	13.41	13.42	13.46	13.43
$K_2CO_3$ in	41.4	43.64	42.81	42.62
g/dm <sup>3</sup>	132.48	149.87	147.14	143.16
C	310.25	335.59	332.61	326.15
	340.89	343.37	338.65	340.97
	368.71	374.26	387.26	376.74
KOH in $g/dm^3$	3.60	4.23	5.14	4.32
	6.15	6.05	4.47	5.56
	8.94	9.54	7.01	8.50
	8.94	9.41	11.09	9.81
	4.27	8.06	5.38	5.90
K <sub>2</sub> CO <sub>3</sub> % of	68.11	68.70	65.49	67.43
leachate	66.20	70.41	68.81	68.47
	61.98	66.76	65.14	64.63
	61.91	62.11	61.50	61.84
T	58.50	60.06	60.50	59.69
KOH % of	5.92	6.67	7.87	6.82
leachate	3.07	2.84	2.09	2.67
	1.79	1.90	1.38	1.69
	1.62	1.70	2.01	1.78
	0.68	1.29	0.84	0.94
Total alkali %	74.03	75.37	73.36	74.25
	69.27	73.25	70.90	71.14
	63.77	68.66	66.52	66.32
	63.53	63.81	63.51	63.62
	59.18	61.35	61.34	60.62

# APPENDIX C: DETERMINATION OF IMPREGNATION RATIO

SAMPLE	Mass Conc.	Volume of	$K_2CO_3/g$	PKS/g	I.R.
I.D.	(g/L)	leachate(mls)			
A <sub>1</sub> -600-0.1	41.400	120	4.968	49.673	0.100
A <sub>1</sub> -600-0.5	132.480	120	15.900	31.692	0.502
A <sub>1</sub> -600-1.0	310.250	120	37.230	37.565	0.991
B <sub>1</sub> -600-0.1	41.400	120	4.968	49.233	0.101
B <sub>1</sub> -600-0.5	132.480	120	15.900	31.892	0.499
B <sub>1</sub> -600-1.0	310.250	120	37.230	37.706	0.987
C <sub>1</sub> -600-0.0	-	-	0.000	30.222	0.000
C <sub>1</sub> -600-1.0	-	120 of H <sub>2</sub> O	40.211	40.332	0.997
A <sub>1</sub> -700-0.1	41.400	120	4.968	49.504	0.100
A <sub>1</sub> -700-0.5	132.480	120	15.900	31.810	0.500
A <sub>1</sub> -700-1.0	310.250	120	37.230	37.217	1.000
B <sub>1</sub> -700-0.1	41.400	120	4.968	49.528	0.100
B <sub>1</sub> -700-0.5	132.480	120	15.900	31.804	0.500
B <sub>1</sub> -700-1.0	310.250	120	37.230	37.253	1.000
C <sub>1</sub> -700-0.0	-		0.000	30.030	0.000
C <sub>1</sub> -700-1.0	-	120 of H <sub>2</sub> O	37.451	37.561	0.997
A <sub>1</sub> -800-0.1	41.400	120	4.968	49.458	0.100
A <sub>1</sub> -800-0.5	132.480	120	15.900	31.781	0.500
A <sub>1</sub> -800-1.0	310.250	120	37.230	37.108	1.003
A <sub>1</sub> -800-1.5	340.888	200	68.176	45.561	1.496
A <sub>1</sub> -800-2.0	368.708	220	81.116	40.825	1.987
B <sub>1</sub> -800-0.1	41.400	120	4.968	49.578	0.100
B <sub>1</sub> -800-0.5	132.480	120	15.900	31.671	0.502
<b>B</b> <sub>1</sub> -800-1.0	310.250	120	37.230	37.119	1.003
B <sub>1</sub> -800-1.5	340.888	200	68.176	45.121	1.511
B <sub>1</sub> -800-2.0	368.708	220	81.116	40.605	1.998
C <sub>1</sub> -800-0.0	-		0.000	30.239	0.000
C <sub>1</sub> -800-1.0	-	120 of $H_2O$	40.409	<b>40</b> .196	1.005
A <sub>2</sub> -600-0.1	43.636	120	5.236	52.441	0.100
A <sub>2</sub> -600-0.5	149.868	120	17.984	35.834	0.502
A <sub>2</sub> -600-1.0	335.588	120	40.271	40.141	1.003
<b>B</b> <sub>2</sub> -600-0.1	43.636	120	5.236	52.462	0.099
B <sub>2</sub> -600-0.5	149.868	120	17.984	35.901	0.501
B <sub>2</sub> -600-1.0	335.588	120	40.271	40.284	1.000
C <sub>2</sub> -600-0.0	-	-	0.000	34.955	0.000
C <sub>2</sub> -600-1.0	-	120 of H <sub>2</sub> O	40.408	40.216	1.005
A <sub>2</sub> -700-0.1	43.636	120	5.236	52.206	0.100
A <sub>2</sub> -700-0.5	149.868	120	17.984	36.057	0.499
A <sub>2</sub> -700-1.0	335.588	120	40.271	40.315	0.999
B <sub>2</sub> -700-0.1	43.636	120	5.236	52.345	0.100
B <sub>2</sub> -700-0.5	149.868	120	17.984	36.056	0.499
B <sub>2</sub> -700-1.0	335.588	120	40.271	40.305	0.999
C <sub>2</sub> -700-0.0	-	-	0.000	33.598	0.000
C <sub>2</sub> -700-1.0	-	120 of H <sub>2</sub> O	40.315	40.105	1.005

A <sub>2</sub> -800-0.1	43.636	120	5.236	52.352	0.100
A <sub>2</sub> -800-0.5	149.868	120	17.984	36.059	0.499
A <sub>2</sub> -800-1.0	335.588	120	40.271	40.389	0.997
A <sub>2</sub> -800-1.5	340.888	200	68.671	45.780	1.500
A <sub>2</sub> -800-2.0	374.256	220	82.336	41.192	1.999
B <sub>2</sub> -800-0.1	43.636	120	5.236	52.242	0.100
B <sub>2</sub> -800-0.5	149.868	120	17.984	36.012	0.499
B <sub>2</sub> -800-1.0	335.588	120	40.271	40.289	1.000
B <sub>2</sub> -800-1.5	340.888	200	68.671	45.450	1.511
B <sub>2</sub> -800-2.0	374.256	220	82.336	41.341	1.992
C <sub>2</sub> -800-0.0	-	-	0	35.202	0.000
C <sub>2</sub> -800-1.0	-	120 of H <sub>2</sub> O	40.206	40.733	0.987
A <sub>3</sub> -600-0.1	42.808	120	5.137	50.259	0.102
A <sub>3</sub> -600-0.5	147.136	120	17.656	35.831	0.493
A <sub>3</sub> -600-1.0	332.608	120	39.913	40.015	0.997
<b>B</b> <sub>3</sub> -600-0.1	42.808	120	5.137	50.891	0.101
B <sub>3</sub> -600-0.5	147.136	120	17.657	35.114	0.503
<b>B</b> <sub>3</sub> -600-1.0	332.608	120	39.913	39.768	1.003
C <sub>3</sub> -600-0.0	-	-	0	35.112	0.00
C <sub>3</sub> -600-1.0	-	120 of H <sub>2</sub> O	40.012	40.227	0.995
A <sub>3</sub> -700-0.1	42.808	120	5.137	50.731	0.101
A <sub>3</sub> -700-0.5	147.136	120	17.657	34.818	0.507
A <sub>3</sub> -700-1.0	332.608	120	39.913	39.825	1.002
B <sub>3</sub> -700-0.1	42.808	120	5.137	50.114	0.103
B <sub>3</sub> -700-0.5	147.136	120	17.657	34.912	0.506
<b>B</b> <sub>3</sub> -700-1.0	332.608	120	39.913	39.59	1.008
C <sub>3</sub> -700-0.0	1-5	Xt I	0	34.892	0.000
C <sub>3</sub> -700-1.0		120 of H <sub>2</sub> O	38.005	38.051	0.999
A <sub>3</sub> -800-0.1	42.808	120	5.137	50.546	0.102
A <sub>3</sub> -800-0.5	147.136	120	17.657	35.045	0.504
A <sub>3</sub> -800-1.0	332.608	120	39.913	40.226	0.992
A <sub>3</sub> -800-1.5	338.652	200	67.730	<mark>44</mark> .829	1.511
A <sub>3</sub> -800-2.0	387.256	220	85.196	42.118	2.023
<b>B</b> <sub>3</sub> -800-0.1	42.808	120	5.137	50.244	0.102
B <sub>3</sub> -800-0.5	147.136	120	17.657	34.789	0.508
<b>B</b> <sub>3</sub> -800-1.0	332.608	120	39.913	40.008	0.998
<b>B</b> <sub>3</sub> -800-1.5	338.652	200	67.730	44.971	1.506
B <sub>3</sub> -800-2.0	387.256	220	85.196	42.487	2.005
C <sub>3</sub> -800-0.0	-	-	0	35.662	0.000
C <sub>3</sub> -800-1.0	-	120 of H <sub>2</sub> O	40.324	40.301	1.001

# APPENDIX D: RESULTS OF APPARENT YIELD ANALYSIS

SAMPLE .ID.		DETERMINATIONS	MEAN VALUE
		(%)	(%)
A <sub>1</sub> -600-0.1		38.78	
A <sub>2</sub> -600-0.1	A-600-0.1	37.18	38.06
A <sub>3</sub> -600-0.1		38.21	
A <sub>1</sub> -600-0.5		49.12	
A <sub>2</sub> -600-0.5	A-600-0.5	49.54	48.99
A <sub>3</sub> -600-0.5		48.31	
A <sub>1</sub> -600-1.0		56.81	
A <sub>2</sub> -600-1.0	A-600-1.0	56.16	56.87
A <sub>3</sub> -600-1.0		57.64	
B <sub>1</sub> -600-0.1		38.28	
B <sub>2</sub> -600-0.1	B-600-0.1	37.66	38.27
B <sub>3</sub> -600-0.1		38.88	
B <sub>1</sub> -600-0.5		49.18	
B <sub>2</sub> -600-0.5	B-600-0.5	49.50	48.73
B <sub>3</sub> -600-0.5		47.52	
B <sub>1</sub> -600-1.0	L N	56.93	
B2-600-1.0	B-600-1.0	56.21	57.04
B <sub>2</sub> -600-1.0	2 000 110	57.98	0,110,1
$C_{1}-600-0.0$		33.98	
$C_{2}-600-0.0$	C-600-0.0	33.85	33.82
$C_2 = 600 - 0.0$		33.62	5
$C_{1}-600-1.0$		57.09	
C <sub>2</sub> -600-1.0	C-600-1.0	56.64	56.70
C <sub>3</sub> -600-1.0	1999	56.36	
A <sub>1</sub> -700-0.1	1 Class	31.98	
A2-700-0.1	A-700-0.1	31.94	31.87
A <sub>3</sub> -700-0.1		31.68	
A <sub>1</sub> -700-0.5		44.34	c /
A2-700-0.5	A-700-0.5	44.17	44.41
A <sub>3</sub> -700-0.5		44.73	
A <sub>1</sub> -700-1.0	C) R	51.96	
A2-700-1.0	A-700-1.0	51.66	51.94
A <sub>3</sub> -700-1.0	31	52.21	
B <sub>1</sub> -700-0.1		31.85	
B <sub>1</sub> 700-0.1	B-700-0.1	31.87	31.75
B <sub>2</sub> -700-0.1		31.52	
B <sub>1</sub> -700-0.5		43.11	
B <sub>1</sub> 700-0.5	B-700-0 5	43.65	43 75
B <sub>2</sub> -700-0.5	2 /00 0.5	44 48	10.75
B <sub>1</sub> -700-1 0		51 69	
B <sub>2</sub> -700-1.0	B-700-1.0	52.63	52.09
B <sub>2</sub> 700-1.0	2,001.0	51.05	52.07
$C_{1-700-0.0}$		25.38	
$C_1 = 700 - 0.0$	C-700-0.0	25.50	25.88
C <sub>2</sub> -700-0.0	C-700-0.0	20.30	23.00

C <sub>3</sub> -700-0.0		25.87	
C <sub>1</sub> -700-1.0		52.49	
C <sub>2</sub> -700-1.0	C-700-1.0	51.85	52.31
C <sub>3</sub> -700-1.0		52.58	
A <sub>1</sub> -800-0.1		27.49	
A <sub>2</sub> -800-0.1	A-800-0.1	27.03	27.55
A <sub>3</sub> -800-0.1		28.13	
A <sub>1</sub> -800-0.5		35.16	
A <sub>2</sub> -800-0.5	A-800-0.5	34.97	35.54
A <sub>3</sub> -800-0.5		36.49	
A <sub>1</sub> -800-1.0		45.72	
A <sub>2</sub> -800-1.0	A-800-1.0	44.24	44.78
A <sub>3</sub> -800-1.0		45.39	
A <sub>1</sub> -800-1.5		46.53	
A <sub>2</sub> -800-1.5	A-800-1.5	45.89	46.03
A <sub>3</sub> -800-1.5		45.67	
A <sub>1</sub> -800-2.0		47.93	
A <sub>2</sub> -800-2.0	A-800-2.0	45.94	47.45
A <sub>3</sub> -800-2.0		48.49	
B <sub>1</sub> -800-0.1		25.99	
B <sub>2</sub> -800-0.1	B-800-0.1	26.16	27.70
B <sub>3</sub> -800-0.1		28.84	
B <sub>1</sub> -800-0.5		36.55	
B <sub>2</sub> -800-0.5	B-800-0.5	37.73	36.74
B <sub>3</sub> -800-0.5	Exer)	35.94	2
B <sub>1</sub> -800-1.0	CAR	44.17	
B <sub>2</sub> -800-1.0	B-800-1.0	42.79	43.91
<b>B</b> <sub>3</sub> -800-1.0	159	44.78	
B <sub>1</sub> -800-1.5	C ( Co	46.33	
B <sub>2</sub> -800-1.5	B-800-1.5	46.68	46.69
B <sub>3</sub> -800-1.5		47.05	
B <sub>1</sub> -800-2.0		47.81	5
B <sub>2</sub> -800-2.0	B-800-2.0	48.19	48.07
B <sub>3</sub> -800-2.0		48.21	
C <sub>1</sub> -800-0.0	SR	20.34	
C <sub>2</sub> -800-0.0	C-800-0.0	20.17	20.13
C <sub>3</sub> -800-0.0	31	19.88	
C <sub>1</sub> -800-1.0		46.11	
C <sub>2</sub> -800-1.0	C-800-1.0	44.97	45.80
C <sub>3</sub> -800-1.0	1	46.31	

# APPENDIX E: RESULTS OF REAL YIELD ANALYSIS

SAM	PLE .ID.	DETERMINATIONS	MEAN VALUE
		(%)	(%)
A <sub>1</sub> -600-0.1		31.73	
A <sub>2</sub> -600-0.1	A-600-0.1	30.30	31.26
A <sub>3</sub> -600-0.1		31.74	
A <sub>1</sub> -600-0.5		28.54	
A <sub>2</sub> -600-0.5	A-600-0.5	27.35	28.32
A <sub>3</sub> -600-0.5		29.06	-
A <sub>1</sub> -600-1.0		26.82	
A <sub>2</sub> -600-1.0	A-600-1.0	24.91	25.70
A <sub>3</sub> -600-1.0		25.37	_
<b>B</b> <sub>1</sub> -600-0.1		32.71	
<b>B</b> <sub>2</sub> -600-0.1	B-600-0.1	28.49	30.44
B <sub>3</sub> -600-0.1		30.13	_
B <sub>1</sub> -600-0.5		28.46	
B <sub>2</sub> -600-0.5	B-600-0.5	25.29	26.98
B <sub>3</sub> -600-0.5		27.20	-
<b>B</b> <sub>1</sub> -600-1.0	- N	27.02	
B <sub>2</sub> -600-1.0	B-600-1.0	26.13	26.06
<b>B</b> <sub>3</sub> -600-1.0		25.02	
$C_{1}$ -600-0.0		33.98	
$C_{2}-600-0.0$	C-600-0.0	33.85	33.82
$C_{3}$ -600-0.0		33.62	
$C_1$ -600-1.0	- E	26.00	
C <sub>2</sub> -600-1.0	C-600-1.0	26.19	25.83
C <sub>3</sub> -600-1.0	1 AGC	25.31	-
A <sub>1</sub> -700-0.1	Parts.	25.17	
A <sub>2</sub> -700-0.1	A-700-0.1	24.32	25.22
A <sub>3</sub> -700-0.1		26.16	
A <sub>1</sub> -700-0.5	F	23.26	7
A <sub>2</sub> -700-0.5	A-700-0.5	23.43	23.85
A <sub>3</sub> -700-0.5	5	24.85	-
A <sub>1</sub> -700-1.0	S C	21.85	
A <sub>2</sub> -700-1.0	A-700-1.0	20.55	21.76
A <sub>3</sub> -700-1.0	03	22.89	_
B <sub>1</sub> -700-0.1		26.04	
B <sub>2</sub> -700-0.1	B-700-0.1	25.56	26.14
B <sub>3</sub> -700-0.1		26.82	_
B <sub>1</sub> -700-0.5		23.78	
B <sub>2</sub> -700-0.5	B-700-0.5	23.77	23.70
B <sub>3</sub> -700-0.5		23.55	_
B <sub>1</sub> -700-1.0		20.21	
B <sub>2</sub> -700-1.0	B-700-1.0	22.32	21.46
B <sub>3</sub> -700-1.0		21.84	1
C <sub>1</sub> -700-0.0		25.38	
C <sub>2</sub> -700-0.0	C-700-0.0	26.38	25.88

C <sub>3</sub> -700-0.0		25.87	
C <sub>1</sub> -700-1.0		22.59	
C <sub>2</sub> -700-1.0	C-700-1.0	22.05	22.59
C <sub>3</sub> -700-1.0		23.12	
A <sub>1</sub> -800-0.1		23.64	
A <sub>2</sub> -800-0.1	A-800-0.1	21.63	21.54
A <sub>3</sub> -800-0.1		19.34	
A <sub>1</sub> -800-0.5		20.68	
A <sub>2</sub> -800-0.5	A-800-0.5	18.93	18.93
A <sub>3</sub> -800-0.5		17.17	
A <sub>1</sub> -800-1.0		16.47	
A <sub>2</sub> -800-1.0	A-800-1.0	15.65	16.04
A <sub>3</sub> -800-1.0		15.99	
A <sub>1</sub> -800-1.5		15.63	
A <sub>2</sub> -800-1.5	A-800-1.5	13.62	14.55
A <sub>3</sub> -800-1.5		14.11	-
A <sub>1</sub> -800-2.0		11.94	
A <sub>2</sub> -800-2.0	A-800-2.0	12.22	12.27
A <sub>3</sub> -800-2.0		12.65	
B <sub>1</sub> -800-0.1		22.20	
B <sub>2</sub> -800-0.1	B-800-0.1	21.44	20.95
<b>B</b> <sub>3</sub> -800-0.1		19.22	-
B <sub>1</sub> -800-0.5		19.73	
B <sub>2</sub> -800-0.5	B-800-0.5	18.94	18.86
B <sub>3</sub> -800-0.5		17.91	
B <sub>1</sub> -800-1.0	CEE	17.40	
B <sub>2</sub> -800-1.0	B-800-1.0	16.39	16.32
<b>B</b> <sub>3</sub> -800-1.0		15.19	-
B <sub>1</sub> -800-1.5	RUG	13.95	
B <sub>2</sub> -800-1.5	B-800-1.5	14.24	14.17
B <sub>3</sub> -800-1.5		14.33	
B <sub>1</sub> -800-2.0		12.39	1
B <sub>2</sub> -800-2.0	B-800-2.0	13.47	12.68
B <sub>3</sub> -800-2.0		12.17	-
C <sub>1</sub> -800-0.0	SR	20.34	
C <sub>2</sub> -800-0.0	C-800-0.0	20.17	20.13
C <sub>3</sub> -800-0.0		19.88	1
C <sub>1</sub> -800-1.0		16.40	
C <sub>2</sub> -800-1.0	C-800-1.0	17.88	16.88
C <sub>3</sub> -800-1.0		16.37	1

# APPENDIX F: RESULTS OF FRESH CARBON ACTIVITY ANALYSIS

SAMPI	LE .ID.	DETERMINATIONS	MEAN VALUE
		(%)	(%)
	A <sub>1</sub> -600-0.1	7.9	
A-600-0.1	A <sub>2</sub> -600-0.1	6.4	7.03
	A <sub>3</sub> -600-0.1	6.8	
	A <sub>1</sub> -600-0.5	8.2	
A-600-0.5	A <sub>2</sub> -600-0.5	7.6	7.70
	A <sub>3</sub> -600-0.5	7.3	
	A <sub>1</sub> -600-1.0	12.1	
A-600-1.0	A <sub>2</sub> -600-1.0	10.8	11.60
	A <sub>3</sub> -600-1.0	11.9	
	B <sub>1</sub> -600-0.1	6.6	
<b>B-600-0.1</b>	B <sub>2</sub> -600-0.1	5.3	5.93
	B <sub>3</sub> -600-0.1	5.9	
	B <sub>1</sub> -600-0.5	7.5	
B-600-0.5	B <sub>2</sub> -600-0.5	7.7	7.53
	B <sub>3</sub> -600-0.5	7.4	
	B <sub>1</sub> -600-1.0	8.9	
<b>B-600-1.0</b>	B <sub>2</sub> -600-1.0	10.1	9.87
	B <sub>3</sub> -600-1.0	10.6	
	C <sub>1</sub> -600-0.0	2.4	
C-600-0.0	C <sub>2</sub> -600-0.0	2.1	2.27
	C <sub>3</sub> -600-0.0	2.3	
	C <sub>1</sub> -600-1.0	11.0	
C-600-1.0	C <sub>2</sub> -600-1.0	11.6	11.23
	C <sub>3</sub> -600-1.0	11.2	
	A <sub>1</sub> -700-0.1	37.6	
A-700-0.1	A <sub>2</sub> -700-0.1	40.8	38.57
	A <sub>3</sub> -700-0.1	37.3	
Z	A <sub>1</sub> -700-0.5	72.2	7
A-700-0.5	A <sub>2</sub> -700-0.5	75.2	73.07
	A <sub>3</sub> -700-0.5	71.8	
	A <sub>1</sub> -700-1.0	84.4	
A-700-1.0	A <sub>2</sub> -700-1.0	83.1	83.90
	A <sub>3</sub> -700-1.0	84.2	
	B <sub>1</sub> -700-0.1	38.4	
B-700-0.1	B <sub>2</sub> -700-0.1	36.7	37.40
	B <sub>3</sub> -700-0.1	37.1	
	B <sub>1</sub> -700-0.5	72.0	
B-700-0.5	B <sub>2</sub> -700-0.5	71.5	71.30
	B <sub>3</sub> -700-0.5	70.4	
	B <sub>1</sub> -700-1.0	80.9	
B-700-1.0	B <sub>2</sub> -700-1.0	79.6	79.60
	B <sub>3</sub> -700-1.0	78.3	1
	C <sub>1</sub> -700-0.0	30.4	
C-700-0.0	C <sub>2</sub> -700-0.0	27.4	28.13

	C <sub>3</sub> -700-0.0	26.6	
	C <sub>1</sub> -700-1.0	80.1	
C-700-1.0	C <sub>2</sub> -700-1.0	79.8	79.50
	C <sub>3</sub> -700-1.0	78.6	
	A <sub>1</sub> -800-0.1	68.3	
A-800-0.1	A <sub>2</sub> -800-0.1	68.8	68.17
	A <sub>3</sub> -800-0.1	67.4	
	A <sub>1</sub> -800-0.5	81.0	
A-800-0.5	A <sub>2</sub> -800-0.5	82.1	81.63
	A <sub>3</sub> -800-0.5	81.8	
	A <sub>1</sub> -800-1.0	92.7	
A-800-1.0	A <sub>2</sub> -800-1.0	92.1	91.67
	A <sub>3</sub> -800-1.0	90.2	
	A <sub>1</sub> -800-1.5	83.6	
A-800-1.5	A <sub>2</sub> -800-1.5	84.7	84.13
	A <sub>3</sub> -800-1.5	84.1	
A-800-2.0	A <sub>1</sub> -800-2.0	69.9	
	A <sub>2</sub> -800-2.0	69.7	69.93
	A <sub>3</sub> -800-2.0	70.2	
	B <sub>1</sub> -800-0.1	66.9	
<b>B-800-0.1</b>	B <sub>2</sub> -800-0.1	68.4	67.33
	B <sub>3</sub> -800-0.1	66.7	
	B <sub>1</sub> -800-0.5	80.1	
B-800-0.5	B <sub>2</sub> -800-0.5	81.3	80.63
	B <sub>3</sub> -800-0.5	80.5	
	B <sub>1</sub> -800-1.0	88.4	
<b>B-800-1.0</b>	B <sub>2</sub> -800-1.0	87.1	87.47
	B <sub>3</sub> -800-1.0	86.9	
	B <sub>1</sub> -800-1.5	73.3	
B-800-1.5	B <sub>2</sub> -800-1.5	72.8	72.77
	B <sub>3</sub> -800-1.5	72.2	
12	B <sub>1</sub> -800-2.0	61.4	<
B-800-2.0	B <sub>2</sub> -800-2.0	61.1	61.40
	B <sub>3</sub> -800-2.0	61.7	
	C <sub>1</sub> -800-0.0	40.8	
C-800-0.0	C <sub>2</sub> -800-0.0	41.5	41.13
	C <sub>3</sub> -800-0.0	41.1	
	C <sub>1</sub> -800-1.0	91.4	
C-800-1.0	C <sub>2</sub> -800-1.0	88.6	88.83
	C <sub>3</sub> -800-1.0	86.5	

SAMPLE .ID.		Mass Before	Mass After Stirring	Attrition (%)	MEAN VALUE
		Stirring (g)	and Drying (g)		(%)
	A <sub>1</sub> -600-0.1	1.016	0.966	4.92	
A-600-0.1	A <sub>2</sub> -600-0.1	1.102	1.042	5.44	4.86
	A <sub>3</sub> -600-0.1	1.161	1.112	4.22	
	A <sub>1</sub> -600-0.5	1.011	0.879	13.06	
A-600-0.5	A <sub>2</sub> -600-0.5	1.130	0.986	12.74	12.33
	A <sub>3</sub> -600-0.5	1.072	0.952	11.19	
	A <sub>1</sub> -600-1.0	1.021	0.801	21.55	
A-600-1.0	A <sub>2</sub> -600-1.0	1.047	0.861	17.77	18.82
	A <sub>3</sub> -600-1.0	1.015	0.841	17.14	
	B <sub>1</sub> -600-0.1	1.019	0.975	4.32	
B-600-0.1	<b>B</b> <sub>2</sub> -600-0.1	1.098	1.007	8.29	6.28
	B <sub>3</sub> -600-0.1	1.171	1.098	6.23	-
	B <sub>1</sub> -600-0.5	1.003	0.862	14.06	
B-600-0.5	B <sub>2</sub> -600-0.5	1.204	1.027	14.70	13.97
	B <sub>3</sub> -600-0.5	1.096	0.952	13.14	
	<b>B</b> <sub>1</sub> -600-1.0	1.012	0.811	19.86	
B-600-1.0	<b>B</b> <sub>2</sub> -600-1.0	1.132	0.921	18.64	19.41
	<b>B</b> <sub>3</sub> -600-1.0	1.100	0.883	19.72	
	C <sub>1</sub> -600-0.0	1.027	0.986	3.99	
C-600-0.0	C <sub>2</sub> -600-0.0	1.003	0.962	4.09	4.06
	C <sub>3</sub> -600-0.0	1.046	1.003	4.11	
	C <sub>1</sub> -600-1.0	1.007	0.798	20.75	
C-600-1.0	C <sub>2</sub> -600-1.0	1.063	0.856	19.47	20.62
	C <sub>3</sub> -600-1.0	1.317	1.032	21.64	-
	A <sub>1</sub> -700-0.1	1.001	0.877	12.39	
A-700-0.1	A <sub>2</sub> -700-0.1	1.086	0.981	9.67	11.25
	A <sub>3</sub> -700-0.1	1.002	0.885	11.68	]
	A <sub>1</sub> -700-0.5	1.091	0.874	19.89	

# APPENDIX G: RESULTS OF HARDNESS (ATTRITION) ANALYSIS

A-700-0.5	A <sub>2</sub> -700-0.5	1.148	0.942	17.94	18.28
	A <sub>3</sub> -700-0.5	1.306	1.084	17.00	
	A <sub>1</sub> -700-1.0	1.108	0.801	27.71	
A-700-1.0	A <sub>2</sub> -700-1.0	1.015	0.763	24.83	24.96
	A <sub>3</sub> -700-1.0	1.160	0.901	22.33	
	B <sub>1</sub> -700-0.1	1.101	0.973	11.63	
B-700-0.1	<b>B</b> <sub>2</sub> -700-0.1	1.217	1.073	11.83	10.53
	<b>B</b> <sub>3</sub> -700-0.1	1.047	0.962	8.12	
	B <sub>1</sub> -700-0.5	1.052	0.835	20.63	
B-700-0.5	B <sub>2</sub> -700-0.5	1.108	0.932	15.88	17.88
	B <sub>3</sub> -700-0.5	1.103	0.914	17.14	
	B <sub>1</sub> -700-1.0	1.127	0.843	25.20	
B-700-1.0	B <sub>2</sub> -700-1.0	1.004	0.801	20.22	23.20
	B <sub>3</sub> -700-1.0	1.204	0.913	24.17	
	C <sub>1</sub> -700-0.0	1.008	0.918	8.93	
C-700-0.0	C <sub>2</sub> -700-0.0	1.203	1.097	8.81	7.99
	C <sub>3</sub> -700-0.0	1.076	1.009	6.23	
	C <sub>1</sub> -700-1.0	1.301	0.924	28.98	
C-700-1.0	C <sub>2</sub> -700-1.0	1.016	0.754	25.79	27.03
	C <sub>3</sub> -700-1.0	1.011	0.745	26.31	
	A <sub>1</sub> -800-0.1	1.009	0.801	20.61	
A-800-0.1	A <sub>2</sub> -800-0.1	1.010	0.816	19.21	21.04
	A <sub>3</sub> -800-0.1	1.283	0.984	23.30	-
	A <sub>1</sub> -800-0.5	1.013	0.681	32.77	
A-800-0.5	A <sub>2</sub> -800-0.5	1.074	0.736	31.47	32.64
	A <sub>3</sub> -800-0.5	1.200	0.796	33.67	]
	A <sub>1</sub> -800-1.0	1.014	0.564	44.38	
A-800-1.0	A <sub>2</sub> -800-1.0	1.044	0.632	39.46	]
	A <sub>3</sub> -800-1.0	1.109	0.674	39.22	41.02

	A <sub>1</sub> -800-1.5	1.107	0.624	43.63	
A-800-1.5	A <sub>2</sub> -800-1.5	1.091	0.596	45.37	44.28
	A <sub>3</sub> -800-1.5	1.033	0.580	43.85	
	A <sub>1</sub> -800-2.0	1.079	0.563	47.82	
A-800-2.0	A <sub>2</sub> -800-2.0	1.112	0.591	46.85	47.44
	A <sub>3</sub> -800-2.0	1.043	0.546	47.65	
	<b>B</b> <sub>1</sub> -800-0.1	1.042	0.796	23.61	
B-800-0.1	<b>B</b> <sub>2</sub> -800-0.1	1.006	0.784	22.07	21.82
	<b>B</b> <sub>3</sub> -800-0.1	1.138	0.913	19.77	
	B <sub>1</sub> -800-0.5	1.006	0.698	30.62	
B-800-0.5	B <sub>2</sub> -800-0.5	1.180	0.857	27.37	29.13
	B <sub>3</sub> -800-0.5	1.214	0.857	29.41	
	<b>B</b> <sub>1</sub> -800-1.0	1.001	0.622	37.86	
B-800-1.0	<b>B</b> <sub>2</sub> -800-1.0	1.007	0.635	36.94	37.17
	<b>B</b> <sub>3</sub> -800-1.0	1.011	0.640	36.70	
	B <sub>1</sub> -800-1.5	1.141	0.638	44.08	
B-800-1.5	B <sub>2</sub> -800-1.5	1.108	0.642	42.06	43.32
	<b>B</b> <sub>3</sub> -800-1.5	1.004	0.564	43.82	
	B <sub>1</sub> -800-2.0	1.121	0.610	45.58	
B-800-2.0	<b>B</b> <sub>2</sub> -800-2.0	1.005	0.537	46.57	46.26
	<b>B</b> <sub>3</sub> -800-2.0	1.068	0.570	46.63	
	C <sub>1</sub> -800-0.0	1.012	0.876	13.44	
C-800-0.0	C <sub>2</sub> -800-0.0	1.292	1.148	11.15	13.07
	C <sub>3</sub> -800-0.0	1.012	0.864	14.62	
	C <sub>1</sub> -800-1.0	1.003	0.589	41.28	
C-800-1.0	C <sub>2</sub> -800-1.0	1.017	0.607	40.31	40.56
	C <sub>3</sub> -800-1.0	1.170	0.701	40.09	
	S <sub>1</sub> -GAC	1.068	0.986	7.68	
S-GAC	S <sub>2</sub> -GAC	1.021	0.947	7.25	7.80
F	S <sub>3</sub> -GAC	1.003	0.918	8.47	

# APPENDIX H: RESULTS OF PARTICLE SIZE RETENTION ANALYSIS

SAMPI	LE .ID.	DETERMINATIONS	MEAN VALUE
		(%)	(%)
	A <sub>1</sub> -600-0.1	82.85	
A-600-0.1	A <sub>2</sub> -600-0.1	80.53	81.67
	A <sub>3</sub> -600-0.1	81.64	
	A <sub>1</sub> -600-0.5	86.11	
A-600-0.5	A <sub>2</sub> -600-0.5	86.04	84.98
	A <sub>3</sub> -600-0.5	82.80	
	A <sub>1</sub> -600-1.0	87.65	
A-600-1.0	A <sub>2</sub> -600-1.0	91.13	88.65
	A <sub>3</sub> -600-1.0	87.18	
	B <sub>1</sub> -600-0.1	81.18	
<b>B-6</b> 00-0.1	B <sub>2</sub> -600-0.1	81.21	82.26
	B <sub>3</sub> -600-0.1	84.38	
	B <sub>1</sub> -600-0.5	82.46	
B-600-0.5	B <sub>2</sub> -600-0.5	85.09	84.80
	B <sub>3</sub> -600-0.5	86.86	
	B <sub>1</sub> -600-1.0	90.50	
<b>B-6</b> 00-1.0	B <sub>2</sub> -600-1.0	90.00	90.25
	B <sub>3</sub> -600-1.0	90.26	
	C <sub>1</sub> -600-0.0	58.42	
C-600-0.0	C <sub>2</sub> -600-0.0	59.35	58.98
	C <sub>3</sub> -600-0.0	59.16	
	C <sub>1</sub> -600-1.0	82.26	
C-600-1.0	C <sub>2</sub> -600-1.0	88.27	86.06
	C <sub>3</sub> -600-1.0	87.06	
	A <sub>1</sub> -700-0.1	77.51	
A-700-0.1	A <sub>2</sub> -700-0.1	76.70	76.86
	A <sub>3</sub> -700-0.1	76.36	
	A <sub>1</sub> -700-0.5	83.13	7
A-700-0.5	A <sub>2</sub> -700-0.5	81.81	82.83
1	A <sub>3</sub> -700-0.5	83.56	
	A <sub>1</sub> -700-1.0	85.25	
A-700-1.0	A <sub>2</sub> -700-1.0	85.45	85.63
	A <sub>3</sub> -700-1.0	86.18	
	B <sub>1</sub> -700-0.1	80.08	
B-700-0.1	B <sub>2</sub> -700-0.1	80.67	79.43
	B <sub>3</sub> -700-0.1	77.55	
	B <sub>1</sub> -700-0.5	82.53	
B-700-0.5	B <sub>2</sub> -700-0.5	81.62	81.19
	B <sub>3</sub> -700-0.5	79.42	
	B <sub>1</sub> -700-1.0	85.06	
B-700-1.0	B <sub>2</sub> -700-1.0	84.21	83.93
	B <sub>3</sub> -700-1.0	82.51	
	$C_1 - 700 - 0.0$	53.84	
C-700-0.0	$C_2 - 700 - 0.0$	56.38	55.12

	C <sub>3</sub> -700-0.0	55.13	
	C <sub>1</sub> -700-1.0	79.24	
C-700-1.0	C <sub>2</sub> -700-1.0	85.82	82.77
	C <sub>3</sub> -700-1.0	83.25	
	A <sub>1</sub> -800-0.1	71.18	
A-800-0.1	A <sub>2</sub> -800-0.1	70.07	70.35
	A <sub>3</sub> -800-0.1	69.80	
	A <sub>1</sub> -800-0.5	75.90	
A-800-0.5	A <sub>2</sub> -800-0.5	80.13	78.86
	A <sub>3</sub> -800-0.5	80.54	
	A <sub>1</sub> -800-1.0	80.57	
A-800-1.0	A <sub>2</sub> -800-1.0	81.65	79.75
	A <sub>3</sub> -800-1.0	77.04	
	A <sub>1</sub> -800-1.5	84.95	
A-800-1.5	A <sub>2</sub> -800-1.5	84.50	84.44
	A <sub>3</sub> -800-1.5	83.86	
	A <sub>1</sub> -800-2.0	90.74	
A-800-2.0	A <sub>2</sub> -800-2.0	90.48	90.21
	A <sub>3</sub> -800-2.0	89.42	
	B <sub>1</sub> -800-0.1	69.22	
<b>B-800-0.1</b>	B <sub>2</sub> -800-0.1	69.57	69.93
	B <sub>3</sub> -800-0.1	71.01	
	B <sub>1</sub> -800-0.5	75.57	
B-800-0.5	B <sub>2</sub> -800-0.5	77.23	77.09
	B <sub>3</sub> -800-0.5	78.47	
	B <sub>1</sub> -800-1.0	79.70	
<b>B-800-1.0</b>	B <sub>2</sub> -800-1.0	80.83	79.88
	B <sub>3</sub> -800-1.0	79.11	
	B <sub>1</sub> -800-1.5	82.52	
B-800-1.5	B <sub>2</sub> -800-1.5	83.62	83.21
	B <sub>3</sub> -800-1.5	83.48	
IZ	B <sub>1</sub> -800-2.0	87.67	
B-800-2.0	B <sub>2</sub> -800-2.0	89.93	88.78
	<b>B</b> <sub>3</sub> -800-2.0	88.74	
	C <sub>1</sub> -800-0.0	53.88	
C-800-0.0	C <sub>2</sub> -800-0.0	56.91	54.53
	C <sub>3</sub> -800-0.0	52.81	
	C <sub>1</sub> -800-1.0	79.82	
C-800-1.0	C <sub>2</sub> -800-1.0	74.47	77.72
	C <sub>3</sub> -800-1.0	78.87	
<b>APPENDIX I:</b>	RESULTS	<b>OF MOISTURE</b>	ANALYSIS
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SAMP	PLE .ID.	Crucible + Lid (g)	Crucible + Lid + AC	Crucible + Lid + AC	Moisture	MEAN VALUE	
			(g)	After Drying (g)	(%)	(%)	
	A <sub>1</sub> -600-0.1	36.878	42.516	42.385	2.32		
A-600-0.1	A <sub>2</sub> -600-0.1	38.878	42.518	42.446	1.98	2.23	
	A <sub>3</sub> -600-0.1	36.878	41.086	40.985	2.40		
	A <sub>1</sub> -600-0.5	38.153	43.214	43.097	2.31		
A-600-0.5	A <sub>2</sub> -600-0.5	34.623	39.811	39.654	3.03	2.56	
	A <sub>3</sub> -600-0.5	35.153	40.175	40.058	2.33		
	A <sub>1</sub> -600-1.0	35.899	40.954	40.841	2.34		
A-600-1.0	A <sub>2</sub> -600-1.0	35.389	39.738	39.641	2.23	2.47	
	A <sub>3</sub> -600-1.0	35.879	41.313	41.158	2.85		
	B <sub>1</sub> -600-0.1	46.848	51.781	51.659	2.47		
B-600-0.1	B <sub>2</sub> -600-0.1	34.616	40.522	40.331	3.23	2.67	
	B <sub>3</sub> -600-0.1	46.848	51.405	51.300	2.30		
	B <sub>1</sub> -600-0.5	36.397	41.455	41.339	2.29		
B-600-0.5	B <sub>2</sub> -600-0.5	33.576	38.472	38.355	2.39	2.42	
	B <sub>3</sub> -600-0.5	36.398	41.739	41.602	2.57		
	B <sub>1</sub> -600-1.0	34.850	39.786	39.669	2.37		
B-600-1.0	B <sub>2</sub> -600-1.0	34.715	38.425	38.352	2.00	2.53	
	B <sub>3</sub> -600-1.0	34.852	39.945	39.781	3.22		
	C <sub>1</sub> -600-0.0	36.657	41.672	41.541	2.61		
C-600-0.0	C <sub>2</sub> -600-0.0	45.540	50.121	49.989	2.88	2.81	
	C <sub>3</sub> -600-0.0	36.657	41.746	41.597	2.93		
	C <sub>1</sub> -600-1.0	45.530	49.685	49.595	2.17		
C-600-1.0	C <sub>2</sub> -600-1.0	34.619	38.900	38.795	2.45	2.41	
	C <sub>3</sub> -600-1.0	45.531	50.008	49.891	2.61		
	A <sub>1</sub> -700-0.1	35.991	41.022	40.896	2.50		
A-700-0.1	A <sub>2</sub> -700-0.1	35.991	40.485	40.378	2.38	2.37	
	A <sub>3</sub> -700-0.1	34.619	39.457	39.349	2.23	]	

	A <sub>1</sub> -700-0.5	38.154	43.186	43.072	2.27		
A-700-0.5	A <sub>2</sub> -700-0.5	38.154	43.506	43.371	2.52	2.37	
	A <sub>3</sub> -700-0.5	35.991	40.445	40.342	2.31		
	A <sub>1</sub> -700-1.0	35.899	40.951	40.817	2.65		
A-700-1.0	A <sub>2</sub> -700-1.0	35.899	40.253	40.134	2.73	2.69	
	A <sub>3</sub> -700-1.0	38.154	43.256	43.118	2.70		
	B <sub>1</sub> -700-0.1	46.849	51.816	51.701	2.32		
B-700-0.1	B <sub>2</sub> -700-0.1	46.859	51.005	50.908	2.34	2.36	
	B <sub>3</sub> -700-0.1	35.899	41.212	41.084	2.41		
	B <sub>1</sub> -700-0.5	36.399	41.401	41.265	2.72		
B-700-0.5	B <sub>2</sub> -700-0.5	36.399	42.179	42.002	3.06	2.87	
	B <sub>3</sub> -700-0.5	46.860	51.631	51.496	2.83		
	B <sub>1</sub> -700-1.0	34.851	39.803	39.678	2.52		
B-700-1.0	B <sub>2</sub> -700-1.0	34.851	39.402	39.259	3.14	2.71	
	B <sub>3</sub> -700-1.0	36.399	40.869	40.759	2.46		
	C <sub>1</sub> -700-0.0	36.658	41.687	41.534	3.04		
C-700-0.0	C <sub>2</sub> -700-0.0	36.657	40.962	40.859	2.39	2.84	
	C <sub>3</sub> -700-0.0	34.851	40.010	39.851	3.08		
	C <sub>1</sub> -700-1.0	45.531	50.171	50.058	2.44		
C-700-1.0	C <sub>2</sub> -700-1.0	45.531	50.459	50.327	2.68	2.62	
	C <sub>3</sub> -700-1.0	36.656	41.274	41.148	2.73		
	A <sub>1</sub> -800-0.1	35.391	40.809	40.652	2.90		
A-800-0.1	A <sub>2</sub> -800-0.1	35.990	40.251	40.128	2.89	2.77	
	A <sub>3</sub> -800-0.1	45.598	50.256	50.139	2.51		
	A <sub>1</sub> -800-0.5	34.851	38.548	38.471	2.08		
A-800-0.5	A <sub>2</sub> -800-0.5	38.154	43.638	43.469	3.08	2.36	
	A <sub>3</sub> -800-0.5	36.878	40.961	40.833	1.91		
	A <sub>1</sub> -800-1.0	36.888	41.618	41.498	2.54		
A-800-1.0	A <sub>2</sub> -800-1.0	35.899	40.105	39.988	2.78	2.63	
	A <sub>3</sub> -800-1.0	35.153	40.058	39.932	2.57		
	A <sub>1</sub> -800-1.5	36.398	40.561	40.467	2.26		

A-800-1.5	A <sub>2</sub> -800-1.5	34.853	39.483	39.373	2.38	2.28
	A <sub>3</sub> -800-1.5	36.657	40.982	40.887	2.20	
	A <sub>1</sub> -800-1.5	45.598	49.501	49.411	2.31	
A-800-2.0	A <sub>2</sub> -800-1.5	36.878	40.673	40.572	2.66	2.46
	A <sub>3</sub> -800-1.5	35.253	39.418	39.315	2.42	
	B <sub>1</sub> -800-0.1	34.718	39.877	39.732	2.81	
B-800-0.1	B <sub>2</sub> -800-0.1	46.849	51.621	51.497	2.60	2.75
	B <sub>3</sub> -800-0.1	35.899	41.060	40.913	2.85	
	B <sub>1</sub> -800-0.5	36.399	40.698	40.572	2.93	
B-800-0.5	B <sub>2</sub> -800-0.5	36.400	40.451	40.371	1.97	2.42
	B <sub>3</sub> -800-0.5	46.848	51.785	51.668	2.37	
	B <sub>1</sub> -800-1.0	34.854	39.103	38.989	2.68	
B-800-1.0	B <sub>2</sub> -800-1.0	34.851	39.235	39.126	2.49	2.52
	B <sub>3</sub> -800-1.0	36.397	41.098	40.986	2.38	
	B <sub>1</sub> -800-1.5	35.899	40.507	40.414	2.02	
B-800-1.5	B <sub>2</sub> -800-1.5	46.848	51.115	51.008	2.51	2.37
	B <sub>3</sub> -800-1.5	34.851	39.143	39.032	2.59	
	B <sub>1</sub> -800-2.0	35.899	39.984	39.883	2.47	
B-800-2.0	B <sub>2</sub> -800-2.0	46.848	51.007	50.896	2.67	2.45
	B <sub>3</sub> -800-2.0	36.397	40.502	40.411	2.22	
	C <sub>1</sub> -800-0.0	33.578	39.075	38.894	3.29	
C-800-0.0	C <sub>2</sub> -800-0.0	36.658	40.954	40.951	2.40	2.88
	C <sub>3</sub> -800-0.0	34.850	39.401	39.267	2.94	
	C <sub>1</sub> -800-1.0	34.619	39.159	39.059	2.20	
C-800-1.0	C <sub>2</sub> -800-1.0	45.531	50.708	50.591	2.26	2.16
	C <sub>3</sub> -800-1.0	36.657	41.207	41.115	2.02	

#### APPENDIX J: ASH CONTENT ANALYSIS

SAME	PLE .ID.	Empty	Crucible $+AC(g)$	Crucible +AC After	Ash Content	MEAN VALUE	
		Crucible(g)		Ashing (g)	(%)	(%)	
	A <sub>1</sub> -600-0.1	19.363	20.306	19.391	2.97		
A-600-0.1	A <sub>2</sub> -600-0.1	19.363	20.345	19.393	3.05	2.98	
	A <sub>3</sub> -600-0.1	19.363	20.322	19.391	2.92		
	A <sub>1</sub> -600-0.5	17.088	18.123	17.122	3.29		
A-600-0.5	A <sub>2</sub> -600-0.5	17.088	18.110	17.124	3.52	3.46	
	A <sub>3</sub> -600-0.5	17.088	18.121	17.125	3.58		
	A <sub>1</sub> -600-1.0	17.847	18.961	17.890	3.86		
A-600-1.0	A <sub>2</sub> -600-1.0	17.847	18 <mark>.864</mark>	17.887	3.93	3.94	
	A <sub>3</sub> -600-1.0	17.847	18.568	17.876	4.02		
	B <sub>1</sub> -600-0.1	17.198	18.369	17.231	2.82		
B-600-0.1	B <sub>2</sub> -600-0.1	17.198	18.194	17.218	2.89	2.95	
	B <sub>3</sub> -600-0.1	17.198	18.245	17.231	3.15		
	B <sub>1</sub> -600-0.5	16.023	17.369	16.068	3.34		
B-600-0.5	B <sub>2</sub> -600-0.5	16.023	17.214	16.066	3.61	3.52	
	B <sub>3</sub> -600-0.5	16.023	17.134	16.063	3.60		
	B <sub>1</sub> -600-1.0	17.124	18.495	17.177	3.87		
B-600-1.0	B <sub>2</sub> -600-1.0	17.124	18.195	17.167	4.01	4.00	
	B <sub>3</sub> -600-1.0	17.124	18.310	17.173	4.13		
	C <sub>1</sub> -600-0.0	17.192	18.390	17.221	2.44		
C-600-0.0	C <sub>2</sub> -600-0.0	17.192	18.003	17.214	2.71	2.76	
	C <sub>3</sub> -600-0.0	17.192	18.211	17.224	3.14		
	C <sub>1</sub> -600-1.0	28.110	29.092	28.151	4.18		
C-600-1.0	C <sub>2</sub> -600-1.0	28.110	29.211	28.155	4.09		
	C <sub>3</sub> -600-1.0	28.110	29.136	28.152	4.09	4.12	
	A <sub>1</sub> -700-0.1	17.088	18.201	17.124	3.23		
A-700-0.1	A <sub>2</sub> -700-0.1	19.363	20.402	19.398	3.37	3.38	
	A <sub>3</sub> -700-0.1	19.363	20.411	19.400	3.53		

	A <sub>1</sub> -700-0.5	19.363	20.376	19.402	3.87	
A-700-0.5	A <sub>2</sub> -700-0.5	17.088	18.122	17.127	3.81	3.98
	A <sub>3</sub> -700-0.5	17.088	18.120	17.132	4.26	
	A <sub>1</sub> -700-1.0	17.847	18.794	17.891	4.65	
A-700-1.0	A <sub>2</sub> -700-1.0	17.847	18.913	17.895	4.50	4.66
	A <sub>3</sub> -700-1.0	17.847	18.822	17.897	4.83	
	B <sub>1</sub> -700-0.1	17.198	18.203	17.233	3.48	
B-700-0.1	B <sub>2</sub> -700-0.1	17.198	18.264	17.234	3.38	3.54
	B <sub>3</sub> -700-0.1	17.198	18.211	17.236	3.75	
	B <sub>1</sub> -700-0.5	16.023	17.101	16.067	4.08	
B-700-0.5	B <sub>2</sub> -700-0.5	16.023	17.225	16.072	4.08	4.11
	B <sub>3</sub> -700-0.5	16.023	17.211	16.063	4.17	
	B <sub>1</sub> -700-1.0	17.124	18.114	17.169	4.55	
B-700-1.0	B <sub>2</sub> -700-1.0	17.124	18.168	17.174	4.79	4.93
	B <sub>3</sub> -700-1.0	17.124	18.222	17.184	5.46	
	C <sub>1</sub> -700-0.0	17.192	18.201	17.224	3.17	
C-700-0.0	C <sub>2</sub> -700-0.0	17.192	18.214	17.224	3.13	3.24
	C <sub>3</sub> -700-0.0	17.192	18.211	17.227	3.43	
	C <sub>1</sub> -700-1.0	28.110	29.001	28.15	4.49	
C-700-1.0	C <sub>2</sub> -700-1.0	28.110	29.201	28.16	4.58	4.91
	C <sub>3</sub> -700-1.0	28.110	29.312	28.178	5.66	
	A <sub>1</sub> -800-0.1	17.847	1 <mark>8.871</mark>	17.892	4.39	
A-800-0.1	A <sub>2</sub> -800-0.1	19.363	20.375	19.407	4.35	4.38
	A <sub>3</sub> -800-0.1	19.363	20.431	19.410	4.40	
	A <sub>1</sub> -800-0.5	17.124	18.116	17.174	5.04	
A-800-0.5	A <sub>2</sub> -800-0.5	17.088	18.096	17.138	4.96	5.05
	A <sub>3</sub> -800-0.5	17.088	18.195	17.145	5.15	
	A <sub>1</sub> -800-1.0	17.192	18.189	17.249	5.72	
A-800-1.0	A <sub>2</sub> -800-1.0	17.847	18.924	17.908	5.66	5.80
	A <sub>3</sub> -800-1.0	17.847	18.860	17.908	6.02	
	A <sub>1</sub> -800-1.5	35.899	36.910	35.963	6.33	

A-800-1.5	A <sub>2</sub> -800-1.5	35.153	36.321	35.226	6.25	6.27
	A <sub>3</sub> -800-1.5	36.398	37.407	36.461	6.24	
	A <sub>1</sub> -800-2.0	34.853	35.932	34.924	6.58	
A-800-2.0	A <sub>2</sub> -800-2.0	36.657	37.775	36.731	6.62	6.61
	A <sub>3</sub> -800-2.0	35.256	36.281	35.324	6.63	
	B <sub>1</sub> -800-0.1	28.110	29.182	28.161	4.76	
B-800-0.1	B <sub>2</sub> -800-0.1	17.198	18.232	17.245	4.55	4.54
	B <sub>3</sub> -800-0.1	17.198	18.240	17.243	4.32	
	B <sub>1</sub> -800-0.5	17.088	18.132	17.144	5.36	
B-800-0.5	B <sub>2</sub> -800-0.5	16.023	17.008	16.073	5.08	5.13
	B <sub>3</sub> -800-0.5	16.023	17.132	16.078	4.96	
	B <sub>1</sub> -800-1.0	19.363	20.461	19.427	5.83	
B-800-1.0	B <sub>2</sub> -800-1.0	17.124	18.226	17.187	5.72	5.80
	B <sub>3</sub> -800-1.0	17.124	18.233	17.189	5.86	
	B <sub>1</sub> -800-1.5	34.853	35.869	34.917	6.30	
B-800-1.5	B <sub>2</sub> -800-1.5	36.657	37.598	36.715	6.16	6.21
	B <sub>3</sub> -800-1.5	35.256	36.114	35.309	6.18	
	B <sub>1</sub> -800-2.0	35.153	36.068	35.211	6.34	
B-800-2.0	B <sub>2</sub> -800-2.0	36.398	37.400	36.461	6.29	6.33
	B <sub>3</sub> -800-2.0	35.897	36.921	35.962	6.35	
	C <sub>1</sub> -800-0.0	17.198	18.203	17.237	3.88	
C-800-0.0	C <sub>2</sub> -800-0.0	17.19 <mark>2</mark>	18.226	17.236	4.26	4.09
	C <sub>3</sub> -800-0.0	17.192	18.207	17.234	4.14	
	C <sub>1</sub> -800-1.0	16.023	17.101	16.087	5.94	
C-800-1.0	C <sub>2</sub> -800-1.0	28.110	29.112	28.166	5.59	5.82
	C <sub>3</sub> -800-1.0	28.110	29.221	28.176	5.94	

#### **APPENDIX K- APPARENT DENSITY**

SAMPLE .ID.		Mass of Rubber	Mass of Rubber	Volume of H <sub>2</sub> O	Determinations	MEAN VALUE
		bag (g)	Bag + AC(g)	Displaced (ml)	(g/ml)	(g/ml)
	A <sub>1</sub> -600-0.1	0.059	5.404	10	0.53	
A-600-0.1	A <sub>2</sub> -600-0.1	0.071	5.188	10	0.51	0.52
	A <sub>3</sub> -600-0.1	0.089	2.665	5	0.52	
	A <sub>1</sub> -600-0.5	0.700	4.615	12	0.33	
A-600-0.5	A <sub>2</sub> -600-0.5	0.074	5.346	17	0.31	0.32
	A <sub>3</sub> -600-0.5	0.100	2.610	8	0.31	
	A <sub>1</sub> -600-1.0	0.065	4.310	13	0.33	
A-600-1.0	A <sub>2</sub> -600-1.0	0.075	5.165	16	0.32	0.33
	A <sub>3</sub> -600-1.0	0.108	2.526	7	0.35	
	B <sub>1</sub> -600-0.1	0.060	5.314	10	0.53	
B-600-0.1	B <sub>2</sub> -600-0.1 0.065		5.138	10	0.51	0.54
	B <sub>3</sub> -600-0.1	0.111	2.399	4	0.57	
	B <sub>1</sub> -600-0.5	0.071	2.955	8	0.36	
B-600-0.5	B <sub>2</sub> -600-0.5 0.061		5.157	35-11	0.46	0.44
	B <sub>3</sub> -600-0.5	0.098	2.550	5	0.49	
	B <sub>1</sub> -600-1.0	0.075	3.648	11	0.32	
B-600-1.0	B <sub>2</sub> -600-1.0	0.069	5.355	13	0.41	0.37
	B <sub>3</sub> -600-1.0	0.083	2.345	6	0.38	
	C <sub>1</sub> -600-0.0	0.067	2.662	4	0.65	
C-600-0.0	C <sub>2</sub> -600-0.0	0.065	5.272	10	0.52	0.56
	C <sub>3</sub> -600-0.0	0.094	3.213	6	0.52	
	C <sub>1</sub> -600-1.0	0.050	3.566	12	0.29	
C-600-1.0	C <sub>2</sub> -600-1.0	0.062	3.402	13	0.26	0.29
	C <sub>3</sub> -600-1.0	0.076	3.683	11	0.33	
	A <sub>1</sub> -700-0.1	0.058	5.398	9	0.59	
A-700-0.1	A <sub>2</sub> -700-0.1	0.054	5.124	9	0.56	0.58
	A <sub>3</sub> -700-0.1	0.097	3.571	6	0.58	
	A <sub>1</sub> -700-0.5	0.065	5.009	16	0.31	0.35
A-700-0.5	A <sub>2</sub> -700-0.5	0.099	5.158	14	0.3	]

	A <sub>3</sub> -700-0.5	0.131	5.112	11	0.45		
	A <sub>1</sub> -700-1.0	0.054	2.590	10	0.25		
A-700-1.0	A <sub>2</sub> -700-1.0	0.078	5.161	16	0.32	0.32	
	A <sub>3</sub> -700-1.0	0.060	5.309	13	0.40	0.02	
	B <sub>1</sub> -700-0.1	0.056	3.943	7	0.56		
B-700-0.1	B <sub>2</sub> -700-0.1	0.056	5.326	9	0.59	0.58	
	B <sub>3</sub> -700-0.1	0.064	5.322	9	0.58		
	B <sub>1</sub> -700-0.5	0.075	4.490	14	0.32		
B-700-0.5	B <sub>2</sub> -700-0.5	0.050	5.111	D 11	0.46	0.40	
	B <sub>3</sub> -700-0.5	0.054	5.080	12	0.42		
	B <sub>1</sub> -700-1.0	0.050	2.782	12	0.23		
B-700-1.0	B <sub>2</sub> -700-1.0	0.069	5.121	13	0.39	0.32	
	B <sub>3</sub> -700-1.0	0.061	5.029	15	0.33		
C-700-0.0	C <sub>1</sub> -700-0.0	0.051	3.445	6	0.57		
	C <sub>2</sub> -700-0.0	0.073	5.087	8	0.63	0.60	
	C <sub>3</sub> -700-0.0	0.063	5.420	9	0.59		
	C <sub>1</sub> -700-1.0	0.056	2.100	9	0.23		
C-700-1.0	C <sub>2</sub> -700-1.0	0.059	3.280	11	0.29	0.28	
	C <sub>3</sub> -700-1.0	0.071	4.372	14	0.31		
	A <sub>1</sub> -800-0.1	0.042	3.410	6	0.56		
A-800-0.1	A <sub>2</sub> -800-0.1	0.058	4.292	8	0.53	0.53	
	A <sub>3</sub> -800-0.1	0.058	3.984	8	0.49		
	A <sub>1</sub> -800-0.5	0.07	3.645	14	0.26		
A-800-0.5	A <sub>2</sub> -800-0.5	0.066	4.583	12	0.38	0.31	
	A <sub>3</sub> -800-0.5	0.070	4.201	15	0.28		
	A <sub>1</sub> -800-1.0	0.063	2.369	12	0.19		
A-800-1.0	A <sub>2</sub> -800-1.0	0.061	3.483	13	0.26	0.22	
	A <sub>3</sub> -800-1.0	0.056	3.652	17	0.21		
	A <sub>1</sub> -800-1.5	0.053	3.879	13	0.29		
A-800-1.5	A <sub>2</sub> -800-1.5	0.085	4.013	12	0.33	0.31	
	A <sub>3</sub> -800-1.5	0.067	3.972	13	0.30		
	A <sub>1</sub> -800-2.0	0.057	3.981	12	0.33		
A-800-2.0	A <sub>2</sub> -800-2.0	0.088	5.133	15	0.34	0.33	

	A <sub>3</sub> -800-2.0	0.062	4.201	13	0.32	
	B <sub>1</sub> -800-0.1	0.045	2.849	5	0.56	
B-800-0.1	B <sub>2</sub> -800-0.1	0.076	4.934	9	0.54	0.55
	B <sub>3</sub> -800-0.1	0.066	4.465	8	0.55	
	B <sub>1</sub> -800-0.5	0.062	4.871	16	0.30	
B-800-0.5	B <sub>2</sub> -800-0.5	0.071	5.600	17	0.33	0.33
	B <sub>3</sub> -800-0.5	0.049	5.008	14	0.35	
	B <sub>1</sub> -800-1.0	0.052	2.781	10	0.27	
B-800-1.0	B <sub>2</sub> -800-1.0	0.045	2.599	9	0.28	0.27
	B <sub>3</sub> -800-1.0	0.064	4.112	16	0.25	
	B <sub>1</sub> -800-1.5	0.055	4.002	13	0.30	
B-800-1.5	B <sub>2</sub> -800-1.5	0.074	4.103	12	0.34	0.32
	B <sub>3</sub> -800-1.5	0.086	4.008	12	0.33	
	B <sub>1</sub> -800-2.0	0.061	4.042	12	0.33	
B-800-2.0	B <sub>2</sub> -800-2.0	0.083	4.231	11	0.38	0.35
	B <sub>3</sub> -800-2.0	0.093	4.005	12	0.33	
	C <sub>1</sub> -800-0.0	0.057	5.018	7	0.71	
C-800-0.0	C <sub>2</sub> -800-0.0	0.071	6.490	9	0.71	0.69
	C <sub>3</sub> -800-0.0	0.061	4.556	7	0.64	
	C <sub>1</sub> -800-1.0	0.074	2.213	12	0.18	
C-800-1.0	C <sub>2</sub> -800-1.0	0.055	3.404	13	0.26	0.22
	C <sub>3</sub> -800-1.0	0.076	3.407	16	0.21	



# APPENDIX L: STATISTICAL ANALYSIS OF DIFFERENCES IN QUALITY PARAMETERS BY THE MODES OF IMPREGNATION Table L-1: Difference in Activity of Samples Impregnated at 85°C and room temperature

		Levene's Equality of	s Test for f Variances			t-1	est for Equality of	of Means		
							Mean	Std Error	95% Confide the Dit	nce Interval of ference
		F	Sig.	t	df	Sig. (2-tailed)	Difference	Difference	Lower	Upper
600-0.1	Equal variances assumed	.232	.655	1. <b>8</b> 80	4	.133	1.10000	.58500	52421	2.72421
	Equal variances not assumed			1.880	3.881	.135	1.10000	.58500	54409	2.74409
600-0.5	Equal variances assumed	3.077	.154	.598	4	.582	.16667	.27889	60765	.94098
	Equal variances not assumed			.598	2.439	.601	.16667	.27889	84832	1.18165
600-1.0	Equal variances assumed	.182	.692	2.6 <mark>82</mark>	4	.055	1.73333	.64636	06124	3.52791
	Equal variances not assumed			2.682	3.818	.058	1.73333	.64636	09541	3.56208
700-0.1	Equal variances assumed	3.612	.130	.947	4	.397	1.16667	1.23198	-2.25386	4.58719
	Equal variances not assumed			.947	2.804	.418	1.16667	1.23198	-2.91353	5.24686
700-0.5	Equal variances assumed	3.69 <mark>3</mark>	.127	1.507	4	.206	1.76667	1.17237	-1.48835	5.02168
	Equal variances not assumed		20	1.507	2.748	.237	1.76667	1.17237	-2.16564	5.69897
700-1.0	Equal variances assumed	.532	.506	5.044	4	.007	4.30000	.85245	1.93323	6.66677
	Equal variances not assumed	(	~ 4	5.044	3.070	.014	4.30000	.85245	1.62171	6.97829
800-0.1	Equal variances assumed	.555	.498	1.235	4	.285	.83333	.67495	-1.04062	2.70729
	Equal variances not assumed	3		1.235	<mark>3.74</mark> 0	.289	.83333	.67495	-1.09304	2.75970
800-0.5	Equal variances assumed	.011	.921	2.075	4	.107	1.00000	.48189	33795	2.33795
	Equal variances not assumed		20	2.075	3.980	.107	1.00000	.48189	34068	2.34068
800-1.0	Equal variances assumed	1.078	.358	4.729	4	.009	4.20000	.88819	1.73398	6.66602
	Equal variances not assumed			4.729	3.353	.014	4.20000	.88819	1.53433	6.86567
800-1.5	Equal variances assumed	.000	1.000	25.277	4	.000	11.36667	.44969	10.11812	12.61521
	Equal variances not assumed			25.277	4.000	.000	11.36667	.44969	10.11812	12.61521
800-2.0	Equal variances assumed	.032	.866	37.745	4	.000	8.53333	.22608	7.90564	9.16103
	Equal variances not assumed			37.745	3.883	.000	8.53333	.22608	7.89808	9.16858

		Levene Equality o	s Test for If Variances				t-test for Equa	ality of Means		
								Std Error	95% Confiden Diff	ce Interval of the erence
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Difference	Lower	Upper
600-0.1	Equal variances assumed	1.853	.245	-1.184	4	.302	-1.42000	1.19957	-4.75054	1.91054
	Equal variances not assumed			-1.184	2.377	.341	-1.42000	1.19957	-5.87119	3.03119
600-0.5	Equal variances assumed	.446	.541	-2.231	4	.090	-1.63667	.73377	-3.67392	.40059
	Equal variances not assumed			-2.231	3. <mark>785</mark>	.093	-1.63667	.73377	-3.72048	.44715
600-1.0	Equal variances assumed	6.639	.062	410	4	.703	58667	1.42999	-4.55696	3.38363
	Equal variances not assumed			410	2.311	.717	58667	1.42999	-6.00935	4.83601
700-0.1	Equal variances assumed	1.112	.351	.495	40	.646	.72000	1.45424	-3.31763	4.75763
	Equal variances not assumed	Ç		.495	3.513	.650	.72000	1.45424	-3.54853	4.98853
700-0.5	Equal variances assumed	1.170	.340	.238	4	.824	.39333	1.65611	-4.20476	4.99142
	Equal variances not assumed		X	.238	3.272	.826	.39333	1.65611	-4.63795	5.42462
700-1.0	Equal variances assumed	.021	.892	.810	4	.463	1.76000	2.17246	-4.27172	7.79172
	Equal variances not assumed			.810	3.998	.463	1.76000	2.17246	-4.27307	7.79307
800-0.1	Equal variances assumed	.033	.865	474	4	.660	77667	1.63862	-5.32621	3.77287
	Equal variances not assumed		3	4 <mark>74</mark>	<mark>3.979</mark>	.660	77667	1.63862	-5.33572	3.78239
800-0.5	Equal variances assumed	.504	.517	3.064	4	.038	3.50333	1.14329	.32904	6.67763
	Equal variances not assumed		~	3.064	3.504	.045	3.50333	1.14329	.14390	6.86276
800-1.0	Equal variances assumed	9.391	.037	2.243	4	.088	3.85333	1.71819	91713	8.62379
	Equal variances not assumed			2.243	2.176	.144	3.85333	1.71819	-2.99370	10.70037
800-1.5	Equal variances assumed	.154	.715	1.150	4	.314	.96333	.83772	-1.36256	3.28922
	Equal variances not assumed			1.150	3.915	.316	.96333	.83772	-1.38256	3.30923
800-2.0	Equal variances assumed	.143	.725	2.604	4	.060	1.18000	.45314	07811	2.43811
	Equal variances not assumed			2.604	3.935	.061	1.18000	.45314	08640	2.44640

Table L -2: Difference in Attrition of Samples Impregnated at 85°C and room temperature

		Levene's Equality of V	Test for /ariances	r es t-test for Equality of Means							
						Sig (2-		Std Error	95% Confidence Differe	Interval of the nce	
		F	Sig.	t	df	tailed)	Mean Difference	Difference	Lower	Upper	
600-0.1	Equal variances assumed	1.495	.289	465	4	.666	58333	1.25540	-4.06887	2.90221	
	Equal variances not assumed			465	3.375	.671	58333	1.25540	-4.33897	3.17230	
600-0.5	Equal variances assumed	.021	.892	.107	4	.920	.18000	1.68108	-4.48742	4.84742	
	Equal variances not assumed			.107	<b>3</b> .905	.920	.18000	1.68108	-4.53274	4.89274	
600-1.0	Equal variances assumed	11.245	.028	-1.27 <mark>6</mark>	4	.271	-1.60000	1.25408	-5.08189	1.88189	
	Equal variances not assumed			-1.276	2.054	.327	-1.60000	1.25408	-6.86286	3.66286	
700-0.1	Equal variances assumed	4.512	.101	-2.536	4	.064	-2.57667	1.01592	-5.39731	.24397	
	Equal variances not assumed		-	-2.536	2.500	.102	-2.57667	1.01592	-6.20809	1.05476	
700-0.5	Equal variances assumed	1.2 <mark>36</mark>	.329	1.546	4	.197	1.64333	1.06275	-1.30734	4.59401	
	Equal variances not assumed		R	1.546	3.177	.215	1.64333	1.06275	-1.63489	4.92156	
700-1.0	Equal variances assumed	2.592	.183	2.122	4	.101	1.70000	.80114	52432	3.92432	
	Equal variances not assumed	(		2.122	2.557	.139	1.70000	.80114	-1.11838	4.51838	
800-0.1	Equal variances assumed	.400	.561	.602	4	.579	.41667	.69160	-1.50352	2.33686	
	Equal variances not assumed	3		.602	3.757	.581	.41667	.69160	-1.55357	2.38690	
800-0.5	Equal variances assumed	2.042	.226	1.036	4	.359	1.76667	1.70447	-2.96570	6.49903	
	Equal variances not assumed		2	1.036	3.164	.373	1.76667	1.70447	-3.50237	7.03571	
800-1.0	Equal variances assumed	3.884	.120	086	4	.936	12667	1.48067	-4.23766	3.98433	
	Equal variances not assumed			086	2.517	.938	12667	1.48067	-5.39449	5.14116	
800-1.5	Equal variances assumed	.132	.735	2.625	4	.058	1.23000	.46853	07085	2.53085	
	Equal variances not assumed			2.625	3.969	.059	1.23000	.46853	07491	2.53491	
800-2.0	Equal variances assumed	9.379	.055	1.427	4	.249	1.41333	.99046	-1.73876	4.56543	
	Equal variances not assumed			1.178	1.261	.415	1.41333	1.19995	-8.06800	10.89467	

 Table L-3: Difference in Particle Size Retention of Samples Impregnated at 85°C and room temperature (r.t)

	-	Levene's T Equality of V	est for ariances	t-test for Equality of Means						
						Sig.	Mean	Std. Error	95% Confider the Dif	nce Interval of ference
		F	Sig.	t	df	(2-tailed)	Difference	Difference	Lower	Upper
600-0.1	Equal variances assumed	3.155	.150	-1.382	4	.239	43333	.31356	-1.30393	.43726
	Equal variances not assumed			-1.382	2.779	.268	43333	.31356	-1.47763	.61096
600-0.5	Equal variances assumed	5.878	.072	.559	4	.606	.14000	.25051	55553	.83553
	Equal variances not assumed			.559	2.472	.623	.14000	.25051	76273	1.04273
600-1.0	Equal variances assumed	1.475	.291	139	4	.896	05667	.40855	-1.19098	1.07764
	Equal variances not assumed			<mark>139</mark>	3.038	.898	05667	.40855	-1.34781	1.23448
700-0.1	Equal variances assumed	1.798	.251	.161	4	.880	.01333	.08273	21637	.24303
	Equal variances not assumed			.161	2.481	.884	.01333	.08273	28403	.31070
700-0.5	Equal variances assumed	.214	.668	-3.974	4	.016	50333	.12667	85502	15165
	Equal variances not assumed			-3.974	3.763	.019	50333	.12667	86391	14276
700-1.0	Equal variances assumed	11.979	.026	061	4	.954	01333	.21861	62028	.59362
	Equal variances not assumed			061	2.046	.957	01333	.21861	93391	.90724
800-0.1	Equal variances assumed	1.808	.250	.089	4	.933	.01333	.14996	40303	.42970
	Equal variances not assumed			.089	3.288	.934	.01333	.14996	44113	.46780
800-0.5	Equal variances assumed	.558	.497	145	4	.892	06667	.45904	-1.34118	1.20785
	Equal variances not assumed	1 Alexandre	SIL	145	3.739	.892	06667	.45904	-1.37708	1.24375
800-1.0	Equal variances assumed	.032	.866	.980	4	.383	.11333	.11566	20780	.43446
	Equal variances not assumed			.980	3.914	.384	.11333	.11566	21058	.43725
800-1.5	Equal variances assumed	6.229	.067	502	4	.642	09333	.18586	60937	.42270
	Equal variances not assumed			502	2.350	.659	09333	.18586	78905	.60238
800-2.0	Equal variances assumed	.086	.784	.060	4	.955	.01000	.16620	45144	.47144
	Equal variances not assumed			.060	3.804	.955	.01000	.16620	46096	.48096

# Table L-4: Difference in Moisture of Samples Impregnated at 85°C and room temperature

		Levene's Test fo of Varian	Levene's Test for Equality of Variances		t-test for Equality of Means							
						Sig	Moon	Std Error	95% Confide the Dif	nce Interval of ference		
		F	Sig.	t	df	(2-tailed)	Difference	Difference	Lower	Upper		
600-0.1	Equal variances assumed	3.903	.119	.249	4	.816	.02667	.10729	27122	.32455		
	Equal variances not assumed			.249	2.558	.822	.02667	.10729	35074	.40408		
600-0.5	Equal variances assumed	.002	.963	427	4	.692	05333	.12499	40036	.29369		
	Equal variances not assumed			427	4.000	.692	05333	.12499	40036	.29369		
600-1.0	Equal variances assumed	.484	.525	755	4	.492	06667	.08825	31170	.17837		
Equal variances not assumed			755	3.328	.500	06667	.08825	33250	.19917			
700-0.1	Equal variances assumed	.365	.578	-1.139	4	.318	16000	.14044	54991	.22991		
	Equal variances not assumed			-1.139	3.785	.322	16000	.14044	55880	.23880		
700-0.5	Equal variances assumed	8.345	.045	901	4	.418	13000	.14422	53042	.27042		
	Equal variances not assumed		9	901	2.181	.456	13000	.14422	70381	.44381		
700-1.0	Equal variances assumed	3.724	.126	947	4	.397	27333	.28852	-1.07440	.52773		
	Equal variances not assumed			947	2.484	.426	27333	.28852	-1.30957	.76290		
800-0.1	Equal variances assumed	3.261	.145	-1.276	4	.271	16333	.12798	51865	.19198		
	Equal variances not assumed	E		-1 <mark>.276</mark>	2.058	.327	16333	.12798	69941	.37274		
800-0.5	Equal variances assumed	2.074	.223	638	4	.558	08333	.13068	44616	.27950		
	Equal variances not assumed		405	638	2.825	.572	08333	.13068	51414	.34748		
800-1.0	Equal variances assumed	4.341	.106	028	NE 410	.979	00333	.11921	33431	.32765		
	Equal variances not assumed			028	2.572	.980	00333	.11921	42105	.41438		
800-1.5	Equal variances assumed	1.178	.339	1.150	4	.314	.06000	.05217	08486	.20486		
	Equal variances not assumed			1.150	3.439	.324	.06000	.05217	09468	.21468		
800-2.0	Equal variances assumed	1.729	.259	5.846	4	.004	.25333	.04333	.13302	.37365		
	Equal variances not assumed			5.846	2.556	.015	.25333	.04333	.10085	.40582		

# Table L-5: Difference in Ash of Samples Impregnated at 85°C and room temperature

		Levene's Tes of Vari	Levene's Test for Equality of Variances		t-test for Equality of Means							
						Sia.	Mean	Std. Error	95% Confide the Dit	ence Interval of fference		
		F	Sig.	t	df	(2-tailed)	Difference	Difference	Lower	Upper		
600-0.1	Equal variances assumed	3.213	.148	898	4	.420	01667	.01856	06820	.03486		
	Equal variances not assumed			898	2.424	.449	01667	.01856	08453	.05119		
600-0.5	Equal variances assumed	7.330	.054	-3.010	4	.040	12000	.03986	23067	00933		
	Equal variances not assumed			-3.010	2.115	.089	12000	.03986	28287	.04287		
600-1.0	Equal variances assumed	3.077	.154	-1.31 <mark>5</mark>	4	.259	03667	.02789	11410	.04076		
	Equal variances not assumed			-1.315	2.439	.299	03667	.02789	13816	.06483		
700-0.1	Equal variances assumed	.000	1.000	.000	4	1.000	.00000	.01247	03463	.03463		
	Equal variances not assumed			.000	4.000	1.000	.00000	.01247	03463	.03463		
700-0.5	Equal variances assumed	.213	.668	731	4	.505	04667	.06386	22396	.13063		
	Equal variances not assumed			731	3.912	.506	04667	.06386	22554	.13221		
700-1.0	Equal variances assumed	.041	.849	.105	- 4	.922	.00667	.06368	17015	.18348		
	Equal variances not assumed		( FT	.105	3.978	.922	.00667	.06368	17053	.18386		
800-0.1	Equal variances assumed	2.560	.185	-1.107	4	.330	02333	.02108	08187	.03520		
	Equal variances not assumed	17		-1.107	2.322	.370	02333	.02108	10299	.05632		
800-0.5	Equal variances assumed	4.105	.113	502	4	.642	02000	.03986	13067	.09067		
	Equal variances not assumed		APS	502	2.599	.655	02000	.03986	15869	.11869		
800-1.0	Equal variances assumed	2.579	.184	-2.064	4	.108	04667	.02261	10944	.01610		
	Equal variances not assumed			-2.064	2.696	.141	04667	.02261	12344	.03010		
800-1.5	Equal variances assumed	.000	1.000	981	4	.382	01667	.01700	06386	.03052		
	Equal variances not assumed			981	4.000	.382	01667	.01700	06386	.03052		
800-2.0	Equal variances assumed	5.765	.074	945	4	.398	01667	.01764	06564	.03231		
	Equal variances not assumed			945	2.473	.428	01667	.01764	08021	.04688		

# Table L-6: Difference in Apparent Density of Samples Impregnated at 85°C and room temperature

#### APPENDIX M: STATISTICAL ANALYSIS OF RESULTS AS PER GOLD MINING

# Table M- 1: Activity Analysis

		Test Value = 55										
					95% Confiden Diffe	ce Interval of the erence						
	Т	df	Sig. (2-tailed)	Mean Difference	Lower	Upper						
A-600-0.1	-106.960	2	.000	-47.96667	-49.8962	-46.0371						
A-600-0.5	-178.777	2	.000	-47.30000	-48.4384	-46.1616						
A-600-1.0	-107.387	2	.000	-43.40000	-45.1389	-41.6611						
B-600-0.1	-130.619	2	.000	-49.06667	-50.6829	-47.4504						
B-600-0.5	-538.221	2	.000	-47.46667	-47.8461	-47.0872						
B-600-1.0	-89.475	2	.000	-45.13333	-47.3037	-42.9630						
C-600-0.0	-120.626	2	.000	-52.73333	-54.6143	-50.8524						
C-600-1.0	-247.945	2	.000	-43.73333	-44.4922	-42.9744						
A-700-0.1	-14.672	2	.005	-16.43333	-21.2524	-11.6143						
A-700-0.5	16.839	2	.004	18.06667	13.4504	22.6830						
A-700-1.0	71.509	2	.000	28.90000	27.16 <mark>1</mark> 1	30.6389						
B-700-0.1	-34.2 <mark>97</mark>	2	.001	-17.60000	<mark>-19</mark> .8079	-15.3921						
B-700-0.5	34.491	2	.001	16.30000	14.2666	18.3334						
B-700-1.0	32.776	2	.001	24.60000	21.3706	27.8294						
C-700-0.0	-23.229	2	.002	-26.86667	-31.8432	-21.8901						
C-700-1.0	53.463	2	.000	24.50000	22.5283	26.4717						
A-800-0.1	32 <mark>.145</mark>	2	.001	13.16667	<mark>11.4</mark> 043	14.9291						
A-800-0.5	81. <mark>126</mark>	2	.000	26.63333	<mark>25</mark> .2208	28.0459						
A-800-1.0	48.661	2	.000	36.6 <mark>66</mark> 7	33.4246	39.9088						
A-800-1.5	91.620	2	.000	29.13333	27.7652	30.5015						
A-800-2.0	102.778	2	.000	14.93333	14.3082	15.5585						
B-800-0.1	22.991	2	.002	12.33333	10.0252	14.6415						
B-800-0.5	219.104	2	.000	25.30000	24.8032	25.7968						
B-800-1.0	69.045	2	.000	32.46667	30.4435	34.4899						
B-800-1.5	55.874	2	.000	17.76667	16.3985	19.1348						
B-800-2.0	36.950	2	.001	6.40000	5.6548	7.1452						
C-800-0.0	-68.390	2	.000	-13.86667	-14.7391	-12.9943						
C-800-1.0	23.838	2	.002	33.83333	27.7265	39.9401						

		Test Value = 7.80									
			-		95% Confidenc	e Interval of the					
					Diffe	rence					
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper					
A-600-0.1	-8.318	2	.014	-2.94000	-4.4608	-1.4192					
A-600-0.5	7.845	2	.016	4.53000	2.0455	7.0145					
A-600-1.0	7.386	2	.018	10.82000	4.5165	17.1235					
B-600-0.1	-1.326	2	.316	-1.52000	-6.4522	3.4122					
B-600-0.5	13.621	2	.005	6.16667	4.2187	8.1147					
B-600-1.0	30.111	2	.001	11.60667	9.9482	13.2652					
C-600-0.0	-100.669	2	.000	-3.73667	-3.8964	-3.5770					
C-600-1.0	20.356	2	.002	12.82000	10.1102	15.5298					
A-700-0.1	4.231	2	.052	3.44667	0580	6.9514					
A-700-0.5	12.310	2	.007	10.47667	6.8147	14.1386					
A-700-1.0	11.038	2	.008	17.15667	10.4688	23.8446					
B-700-0.1	2.263	2	.152	2.72667	-2.4568	7.9101					
B-700-0.5	7.098	2	.019	10.08333	<b>3.9706</b>	16.1960					
B-700-1.0	10.144	2	.010	15.39667	8.8663	21.9270					
C-700-0.0	.216	2	.849	.19000	-3.5993	3.9793					
C-700-1.0	19.458	2	.003	19.22667	14.9751	23.4783					
A-800-0.1	11.032	2	.008	13.24000	8.0764	18.4036					
A-800-0.5	38.894	2	.001	24.83667	22.0891	27.5842					
A-800-1.0	1 <mark>9.757</mark>	2	.003	33.22000	25.9854	40.4546					
A-800-1.5	66.693	2	.000	36.48333	34.1296	38.8370					
A-800-2.0	132.551	2	.000	39.64000	38.3533	40.9267					
B-800-0.1	12.563	2	.006	14.01667	9.2161	18.8172					
B-800-0.5	22.496	2	.002	21.33333	17.2530	25.4137					
B-800-1.0	83.069	2	.000	29.36667	27.8456	30.8877					
B-800-1.5	55.985	2	.000	35.52000	32.7902	38.2498					
B-800-2.0	112.971	2	.000	38.46000	36.9952	39.9248					
C-800-0.0	5.174	2	.035	5.27000	.8871	9.6529					
C-800-1.0	89.616	2	.000	32.76000	31.1871	34.3329					

 Table M-2: Attrition% Analysis

	Test Value = 90									
					95% Confidence	e Interval of the				
					Differ	ence				
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper				
A-600-0.1	-12.429	2	.006	-8.32667	-11.2092	-5.4442				
A-600-0.5	-4.595	2	.044	-5.01667	-9.7145	3188				
A-600-1.0	-1.081	2	.393	-1.34667	-6.7067	4.0133				
B-600-0.1	-7.293	2	.018	-7.74333	-12.3115	-3.1752				
B-600-0.5	-5.080	2	.037	-5.46333	-10.0905	8361				
B-600-1.0	1.755	2	.221	.25333	3679	.8745				
C-600-0.0	-109.358	2	.000	-31.02333	-32.2439	-29.8027				
C-600-1.0	-2.254	2	.1 <mark>53</mark>	-4.13667	-12.0330	3.7596				
A-700-0.1	-38.533	2	.001	-13.14333	-14.6109	-11.6757				
A-700-0.5	-13.612	2	.005	-7.16667	-9.4320	-4.9013				
A-700-1.0	-15.474	2	.004	-4.37333	-5.5894	-3.1573				
B-700-0.1	-11.042	2	.008	-10.56667	-14.6841	-6.4493				
B-700-0.5	-9.543	2	.011	-8.81000	<mark>-12.78</mark> 21	-4.8379				
B-700-1.0	-8.102	2	.015	-6.07333	-9.2987	-2.8479				
C-700-0.0	-47.573	2	.000	-34.88333	-38.0383	-31.7283				
C-700-1.0	-3.776	2	.064	-7.23000	-15.4678	1.0078				
A-800-0.1	-46.536	2	.000	-19.65000	-21.4668	-17.8332				
A-800-0.5	-7.514	2	.017	-11.14333	-17.5244	-4.7622				
A-800-1.0	- <mark>7.361</mark>	2	.018	-10.24667	- <mark>16</mark> .2361	-4.2572				
A-800-1.5	-17.5 <mark>92</mark>	2	.003	-5.56333	-6.9240	-4.2026				
A-800-2.0	.528	2	.650	.21333	-1.5237	1.9503				
B-800-0.1	-36.636	2	.001	-20.06667	-22.4234	-17.7100				
B-800-0.5	-15.368	2	.004	-12.91000	-16.5246	-9.2954				
B-800-1.0	-20.055	2	.002	-10.12000	-12.2912	-7.9488				
B-800-1.5	-19.651	2	.003	-6.79333	-8.2808	-5.3059				
B-800-2.0	-1.869	2	.203	-1.22000	-4.0284	1.5884				
C-800-0.0	-28.886	2	.001	-35.46667	-40.7496	-30.1838				
C-800-1.0	-7.452	2	.018	-12.28000	-19.3707	-5.1893				

#### **Table M-3: Particle Size Retention**

	Test Value = 4										
					95% Confidence	e Interval of the					
					Differ	ence					
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper					
A-600-0.1	-13.721	2	.005	-1.76667	-2.3207	-1.2127					
A-600-0.5	-6.097	2	.026	-1.44333	-2.4619	4247					
A-600-1.0	-7.993	2	.015	-1.52667	-2.3484	7049					
B-600-0.1	-4.663	2	.043	-1.33333	-2.5635	1032					
B-600-0.5	-19.327	2	.003	-1.58333	-1.9358	-1.2309					
B-600-1.0	-4.070	2	.055	-1.47000	-3.0239	.0839					
C-600-0.0	-12.007	2	.007	-1.19333	-1.6210	7657					
C-600-1.0	-12.366	2	.006	-1.59000	-2.1432	-1.0368					
A-700-0.1	-20.870	2	.002	-1.63000	-1.9660	-1.2940					
A-700-0.5	-21.067	2	.002	-1.63333	-1.9669	-1.2997					
A-700-1.0	-56.000	2	.000	-1.30667	-1.4071	-1.2063					
B-700-0.1	-60.230	2	.000	-1.64333	-1.7607	-1.5259					
B-700-0.5	-11.281	2	.008	-1.13000	<mark>-1.5</mark> 610	6990					
B-700-1.0	-5.950	2	.027	-1.29333	-2.2285	3581					
C-700-0.0	-5.202	2	.035	-1.16333	-2.1255	2011					
C-700-1.0	-15.455	2	.004	-1.38333	-1.7684	9982					
A-800-0.1	-9.608	2	.011	-1.23333	-1.7856	6810					
A-800-0.5	-4.503	2	.046	-1.64333	-3.2137	0729					
A-800-1.0	-1 <mark>8.146</mark>	2	.003	-1.37000	-1.6948	-1.0452					
A-800-1.5	-32.505	2	.001	-1.72000	-1.9477	-1.4923					
A-800-2.0	-14.871	2	.004	-1.5 <mark>3667</mark>	-1.9813	-1.0921					
B-800-0.1	-16.080	2	.004	-1.24667	-1.5803	9131					
B-800-0.5	-5.663	2	.030	-1.57667	-2.7746	3788					
B-800-1.0	-16.929	2	.003	-1.48333	-1.8603	-1.1063					
B-800-1.5	-9.130	2	.012	-1.62667	-2.3933	8601					
B-800-2.0	-11.882	2	.007	-1.54667	-2.1067	9866					
C-800-0.0	-4.339	2	.049	-1.12333	-2.2371	0095					
C-800-1.0	-25.516	2	.002	-1.84000	-2.1503	-1.5297					

 Table M-4: Moisture Content Analysis

		Test Value = 4										
					95% Confidence	e Interval of the						
					Differ	ence						
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper						
A-600-0.1	-26.942	2	.001	-1.02000	-1.1829	8571						
A-600-0.5	-6.072	2	.026	53667	9169	1564						
A-600-1.0	-1.368	2	.305	06333	2626	.1359						
B-600-0.1	-10.426	2	.009	-1.04667	-1.4786	6147						
B-600-0.5	-5.469	2	.032	48333	8636	1031						
B-600-1.0	.044	2	.969	.00333	3199	.3266						
C-60-00.0	-6.067	2	.026	-1.23667	-2.1137	3597						
C-600-1.0	4.000	2	.057	.12000	0091	.2491						
A-700-0.1	-7.192	2	.019	62333	9962	2504						
A-700-0.5	142	2	.900	02000	6270	.5870						
A-700-1.0	6.919	2	.020	.66000	.2496	1.0704						
B-700-0.1	-4.193	2	.052	46333	9388	.0121						
B-700-0.5	3.667	2	.067	.11000	0191	.2391						
B-700-1.0	3.428	2	.076	.93333	2383	2.1049						
C-700-0.0	-8.046	2	.015	75667	-1.1613	3520						
C-700-1.0	2.421	2	.137	.91000	7074	2.5274						
A-800-0.1	24.877	2	.002	.38000	.3143	.4457						
A-800-0.5	19.065	2	.003	1.05000	.8130	1.2870						
A-800-1.0	1 <mark>6.164</mark>	2	.004	1.80000	1.3209	2.2791						
A-800-1.5	79.822	2	.000	2.27333	2.1508	2.3959						
A-800-2.0	170.865	2	.000	2.61000	2.5443	2.6757						
B-800-0.1	4.276	2	.051	.54333	0034	1.0900						
B-800-0.5	9.563	2	.011	1.13333	.6234	1.6432						
B-800-1.0	42.374	2	.001	1.80333	1.6202	1.9864						
B-800-1.5	50.630	2	.000	2.21333	2.0252	2.4014						
B-800-2.0	125.365	2	.000	2.32667	2.2468	2.4065						
C-800-0.0	.832	2	.493	.09333	3892	.5759						
C-800-1.0	15.629	2	.004	1.82333	1.3214	2.3253						

# Table M-5: Ash Content Analysis

# Table M-6: Apparent Density

	Test Value = 0.49										
					95% Confidenc	e Interval of the					
					Diffe	rence					
	t	df	Sig. (2-tailed)	Mean Difference	Lower	Upper					
A-600-0.1	5.196	2	.035	.03000	.0052	.0548					
A-60005	-26.000	2	.001	17333	2020	1446					
A-600-1.0	-17.764	2	.003	15667	1946	1187					
B-600-0.1	2.646	2	.118	.04667	0292	.1226					
B-600-0.5	-1.357	2	.308	05333	2224	.1158					
B-600-1.0	-4.536	2	.045	12000	2338	0062					
C-600-0.0	1.692	2	.233	.07333	1131	.2598					
C-600-1.0	-9.700	2	.010	19667	2839	1094					
A-700-0.1	9.827	2	.010	.08667	.0487	.1246					
A-70005	-2.823	2	.106	13667	3450	.0717					
A-700-1.0	-3.846	2	.061	16667	3531	.0198					
B-700-0.1	9.827	2	.010	.08667	.0487	.1246					
B-700-0.5	-2. <mark>162</mark>	2	.163	09000	2691	.0891					
B-700-1.0	-3.714	2	.065	17333	3741	.0275					
C-700-0.0	6.047	2	.026	.10667	.0308	.1826					
C-700-1.0	-8.875	2	.012	21333	3168	1099					
A-800-0.1	1.808	2	.212	.03667	0506	.1239					
A-800-0.5	- <mark>4.939</mark>	2	.039	18333	3430	0236					
A-800-1.0	-12. <mark>970</mark>	2	.006	27000	3596	1804					
A-800-1.5	-13.000	2	.006	173 <mark>33</mark>	2307	1160					
A-800-2.0	-27.713	2	.001	16000	1848	1352					
B-800-0.1	10.392	2	.009	.06000	.0352	.0848					
B-800-0.5	-11.241	2	.008	16333	2258	1008					
B-800-1.0	-25.324	2	.002	22333	2613	1854					
B-800-1.5	-13.868	2	.005	16667	2184	1150					
B-800-2.0	-8.600	2	.013	14333	2150	0716					
C-800-0.0	8.429	2	.014	.19667	.0963	.2971					
C-800-1.0	-11.714	2	.007	27333	3737	1729					

#### **APPENDIX N: LIST OF PHOTOGRAPHS**



Figure A.1: Precursor – PKS



Figure A.3: Potash Leaching



Figure A.2: Proximate Analysis – V.M.



Figure A.4: Alkali Analysis



Figure A.5: Impregnation – soaking



Figure A.6: Impregnation – drying



Figure A.7: Oven used for Drying



Figure A.9: Ash Content Determination



Figure A.8: Sample GAC Produced



Figure A.10: Attrition Determination



Figure A.11: Activity Test – Bottle Rolling



Figure A.12: AAS Machine for Activity