

**KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY
COLLEGE OF SCIENCE DEPARTMENT OF ENVIRONMENTAL
SCIENCE**

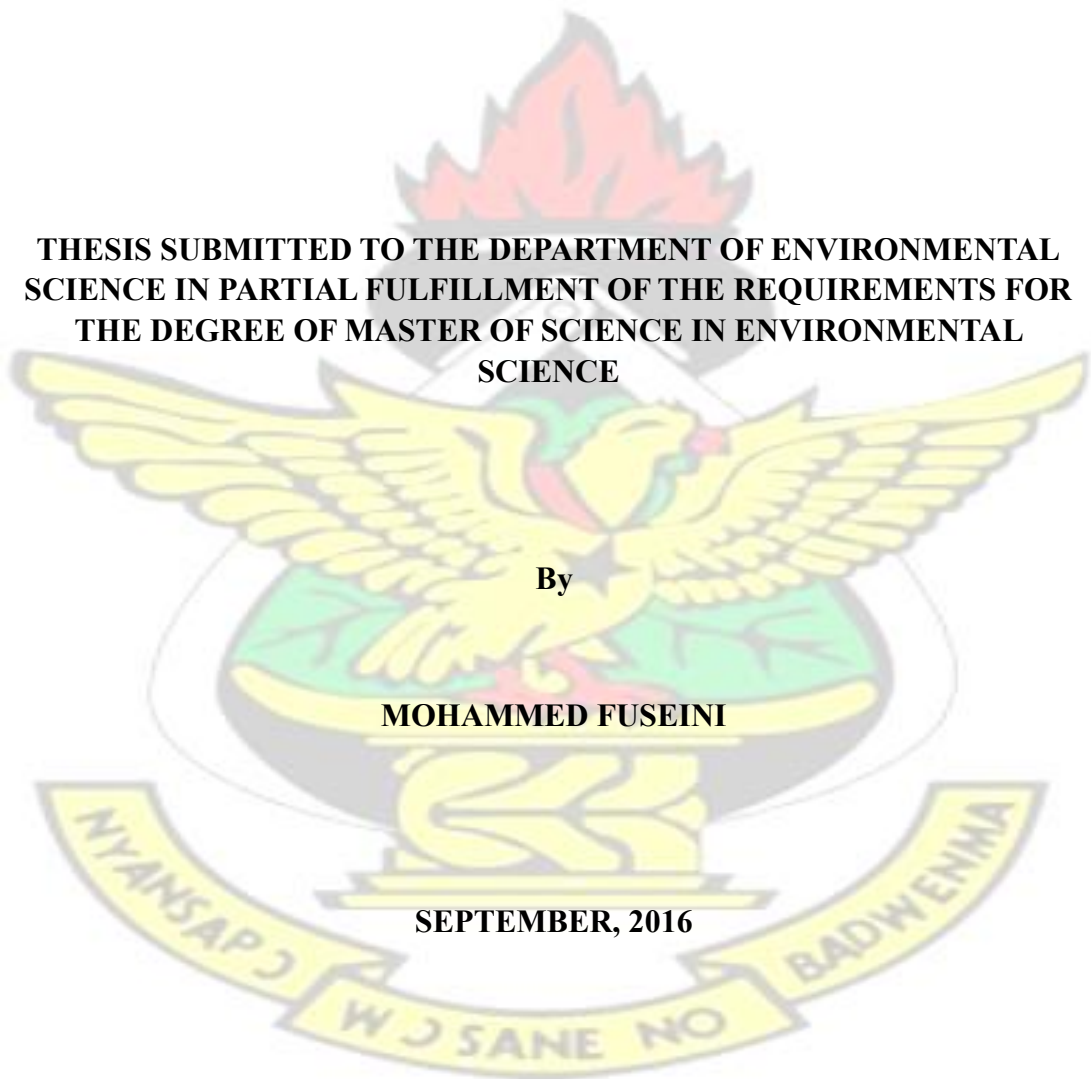
**LABORATORY BASED EXPERIMENT TO DECONTAMINATE ARSENIC
AND LEAD LADEN WATER USING PALM KERNEL AND SHEA NUT
CAKES**

**THESIS SUBMITTED TO THE DEPARTMENT OF ENVIRONMENTAL
SCIENCE IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF MASTER OF SCIENCE IN ENVIRONMENTAL
SCIENCE**

By

MOHAMMED FUSEINI

SEPTEMBER, 2016



DECLARATION

I hereby declare that this thesis is the result of my own work towards the award of M.Sc and has been composed under supervision. It has not been submitted previously either wholly or partially for a degree in Kwame Nkrumah University of Science and Technology or elsewhere except where due acknowledgement has been made in the text.

Mohammed Fuseini

.....

.....

(Student)

Signature

Date

Certified by

Dr. B. Fei Baffoe

.....

.....

(Supervisor)

Signature

Date

Certified by

Dr. M.G. Addo

.....

.....

(Head of Department)

Signature

Date

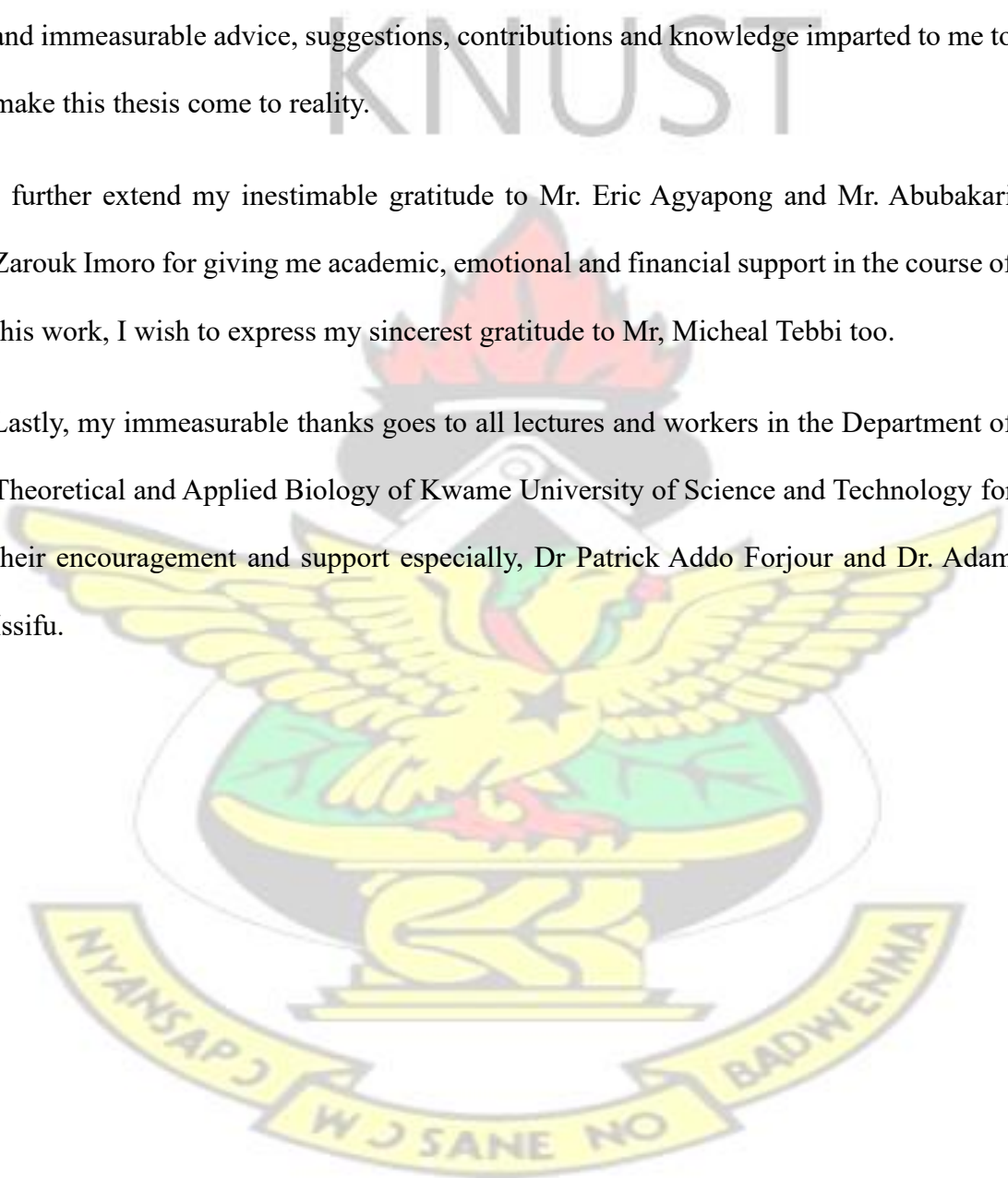
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I am also greatly indebted to my supervisor Dr. Bernard Fei-Baffoe for his invaluable and immeasurable advice, suggestions, contributions and knowledge imparted to me to make this thesis come to reality.

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Lastly, my immeasurable thanks goes to all lectures and workers in the Department of Theoretical and Applied Biology of Kwame University of Science and Technology for their encouragement and support especially, Dr Patrick Addo Forjour and Dr. Adam Issifu.



DEDICATION

I dedicate this work to the almighty Allah the omnipotent, Ghanaians who fought against illegal mining (Galamsey), my family especially four most important women in my life namely, my lovely daughter, Jamila Mohammed Kada, My sweet mother Rakiatu Abubakar, my beloved wife Shafaatu Alhassan Daud , my mother- in law Hajia Jamila Mohammed s Adago.



ABSTRACT

The adsorption efficiencies of Palm Kernel Cake (PKC) and Shea Nut Cake (SNC) in adsorbing arsenic and lead laden water were studied in the laboratory (KNUST and Anglo Gold Ashanti). Sorption capacities of PKC and SNC were tested at varying concentrations of As and Pb and also, at varying masses of the adsorbents. Results of the study show that sorption efficiencies of PKC and SNC depended on the mass of the adsorbent material and as well on the concentration of the metal ions (As and Pb) in solution. With fixed mass of adsorbents, maximum sorption efficiencies of 55.20% and 71.40% were obtained with PKC and SNC respectively at varying concentrations of arsenic solution wherein efficiencies increased with increasing concentration of the ion. However, sorption efficiencies decreased with increasing concentrations of lead in the contaminated water. Maximum efficiencies of 88.8% and 98% were achieved by PKC and SNC respectively. With varying masses of adsorbents in 100 mg/l solution of arsenic, maximum sorption efficiencies of 62.7% and 74% were achieved with PKC and SNC respectively. Sorption efficiency increased with mass of SNC whereas it decreased with that of PKC. However, sorption efficiency increased with masses of both PKC and SNC when 100 mg/l solution of Pb was prepared. At varying concentrations of lead solution, only PKC fitted the Lagmuir model ($R^2 = 0.92$). Whereas, only SNC fitted the model when its masses were varied for adsorption of As and Pb

($R^2 = 0.91$ and $R^2 = 0.99$ respectively). For Freundlich's isotherm, the following fitted the model. Adsorption by PKC at varying concentrations of As solution ($R^2 = 0.99$), SNC at varying concentration of Pb solution ($R^2 = 0.92$), adsorption of As at varying masses of SNC ($R^2 = 0.98$) and adsorption of Pb at varying masses of SNC ($R^2 = 0.98$). Analysis of variance and percentages were computed using Graphpad prism software. Generally, it can be concluded that both Shea Nut Cake and Palm Kernel Cake are good adsorbents and can be used to effectively clean-up arsenic and lead contaminated water.

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CHAPTER ONE

INTRODUCTION Background of the study

Mining in Ghana contributes 5% of the country's Gross Domestic Product (www.ghanachamberofmines.com). In terms of country total exports, 37% is minerals with gold making up over 90% of the total mineral exports. Ghana alone produced 80.5 tonnes of Gold in 2008 (www.ghanachamberofmines.com). Bauxite, manganese and diamond are other minerals that the country is endowed with.

Despite the economic benefits of minerals to the country, the operations of the mining industry have brought the nation a number of negative environmental and health implications, especially in the areas of heavy metal pollution of fresh water bodies. Heavy metals are significant pollutants in ocean waters, underground, industry and interestingly treated wastewaters (Valdman *et al.*, 2001). Pollution of water bodies by heavy metals is a global challenge because of their fatal nature and build ups in food chains (Kapoor *et al.*, 1999). The presence of heavy metals in potable water can be very dangerous to users as these elements can destroy nerves, liver, bones and inhibit functional groups of essential enzymes (Ewan & Pamphlet, 1996).

In the past few years, education on water pollution and its deleterious effects has awakened efforts towards pollution abatement (Dönmez *et al.*, 1999). Albert SzentGyorgyi, the Hungarian Nobel Prize winner is reported to have said, "Water is life's master and matrix, mother and medium. Consequently, there isn't life in the absence of water." He explained that contamination of water resources will mean contaminating life as well.

Making available potable water for the ‘exploding’ population of the world is amongst the great challenges facing the world. Water quality is affected in some cases by natural processes while in many by anthropogenic activities (Asklund & Eldvall, 2005).

The health implications of heavy metal contamination of drinking water sources in mining areas of the country are alarming. According to the commission on Human Rights and Administrative Justice (CHRAJ), (2008) report, water bodies in mining areas are contaminated which makes the populace in these communities vulnerable to water related illness thus an infringement on their human rights.

Arsenic for instance is a class ‘A’ human cancer causative agent (USEPA), which according to CHRAJ (2008) is present in the communities studied. The study revealed that, skin diseases increased from 1,634 to 3,825 in 2004 and further to 4066 in 2006, and this happening was associated to arsenic contamination.

Lead has also been identified amongst the highest pollutant in Ghana especially in and around the scrap yards (www.TV3net.com). There are a number of methods for the containment and decontamination of heavy metal polluted water. These include chemical stabilization, adsorption, physical stabilization and biological remediation. However, adsorption is considered amongst the most cost-effective method, consequently used mostly in decontaminating effluents (Balkose & Baltacioglu, 1992).

Agricultural waste has proven to be a good adsorbent in the treatment of heavy metal polluted waters. Examples of such agricultural waste that have been used as adsorbents are soap seeds (Oboh & Alluyor, 2008), and maize leaf and pomegranate peels (Babarinde & Oyidipe, 2006).

The applicability of these materials as adsorbents is based on factors such as large surface area and effective binding sites. Shea nut and palm kernel nut cakes are thought to possess these properties. Hence, the application of these materials in this study for the treatment of mine processed wastewater.

1.2 Problem Statement

Ghana is blessed with a variety of mineral deposits including gold, bauxite, diamond and manganese. The mining and processing of these minerals, both by large companies and legal and illegal (galamsey) operators can be dated back to the colonial era and has brought the nation a lot of economic benefits. However, the extraction and processing of these minerals have equally brought the nation some environmental concerns. Some of these environmental problems that have resulted from mining activities include contamination of water bodies, destruction of forests and other ecosystems and loss of irrigation water sources due to the release of heavy metals from the Earth's crust by mining activities. Many mining areas in the country are documented to often suffer contamination of potable water sources due to the release of heavy metals by mining activities (Akordor, 2012).

There are a number of methods which can be applied for the containment and treatment of heavy metal contaminated media. These include chemical stabilization, physical stabilization and biological remediation. However, adsorption is considered amongst the most cost-effective method, consequently used mostly in decontaminating effluents (Balkose & Baltacioglu, 1992). Agricultural waste has proven to be a good adsorbent in the treatment of heavy metal polluted waters. In this study, shea nut cake and palm kernel cake are used as adsorbents to remediate Arsenic and Lead contaminated wastewater.

1.3 Justification of the Study

Examining the current state of environmental degradation, coupled with effects on human health and economic activities in mining communities, there is the need to find an effective method for the treatment of mine wastewaters. Though certain measures including discharge into tailing dams, stabilization and solidification have been put in place as control measures by miners, these methods seem not to produce expected results so far as environmental protection is concerned. Communities are continuously reporting of contamination of their drinking water sources and ailments associated with mine contaminants.

However, the application of agricultural waste materials as adsorbents is on record to be effective in removing heavy metals from contaminated effluents (Abia *et al.*, 2003). What is also important in this application is that, agricultural waste is converted to useful materials (adsorbent). Thus, this approach encourages reuse of materials and thus, encourages waste minimization.

Shea nut cake is popularly known to be used as a water proofing agent to repair and mend cracks in huts, doors and traditional beehives (Wallance-Bruce, 1995). However, nothing is known about its ability to adsorb heavy metals from contaminated water. Consequently, this research seeks to fill this knowledge gap.

1.4 Objectives of Study

1.4.1 Main Objective

The main objective of this research was to remediate Arsenic and Lead contaminated mine processed laden water using palm kernel cake and shea nut cake as the adsorbents.

1.4.2 Specific Objectives:

The specific objectives were to determine:

- the rate of adsorption of arsenic (As) in simulated wastewater using sheanut cake as the adsorbent;
- the rate of adsorption of arsenic (As) in simulated wastewater using palm kernel cake as the adsorbent;
- the rate of adsorption of lead (Pb) in simulated wastewater using sheanut cake as the adsorbent; and
- the rate of adsorption of lead (Pb) in simulated wastewater using palm kernel cake as the adsorbent.

1.5 Limitations of the study

- i. This study will be carried out in the laboratory and may not necessarily reflect field observations
- ii. Only two metals (arsenic and lead) will be studied in this work, thus results obtained cannot be generalized for all metals.

1.6 Organization of Study

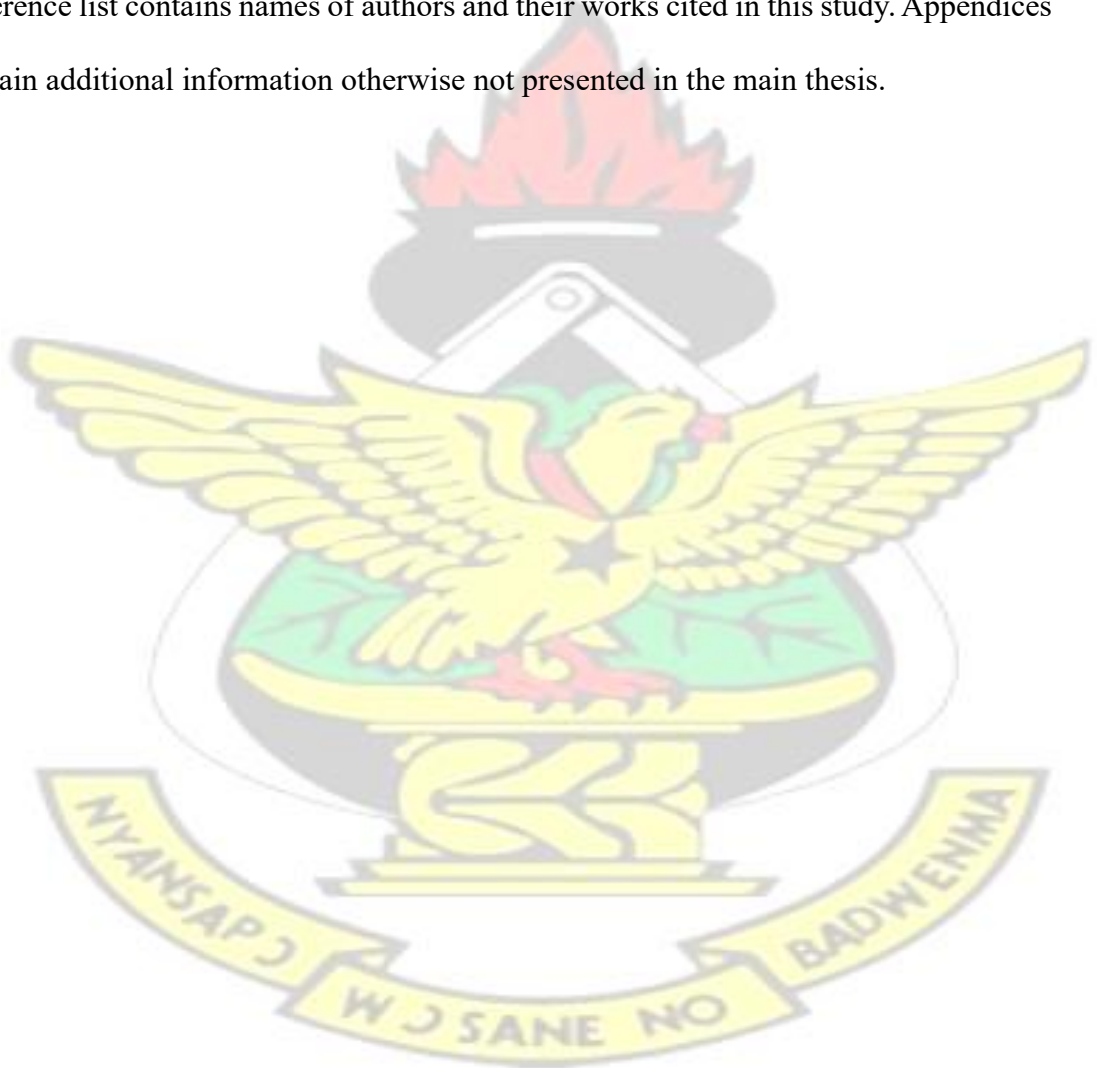
This study is organized into six chapters, list references and appendices. Chapter one gives the background of the study. It also contains the problem statement, objectives, justification, limitation and organization of the study.

Chapter two reviews relevant literature on the study topic considering previous works related to this study. Basic concepts, subjects and methods are also discussed in this chapter.

Methodology of the study is presented in chapter three. The research methodology is a systematic presentation of the approaches/methods used to arrive at the research findings.

Results obtained are presented in chapter four. Chapter five discusses the results of the study and in chapter six, conclusions and recommendations for further studies are presented..

Reference list contains names of authors and their works cited in this study. Appendices contain additional information otherwise not presented in the main thesis.



CHAPTER TWO

LITERATURE REVIEW

2.1 Mining in Ghana

Ghana has been mining for decades now, but it was until the 1890's that it commissioned corporate mining. Ashanti Gold Field Corporation, a pioneer mining company began work in the Obuasi district of Ghana in 1895 developing the Ashanti and other mines which have produced most of the gold in the country since 1900. In 2004, Ashanti Gold Corporation (A.G.C) merged with AngloGold to become AngloGold Ashanti, making it the world's second largest producer of gold (www.sikabourcrystalmining.blogspot.com).

Preceding 1989, small scale mining activities particularly gold was considered illegitimate due to the absence of legislative instruments to control their activity. Therefore, illegal operators smuggled their merchandise to neighboring countries for business. About US\$15 million worth of gold from illegal mining was smuggled out in 1985 to neighboring countries (Tetteh, 2011). To sort of control illegal mining, government made a policy decision in 1989 to regularize the operations of artisanal and small- scale miners. The result was the promulgation of small scale mining law, PNDC law 218 (1989). The year 2006 was greeted with the legal regime of small scale mining being integrated into the new minerals and mining Act, (Act 703).

2.2 Types of Mining in Ghana

There are three types of mining in Ghana, but only two are legally backed by law. They are large and small scale mining. With small scale mining, mining is usually 25 acres in size allocated only to Ghanaian Nationals to undertake mining in accordance with the minerals Act 2006 guidelines. Usually, the holders of license for small scale

concessions seek the collaboration of foreign mining experts to make available to them expertise, equipment, etc for their mutual benefits. The holders of such concessions act as dominant partners taking their share of resource mined when such is declared by the license usually come with EPA permit and as such holders are allowed to go into mining straight away without any legal restriction.

Small scale consession license is usually valid for five years but can be renewed for another five years upon expiration of the first term (Minerals and Mining Act of 2006).

Large scale consession is usually more than 25 acres either as a block (21 hectares) or blocks of mining concession joined together. Mostly, large scale concession run into hundreds of kilometers cutting across districts within a particular region of Ghana. There is a rigid legal framework that governs large scale concessions.

However, in certain circumstance, the mineral commission can recommend to the Minister in charge of mining in Ghana to issue mining lease when application is made after reconnaissance stage. In large scale concessions mining lease is usually given for 30 years or less and renewable for further 30 years after expiration of the forest lease (Minerals and Mining Act 703 of 2006).

Artisanal gold mining as indicated earlier is not backed by law. The artisans, usually Ghanaians, may or may not require concession permit, machines or expertise. This situation has led to the deterioration of the environment. It is the commonest form of gold mining in Ghana. In Ghana, artisanal mining is commonly called '*galamsey*' and usually requires the use of simple equipment

(www.sikabourcrystalmining.blogspot.com).

With small scale mining, there are two main types. Those that are done with small equipment along the banks of rivers (They usually involve the use of Chinese-made

processing equipment known as “changfa” equipment along the banks of major rivers- Ankobra, Offin, Birim). The main difference between the two main mining types in Ghana is that, small scale mining is reserved for the Ghanaian nationals while foreigners can engage in the large scale mining as indicated by the PNDC law on small scale mining (Amankwah *et al.*, 2003).

2.3 Impacts of Mining on the Environment

Mining generally has its environmental challenges whether operating legally or otherwise. The obvious ones are forest degradation, water pollution, noise pollution and the destruction of farm lands and crops (Akordor, 2012).

Profound environmental challenges seen in mining communities were caused by the mining boom and its associated misuse of mining chemicals which since has resulted in decline in safe drinking water and air quality as well as loss of ecological biodiversity (Akabza, 2001).

Nature and human activities both account for the deterioration of surface and subsurface water (Carpenter *et al.*, 1998). Heavy metal contaminations of surface and ground water have especially been experienced in gold mining communities (Davis, 1994). In recent times the popularity of gold mining has dwindled because of its association with arsenic, mercury, lead and other heavy metal contamination of the environment (Essumang *et al.*, 2007). Weathering of the heaped mine waste for instance results in the release of metals including mercury, arsenic, lead and cadmium (Essumang *et al.*, 2007). Increase in illegal mining activities in recent times poses some dangers to the environment including increase in soil erosion, destruction of most forest, release of chemicals and heavy metals due to pit collapse, and destruction of farm lands.

2.4 Arsenic (As)

2.4.1 Arsenic in the environment

Arsenic is common in the Earth's crust, thus it is naturally occurring. The adult human body is believed to contain approximately 20 mg of arsenic distributed in all tissues with higher concentrations in the skin, hair and nails (Abermathy *et al.*, 1999).

Since arsenic is naturally occurring, human exposure can occur through food chain, via dermal adsorption and breathing. Generally, the extent of non-occupational exposure to arsenic depend on a number of factors including the local geochemistry as well as the level of anthropogenic activity. Given the above factors, it can be said that arsenic is a natural contaminant of groundwater and possibly drinking water and therefore poses as a worldwide public health issue (Abermathy *et al.*, 1999).

As the world population increases, one of the most crucial resources for man is potable water; however, its accessibility is decreasing as a result of illegal mining activities. Also it is reported that the world's water resources is diminishing at a very fast rate due to illegal mining activities and that, in every group of five people, one person lacks access to potable water (BBC News, 2009). Dealing with arsenic exposure is quite a challenge because of its various forms of existence that is in both organic and inorganic forms (Chapman, 1996).

Soil arsenic contents are usually higher at areas closer arseniferous stockpiles and in zones of gold, silver, sulphides of zinc and lead (Chapman, 1996). Natural weathering of rocks and soil add some forty thousand tons of arsenic to ocean annually contributing < 0.01 mg/l input to water on worldscale (WHO, 1992).

Arsenic also gets into the environment through combustion of arsenic-based products and also from fly-off of fly-ash storage areas closer to power plants and nonferrous smelters (Chapman, 1996).

Arsenic concentration of 0.5 mg/l is recommended as WHO guideline value (tolerable level) (WHO, 1985). Areas of high arsenic levels are the mining areas, especially where the mineral is in association with pyrite and arsenopyrite.

Inorganic arsenic and its derivative compounds in food chains are gradually degraded to a less hazardous form of arsenic by methylation (Whelan *et al.* , 1960).

2.4.2 Modes of exposure to arsenic

According to WHO (1985), the commonest mode of introducing arsenic into the body is through ingestion. Intake around 20 to 30 mg/day has been recorded and this wide range is a reflection of the composition of diets and sources of water used in the preparation of beverages. In addition to food, drinking water is a significant source of arsenic contamination and that all other intake of arsenic (inhalation, oral and dermal) is usually small in composition (ATSDR, 2002).

2.5 Lead (Pb)

2.5.1 Sources of lead in the environment

Lead is a toxic contaminant. According to Green Peace International (2010), lead is the commonest pollutant in Ghana especially in and around the scrap yards. The conventional method of treatment of lead waste includes membrane related technology precipitation, oxidation-reduction reactions, removal with activated carbon, and ion exchange (Wang *et al.*, 2003). These methods are cost ineffective and usually generates sludge, whose disposal is troubling. Bio-sorption is a process that utilizes lowcost

biosorbents to sequester toxic heavy metal. Lead is a metal with no known biological importance to humans (WHO, 2012), therefore should not be found in food and drinking water.

2.5.2 Effect of Lead Pollution

Lead is a toxic contaminant even at very low amounts. Heavy lead poisoning can cause encephalopathy, while moderate lead poisoning results in neurobehavioral and intelligent deficit (Ozer, 2009). Lead (Pb) poisoning in human can cause serious damage to the kidney, nervous system, reproductive system, liver and brain. Pb accumulates in the bones and poisoning may be exposure especially where lead pipes are common and lead poisoning may be diagnosed from a blue line harmful to the developing brain of a foetus, younger children and pregnant women (Ozer, 2009).

2.5.3 Mode of exposure of Lead

Water is the rare but important source of lead exposure especially where lead pipes are common (WHO, 2012). Vehicles exhaust pipes are another way of releasing lead into the environment. Pollutants from industry can contaminate water and the atmosphere through the effluent produced from the industries. Lead in the environment can be distributed through air and also, discharge into soil and water.

2.6 Decontamination of Arsenic and Lead in the Environment

Some plants are able to accumulate Pb and Ag (Garbisu & Alkorta, 2003). In this context, plants have been described as solar-driven pumping stations (Cunningham *et al.*, 1995) which can actually remove these contaminants from the environment. As a rule, arsenic is readily bioavailable while lead is moderately bioavailable thus arsenic is easily biodegraded as compared to lead (Garbisu & Alkorta, 2003).

Also, a number of microbes are able to use some metals or metalloids as electron donors or acceptors in energy metabolism (Ehrlich, 1997) in which processes they decontaminate them. For example *Pseudomonas arsenitoxidans* reduces the toxicity of arsenic by oxidizing AsO_2^- to AsO_3^{3-} (Ehrlich, 1997, Ilyaletdinov & Abrashitova, 1981). Microbial detoxification of lead on the other hand involves enzymatic and or non-enzymatic methylation of the metal (Ehrlich, 1997, Frankenberger & Karlson, 1992).

Adsorption is one other method use to contain heavy metals including As and Pb. Generally, agricultural materials are used as adsorbents for heavy metal containments. These include maize leaves (Adesola *et al.*, 2006); plants seeds (Edogbanya *et al.*, 2013; Oboh & Alluyor, 2008; Kumari *et al.*, 2006); pomegranate peels (El-Ashtoukhy *et al.*, 2007) and orange and banana peels (Annadurai *et al.*, 2002). The mode of sorption by these agricultural waste products has been attributed to intrinsic adsorption and Columbic interaction between the biosorbent and the toxic metal ions (Igwe *et al.*, 2006). In an experiment by El-Ashtoukhy *et al.* (2008), an over 95% adsorption of Pb was achieved using pomegranate peels. Also, a study by Roy *et al.* (1993) achieved over 90% removal of as and Pb using green algae and ground rice hulls.

2.7. Adsorption

Adsorption of metal or metalloid species such as arsenate at difference pH tends to increase with concentration of adsorbate and converges towards the maximum adsorption capacity value for the sorbents (Dzombak & Morel, 1990; Goldberg, 1986).

Chilton *et al.* (2002), also define adsorption isotherm as a graphical representation which illustrates the relationship between the amount adsorbed by a unit weight of adsorbent and the amount of adsorbate remaining in a test medium at equilibrium.

Sorption isotherms are used as the empirical models.

Adsorption isotherm can be generated based on number of theoretical explanations. The simplest model that can be used to explain a single layer adsorption is the Langmuir equation (Meghea *et al.*, 1998). The Langmuir equation is based on the principles of kinetics with the assumption that, a uniform surface is a single layer of adsorbed material at constant temperature. Langmuir constant is related to the maximum adsorption capacity and energy of adsorption, respectively.

According to Maltson and mark (1971) and Chilton *et al.* (2002), Freundlich isotherm is the most popular adsorption model used to describe a single solute system. They stated that, the model is an empirical equation based on the distribution of solute between the solid phase and the aqueous phase at equilibrium.

2.7.1 Freundlich Adsorption Isotherm

It is a curve relating the amount of a solute on the surface of an adsorbent to the amount of the solute in the liquids with which it makes contact. With respect to gases, it is an empirical expression representing the isothermal variation of adsorption of a quantity of gas adsorbed by unit mass of solid adsorbent with pressure.

For temperature effects, the amount of mass that is adsorbed is plotted against the temperature which gives an idea about the variation of adsorption with temperature.

Freundlich adsorption isotherm is mathematically represented by

$$\frac{x}{m} = Kp^{1/n}$$

Where:

x = mass of adsorbate m =

mass of adsorbent p = equilibrium

pressure of adsorbate

K and n are constants for a given adsorbent and adsorbate at a particular temperature.

At higher pressure, $1/n=0$, hence the extent of adsorption becomes independent of pressure.

Contaminants transport in subsurface waters may be hindered when they bind and react with the soil. A typical laboratory measurement of this binding employs batch equilibrium test with adsorption represented by isotherms that are typically linear at higher contaminant concentration. To illustrate transport of contaminant through a surface, a linear adsorption isotherm is usually used because of its simplicity (Sharma & Reddy, 2004). Following an increased amount of contaminant and an initial linear increase in sorption, is a slowed and nonlinear rate of sorption as the capacity of the sorbent become exhausted.

2.7.2 Langmuir Adsorption Isotherm

The Langmuir adsorption isotherm is the most common model used to quantify the amount of adsorbate adsorbed on an adsorbent as a function of partial pressure or concentration at a given temperature (Willey Inter-Science, 1996). Langmuir isotherm

is based on different assumptions; one of which is that, dynamic equilibrium exists between adsorbed molecules and the free gaseous molecules.

2.7.2.1 Basic Assumptions of the Langmuir Isotherm

For the simplest case the following assumptions hold true:

- (I) Surfaces with adsorption sites are perfectly flat planed with no corrugation (assumed the surface is homogeneous).
- (II) Each site can accommodate at most a molecule of mono-layer coverage only.
- (III) All sites are equivalent.
- (IV) Interactions between adsorbate molecules on adjacent site are non-existent.

The model assumes adsorption and desorption are elementary processes in which the rate of adsorption R_{ad} and the rate of desorption R_d are given by

$$R_{ad} = K_{ad} P_a (S)$$

$$R_d = K_d (A_{ad})$$

Where P_a is the partial pressure of A over the surface, (S) is the concentration of bare site in number/m², (A_{ad}) is the surface concentration of A in molecules/m² and K_{ad} and K_d are constants. At equilibrium the rate of adsorption is equivalent to the rate of desorption (Willey Inter-Science, 1996).

2.7.4 Applications of Adsorption

Examples of the industrial uses of adsorption include air-conditioning, adsorption chillers, synthetic resin and water purification. In pharmaceutical industry adsorption is used as a means to prolong neurological exposure to specific drugs or parts thereof (en.Wikipedia.org/wiki sorption). Adsorption of molecules onto polymer surface is used in various applications such as in the development of non-stick coatings and in various biomedical devices (en.Wikipedia.org/wiki sorption).

2.8 Palm Kernel Cake (PKC)

Palm kernel cake is an agricultural waste common in West Africa particularly in Ghana. PKC is obtained out of the extraction of oil from crushed kernel. The estimated annual total production of PKC is at 1.4 million tons (Mustaffa, 1987).

PKC is considered a protein feed with high fibre content. It is however lower than fish meal and groundnut cake especially in protein value (Devendra, 1977). Its utilization has effectively reduced the cost of milk production as a result of substituting expensive conventional feedstuffs, such as fish meal for it (Yusoff et al., 1987).

2.9 Shea Nut Cake (SNC)

Shea tree grows naturally throughout Guinea savanna regions. It is perennial and its mature tree height varies considerably with some attaining heights of over 14 metres and girth of over 1.75 metres (Yidana, 1994). It is an economic crop indigenous to the Guinea and Sudan savanna zones of Nigeria and Northern Ghana. It is clear that apart from the nutritional and medicinal properties of shea nut tree, the evidence on its economic and environmental importance to the economy of the nation are enormous (www.lautechaeedu.com). With adequate exploitation, the crop has the potential to make significant contribution to the country's Gross Domestic Product (GDP). Recently the shea tree has gained significant economic value because of the heavy demand for its butter and the need to find substitutes for cocoa butter (www.lautechaeedu.com).

Shea butter is useful cocoa butter substitute due to its similar melting point (32-45°C) and a high amount of di-stearin (30%) and some slearo-palmitine (6.5%) which make it blend with cocoa butter without altering flow properties (Nikrema & Umali, 2007).

The residual meal of shea butter is also used as a water proofing agent to repair and mend cracks in huts, windows, doors, and traditional beehives (Wallance-Bruce, 1995).

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CHAPTER THREE

MATERIALS AND METHOD

3.1 Sample Collection

Palm Kernel Cake (PKC) was sourced from Asante Bekwai palm kernel oil extraction industrial area in the Ashanti Region of Ghana while sheanut cake (SNC) was obtained from Buipe in the Northern Region of Ghana.

3.2 Processing of Adsorbents

The collected samples of PKC and SNC were sun-dried for three days and blended separately to produce uniform samples. Each sample was sieved through 2 mm pore spaces. To enhance the adsorption capability of the adsorbents, the sieved samples were dried at room temperature for 7 days followed by drying at a temperature of 50°C in an electric oven for 3 hours.

3.3 Preparation of Arsenic and Lead Solutions

3.3.1 Simulated Arsenic “Wastewater”

The stock solution of arsenic for this study was a concentration of 1000 ppm arsenic in approximately 0.5 nitric acid. All the working solutions (‘wastewater’) of arsenic were obtained by serial dilutions of the stock solution with deionized water. Initial pH and temperature of the working solutions were 2.67 and 25°C, respectively.

Using the dilution formula:

$$C_1V_1 = C_2V_2,$$

the following concentrations of arsenic solution were obtained: 10, 20, 30, 40 and 50 mg/l.

To prepare a solution containing 10 mg/l of arsenic, the following computations were carried out:

$$C_1 = 1000 \text{ ppm} = 1000 \text{ mg/L}$$

$$C_2 = 10 \text{ mg/L}$$

$$V_2 = 500 \text{ ml}$$

$$V_1 = ?$$

$$\text{From; } C_1 V_1 = C_2 V_2 \quad V_1 = \frac{C_2 V_2}{C_1}$$

$$V_1 = \frac{10 \frac{\text{mg}}{\text{L}} \times 500 \text{ ml}}{1000 \text{ mg/L}} = 5 \text{ ml}$$

Therefore, 5 ml of stock solution was taken using a pipette with standard error ± 0.05 ml. This was dispensed into a volumetric flask and distilled water was added to the 500 ml mark. The mixture was properly shaken and labeled as 10 mg/l of arsenic. The same procedure was used to obtain concentrations of 20, 30, 40 and 50 mg/l of arsenic solutions.

3.3.2 Simulated Lead “Wastewater”

The stock solution of lead for the study was 1000 ppm (1000 mg/l) lead in approximately 0.5 nitric acid. All measurements were carried out with a pipette of standard error ± 0.5 ml. All the working solutions of the lead were also prepared by serial dilution of the stock solution with distilled water. Temperature and pH of working solutions were 25°C and 2.67, respectively.

Using the dilution formula, 10, 20, 30, 40 and 50 mg/l were obtained. With respect to 10 mg/l lead solution, the following procedure was applied.

$$C1 = 1000ppm = 1000mg/L$$

$$C2 = 10 mg/L$$

$$V2 = 500ml$$

$$V1 = ?$$

$$\text{From } C1V1 = C2V2 \quad V1 = \frac{C2V2}{C1}$$

$$V1 = \frac{10mg/L \times 500ml}{1000mg/L} = 5 ml$$

Thus, 5 ml of stock solution was taken using a pipette of standard error ± 0.05 ml. It was dispensed into a volumetric flask and deionized water added to the 500 ml mark. The mixture was appropriately shaken and labeled as 10 mg/l of lead. The same procedure was used to obtain concentrations of 20, 30, 40 and 50 mg/l of lead solutions.

3.4 Adsorption of Arsenic by PKC and SNC

Using an electronic balance, 2, 4, 6, 8 and 10 g of the adsorbent (PKC) were weighed into small sized transparent polythene bags and stored in a desiccant until use. In determining the adsorption of arsenic by 2 grams of PKC, 50 ml of arsenic solution at concentrations of 10, 20, 30, 40 and 50 mg/l were measured into 5 separate beakers containing the 2 grams of PKC (Table 1).

All beakers were manually swirled for a minute and their contents transferred into corresponding labeled bottles. The bottles were packed in HS501 digital shaker and swirled for two hours. In the first one hour, samples were swirled gently to ensure a uniform mixture. A more rigorous swirling was programmed for the remaining one hour to ensure an effective adsorption process. The samples were allowed to settle for one hour. Subsequently, the samples were filtered using a vacuum filter and the filtrates

transferred into corresponding labeled test tubes. The experiments were triplicate and the samples were processed for Atomic Absorption Spectrophotometer (AAS) measurements.

The procedure was repeated with 4, 6, 8 and 10 grams of PKC. Each mass of PKC was added to 50 ml arsenic solution containing arsenic concentrations of 10, 20, 30, 40 and 50 mg/l.

The whole procedure was repeated using SNC as the adsorbent.

Table 3.1: Concentration of Arsenic solution to 2 g of PKC

Sample code	Mass of PKC (g)	Concentration of Arsenic (mg/l)
AP ₁	2	10
AP ₂	2	20
AP ₃	2	30
AP ₄	2	40
AP ₅	2	50

Likewise, in determining the rate of adsorption of arsenic by SNC at a fixed mass (2 g), 50 ml of 10, 20, 30, 40 and 50 mg/l arsenic solutions were measured into 5 separate beakers containing 2 g each of SNC labeled AS₁, AS₂, AS₃, AS₄ and AS₅, respectively as illustrated Table 2 below.

The procedure was repeated with 4, 6, 8 and 10 grams of SNC. Each mass of SNC was added to 50 ml arsenic solution containing arsenic concentrations of 10, 20, 30, 40 and 50 mg/l.

Table 3.2: Concentration of Arsenic solution to 2 g of SNC adsorbent

Sample code	Mass of SNC (g)	Conc. of As (mg/l)
AS ₁	2	10
AS ₂	2	20
AS ₃	2	30
AS ₄	2	40

3.5 Adsorption of Arsenic by PKC and SNC at a Fixed Concentration

To determine the adsorption efficiency of arsenic by PKC at a fixed concentration, 50 ml each of 100 mg/l arsenic solution was measured into 5 separately labeled beakers. To each of these beakers containing arsenic solutions, 2, 4, 6, 8 and 10 g of PKC were added separately as illustrated in Table 3.

Table 3.3: Different masses of PKC adsorbent in 100 mg/l arsenic solution

Sample	Mass of PKC (g)	Conc. of As (mg/l)
APM ₁	2	100
APM ₂	4	100
APM ₃	6	100
APM ₄	8	100
APM ₅	10	100

To determine the adsorption efficiency of arsenic by SNC at a fixed concentration, 50 ml each of 100 mg/l arsenic solution was measured into 5 separately labeled beakers. To each of these beakers containing arsenic solutions, 2, 4, 6, 8 and 10 g of SNC were added separately as illustrated in Table 4 below. These masses were chosen considering the availability of equipment.

Table 3.4: Different masses of SNC adsorbent in 100 mg/l arsenic solution

Sample	Mass of SNC (g)	Conc. of As (mg/l)
ASM ₁	2	100
ASM ₂	4	100
ASM ₃	6	100
ASM ₄	8	100
ASM ₅	10	100

All beakers in each batch experiment were swirled for a minute and their contents transferred into corresponding labeled bottles. The bottles were packed in HS₅₀₁ digital *shaker* and scheduled for two hour's duration of swirling. In the first one hour, samples were swirled gently to ensure a uniform mixture. A more rigorous swirling was programmed for the remaining one hour of scheduled time to ensure an effective adsorption process. The samples were allowed to settle for an hour. Subsequently, samples were filtered using a vacuum filter and filtrates transferred into corresponding labeled test tubes. The experiments were triplicated and the samples processed for Atomic Absorption Spectrophotometer (AAS) measurements.

3.6 Adsorption of Lead by PKC and SNC at a Fixed Mass

Adsorption of Lead by PKC at a fixed mass (2g) was investigated by measuring 50 ml of 10, 20, 30, 40, and 50 mg/l lead solutions into 5 separate beakers containing 2g each of PKC labeled LP₁, LP₂, LP₃, LP₄ and LP₅ as illustrated in Table 5 below.

Table 3.5: Concentration of lead solution to 2 g of PKC adsorbent

Sample code	Mass of PKC (g)	Conc. of Pb
LP ₁	2	10
LP ₂	2	20
LP ₃	2	30
LP ₄	2	40
LP ₅	2	50

Likewise, in investigating adsorption of lead by SNC at a fixed mass (2 g), 50 ml of 10, 20, 30, 40 and 50 mg/l lead solutions were measured into 5 separate beakers containing 2 g each of SNC labeled LS₁, LS₂, LS₃, LS₄ and LS₅ as illustrated in Table 6 below.

Table 3.6: Concentration of Pb to 2 g of SNC adsorbent

Sample code	Mass of SNC (g)	Conc. of Pb
LS ₁	2	10
LS ₂	2	20
LS ₃	2	30
LS ₄	2	40

All beakers in each batch experiment were swirled for a minute and their contents transferred into corresponding labeled bottles. The bottles were packed in HS₅₀₁ digital shaker and scheduled for two hour's duration of swirling. In the first one hour, samples were swirled gently to ensure a uniform mixture. A more rigorous swirling was programmed for the remaining one hour of scheduled time to ensure an effective adsorption process. The samples were allowed to settle for an hour. Subsequently, samples were filtered using a vacuum filter and filtrates transferred into corresponding labeled test tubes. The experiments were triplicate and sample processed for Atomic Adsorption Spectrophotometer (AAS) measurements.

3.7 Adsorption of Lead by PKC and SNC at a Fixed Concentrations

In determining the adsorption of PKC at a fixed concentration (100 mg/l), 50 ml each 100 mg/l lead solution were measured using a pipette into 5 separately labeled beakers. To each of these beakers containing 100 mg/l lead solution, 2, 4, 6, 8 and 10 g of PKC were added separately and respectively as illustrated in Table 7 below. These masses were chosen considering the availability of equipment.

Table 3.7: Concentration of Pb to different masses of PKC adsorbent

Sample code	Mass of PKC (g)	Conc. of Pb
LPM ₁	2	100
LPM ₂	4	100
LPM ₃	6	100
LPM ₄	8	100
LPM ₅	10	100

Likewise in determining the adsorption of Lead by SNC at a Fixed Concentration (100 mg/l), 50 ml each 100 mg/l arsenic solution were measured with a pipette into 5 separately labeled beakers. To each of these beakers containing 100 mg/l lead solution,

2, 4, 6, 8 and 10 g SNC were added separately and respectively as illustrated in Table 8 below.

Table 3.8: Concentration of Pb to different masses of SNC adsorbent

Sample code	Mass of SNC (g)	Conc. of Pb
LSM ₁	2	10
LSM ₂	2	20
LSM ₃	2	30
LSM ₄	2	40
LSM ₅	2	50

Beakers in each batch of experiment were swirled for a minute and their contents transferred into corresponding labeled bottles. The bottles were packed in HS₅₀₁ digital shaker and scheduled for two hours duration of swirling. In the first one hour, samples were swirled gently to ensure a uniform mixture. A more rigorous swirling was programmed for the remaining one hour of scheduled time to ensure an effective adsorption process. The samples were allowed to settle for an hour. Subsequently, samples were filtered using a vacuum filter and filtrates transferred into corresponding labeled test tubes. The experiments were triplicated and sample processed for Atomic Absorption Spectrophotometer (AAS) measurements.

3.8 Statistical Analysis and Modeling

Results obtained were subjected to analysis of variance to determine levels of variations between treatments. The data obtained were also modeled in accordance with Langmuir adsorption isotherm as described by Cornansky *et al.* (2007). The Langmuir model is given by

$$C_e/m \dots\dots\dots (1)$$

$$C_e/m = 1/k + C_e/k \dots\dots\dots (2)$$

where; C_e is equilibrium concentration (mg/l) m is the mass of sorbent (mg/g) b is the ratio of adsorption and desorption rates k is the maximum uptake of the metal corresponding to site saturation C_e/m was plotted as the response variate and C_e as the explanatory variate in linear regression model.

Additionally, the data was also modeled along the Freundlich isotherm. The Freundlich isotherm as described by Ninlanja (2008) and Cernonsky *et al.* (2007) is given by:

$$M = KC_e^N \dots\dots\dots (3)$$

where; M is mass of the metal adsorbed per unit adsorbent (mg/l)

C_e is the equilibrium concentration (mg/l)

K and N are Freundlich constants.

The model is 'linearized' by applying logarithm to both sides of the equation giving;

$$\log M = \log K + N \log C_e \dots\dots\dots (4)$$

This modified model follows the equation of a straight line $y = mx + c$. Thus $\log M$ is used to plot response variate and $\log C_e$ as the explanatory variate in the linear regression model.

CHAPTER FOUR

RESULTS

4.1 Adsorption of Arsenic by PKC and SNC at varying Concentration

The highest mean percentage sorption by 2 g of PKC was 55.20% and was attained in arsenic concentration of 50 mg/l while the lowest (19.70%) was in arsenic concentration of 10 mg/l (Table 4.1). The table 4.1 also shows that the percentage sorption increased with increasing concentration of arsenic in the solution.

Table 4.1: Mean sorption of Arsenic by 2 g PKC in varying arsenic concentrations

Initial conc. (mg/l)	Mass Of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
10	2	1.97	19.70
20	2	7.67	38.35
30	2	13.60	45.33
40	2	19.87	50.00
50	2	27.60	55.20

(Laboratory work, 2013)

Sorption of as by 2 g SNC behaved in a similar fashion as the 2 g PKC. Highest mean percentage sorption was 71.40% in 50 mg/l arsenic solution and the lowest was 30.00% in 10 mg/l arsenic solution (Table 4.2).

Table 4.2: Mean Sorption of As by 2 g SNC in varying arsenic concentrations

Initial Conc. (mg/l)	Mass of SNC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
10	2	33.0	30.0
20	2	11.83	59.17
30	2	19.8	66.0
40	2	27.3	68.25
50	2	35.7	71.4

(Laboratory work, 2013)

4.2 Adsorption of Arsenic by varying masses of PKC and SNC

According to Table 4.3, the highest and lowest mean percentage sorption by PKC in arsenic concentration of 100 mg/l was 62.66% and 49.51%, respectively. The Table also reveals that the rate of sorption decreased as the mass of PKC increased. On the contrary, rate of sorption decreased as the mass of SNC increased in 100 mg/l arsenic solution (Table 4.3).

Table 4.3: Mean sorption of as by PKC in fixed concentration of as solution

Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
100	2	62.66	62.66
100	4	61.37	61.37
100	6	60.51	60.51
100	8	56.19	56.19
100	10	49.51	49.51

(Laboratory work, 2013)

According to table 4.4 the highest and lowest sorption by SNC in a varied masses of fixed concentration are 73.98% and 52.25% respectively. Hence percentage sorption increases as the mass of SNC increases.

Table 4.4: Mean sorption of as by SNC in fixed conc. of as solution

Initial conc. (mg/l)	Mass of SNC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
100	2	52.25	52.25
100	4	63.05	63.05
100	6	67.81	67.81
100	8	69.8	69.8
100	10	73.98	73.98

(Laboratory work, 2013)

4.3 Adsorption of Lead by PKC and SNC in varying concentrations of Lead solution

With a fixed mass of 2 g of PKC as adsorbent, percentage sorption of between 31.00 and 88.80% of lead was achieved (Table 4.5). The rate decreased as lead concentration in the solution increased.

Table 4.5: Mean sorption of lead by 2 g of PKC in varying concentrations of lead solution

Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
10	2	8.88	88.80
20	2	17.63	88.0
30	2	20.01	33.0
40	2	26.71	32.0
50	2	34.57	31.0

(Laboratory work, 2013)

With the fixed mass of 2 g SNC adsorbent, mean percentage sorption of between 93.00% and 98.00% were achieved. Generally, sorption decreased with increase in concentration of Pb in the solutions (Table 4.6).

Table 4.6: Mean sorption of lead by 2 g of SNC in varied concentrations of lead solution

Initial Conc. (Mg/l)	Mass of SNC (g)	Average Sorption(Mg/l)	Percentage Sorption (%)
10	2	9.30	93.0
20	2	19.60	98.0
30	2	29.16	97.0
40	2	38.36	96.0
50	2	46.25	93.0

(Laboratory work, 2013)

4.4 Adsorption of Lead by varying masses of PKC and SNC

Sorption of lead by PKC varied with mass of the adsorbent (PKC). With 10 g of PKC, adsorption rate of 95.1% was attained whereas 77.6% was attained with 2 g of PKC in 100 mg/l of lead solution (Table 4.7).

Table 4.7: Mean Sorption of Pb by varying masses of PKC in fixed concentration of Pb solution

Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
100	2	22.05	77.96
100	4	21.14	78.87
100	6	16.48	83.52
100	8	10.55	89.45
100	10	4.94	95.06

(Laboratory work, 2013)

With different masses of SNC as adsorbent for lead, sorption rates of 83.18, 96.16, 98.29, 98.79 and 99.13% were achieved for SNC masses of 2, 4, 6, 8 and 10 g, respectively, in 100 mg/l lead solution (Table 4.8). The figure again shows that the effectiveness of sorption increased with the mass of the adsorbent.

Table 4.8: Mean Sorption of Pb by varying masses of PKC in fixed concentration of Pb solution

Initial Conc. (Mg/l)	Mass of SNC (g)	Average Sorption (Mg/l)	Percentage Sorption (%)
100	2	83.18	83.18
100	4	96.16	96.16
100	6	98.29	98.29
100	8	98.79	98.79
100	10	99.13	99.13

4.5 Comparison of adsorption of arsenic by PKC and SNC in varying concentrations of Arsenic solution (Laboratory work, 2013)

Figure 4.1 compares the efficiency of adsorption of arsenic by SNC and PKC adsorbents. It can be seen from the figure that SNC performed better than PKC in adsorbing arsenic from solution at all concentrations. For both adsorbents, efficiency of adsorption increased with increasing concentration of arsenic solution. There was no statistical significant difference ($p = 0.48$) between the means of PKC and SNC (Appendix C).

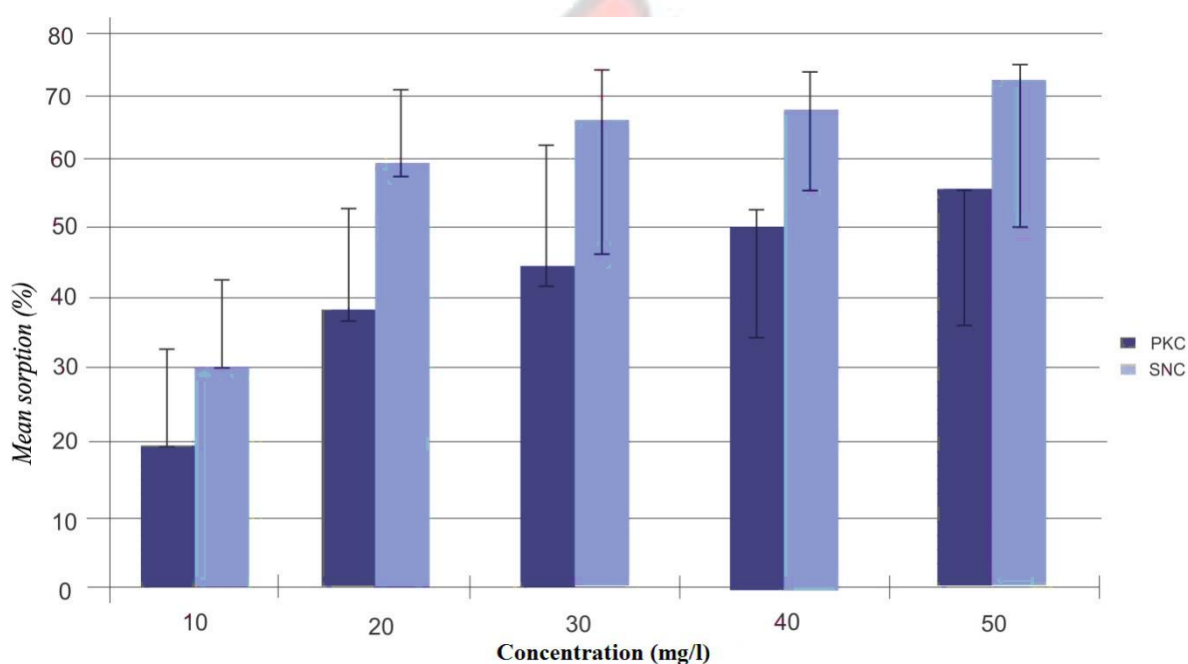


Figure 4.1: Comparison of adsorption efficiency of as by PKC and SNC in varying concentration of as solution

4.6 Comparison of adsorption efficiency of arsenic by varying masses of PKC and SNC

At a given mass of adsorbent, SNC performed better than PKC in adsorbing arsenic ions from the solution. For both adsorbents, sorption increased with increasing the

concentration of the adsorbates (Figure 4.1). There was statistically significant difference ($p=0.01$) between the means of PKC and SNC (Appendix C).

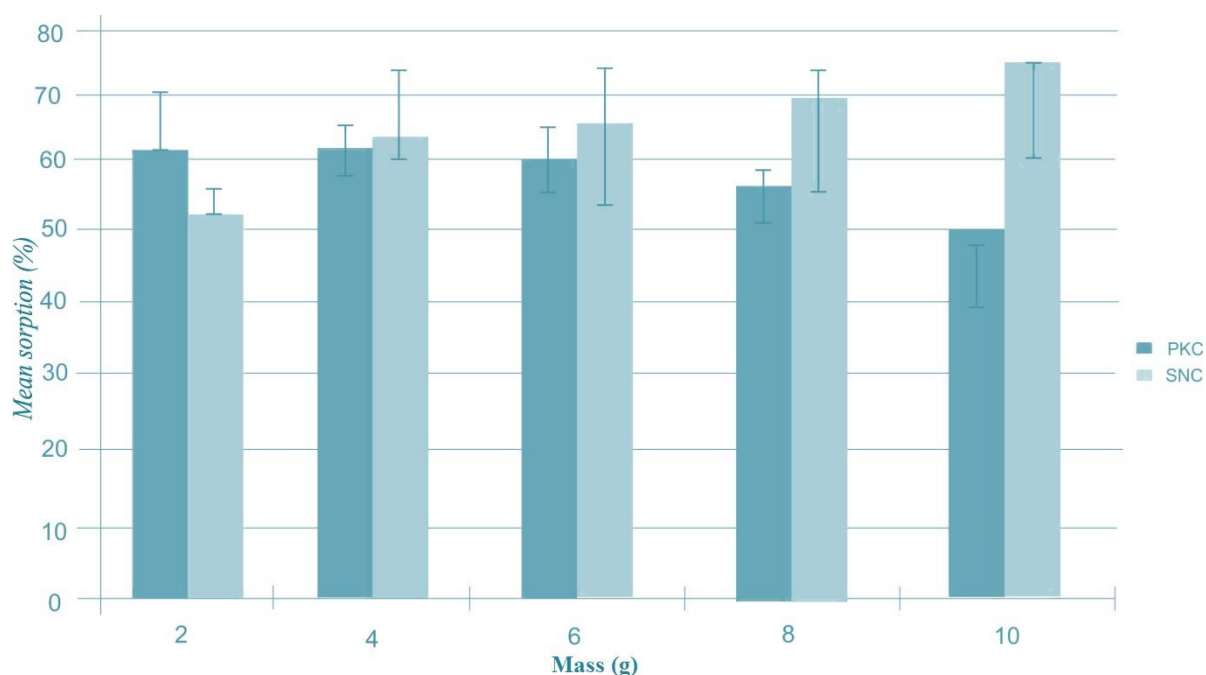


Figure 4.2: Comparison of adsorption efficiency of as by PKC and SNC in varying masses of adsorbents

4.7 Comparison of adsorption of Pb by PKC and SNC in varying concentrations of lead solution

SNC performed better than PKC in adsorbing lead ions from the solution (Figure 4.2). Maximum rate of sorption was attained at the mass SNC at 10 g, but generally did not show any particular trend. No statistically significant difference existed between PKC and SNC ($p = 0.08$).

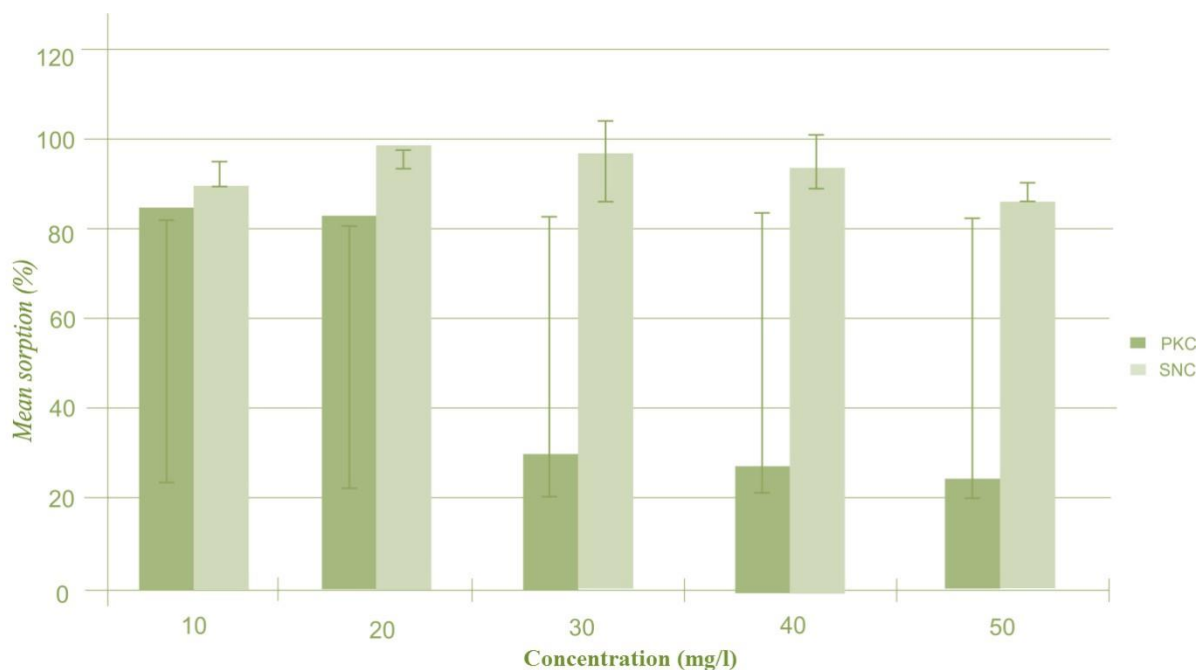


Figure: 4.3 Mean sorption of lead by PKC and SNC at varying concentrations of lead solution

4.8 Comparison of adsorption of lead by varying masses of PKC and SNC It can be seen from Figure 4.3 that SNC performed better than PKC as adsorbate for lead ions in solution. Also, for both SNC and PKC, sorption increased with increasing concentration of the adsorbates. PKC at concentration 30 mg/L started declining. There was no statistical significant difference ($p = 0.56$) between PKC and SNC (Appendix C).

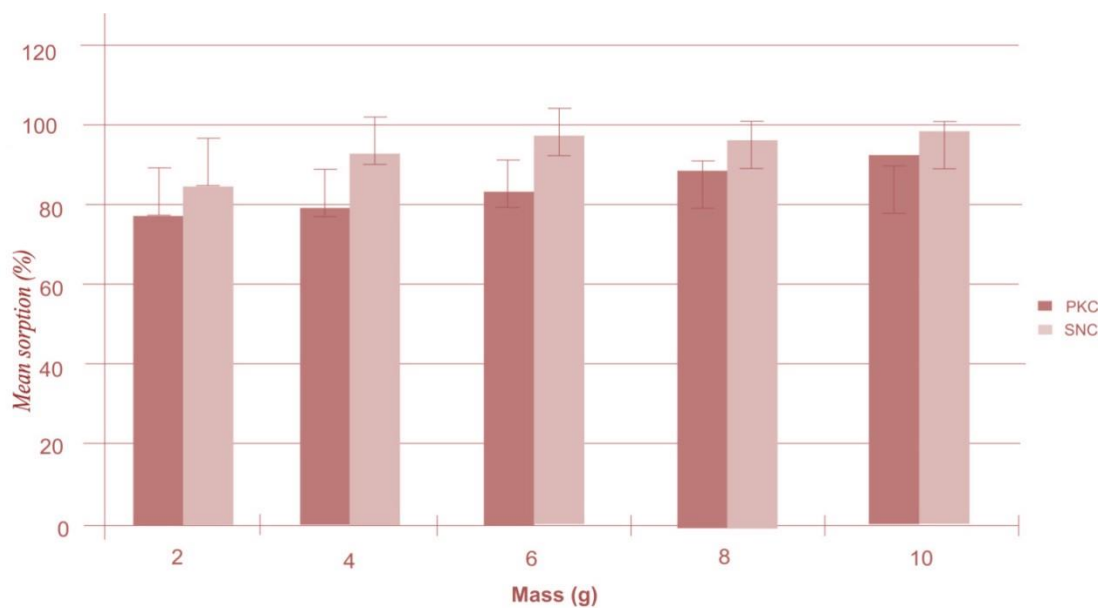


Figure 4.4: Mean sorption of lead by varying masses of PKC and SNC at fixed concentration of lead solution

4.9 Langmuir Sorption Models for PKC and SNC

To test the sorption of lead and arsenic using the Langmuir sorption model, the percentage sorption for the various concentrations were correlated with the equilibrium concentration. According to Jaynes and Boyd (1991), adsorption conforms to the Langmuir model when the value of the correlation coefficient is greater than 0.89.

4.9.1 Langmuir Model for PKC at varying concentrations of Arsenic solution

Results obtained from PKC moderately fitted into the model since R^2 value of 0.77 was obtained as shown in figure 4.5.

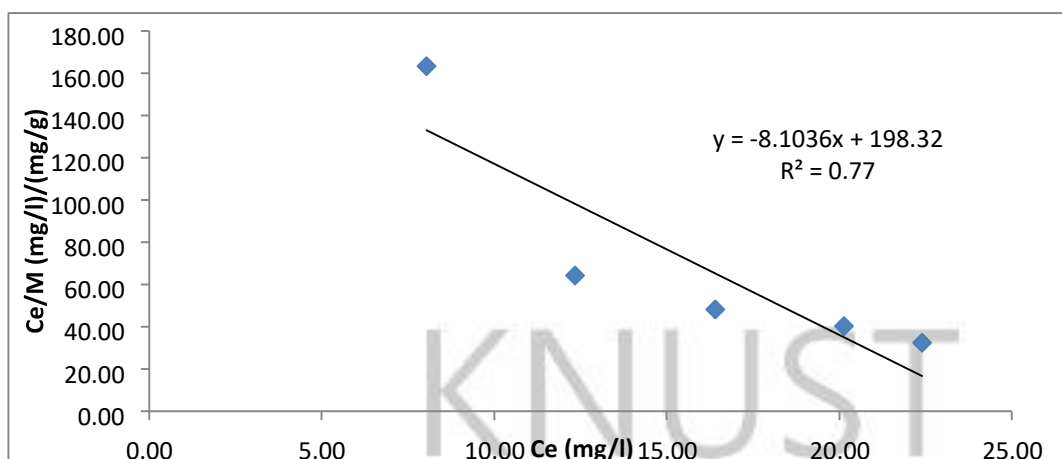


Figure 4.5: Langmuir model for PKC at varied concentrations of arsenic solution.

4.9.2 Langmuir Model for SNC at varying Concentrations of Arsenic Solution

Results obtained from SNC did not fit well into the model since R^2 value obtained was 0.53

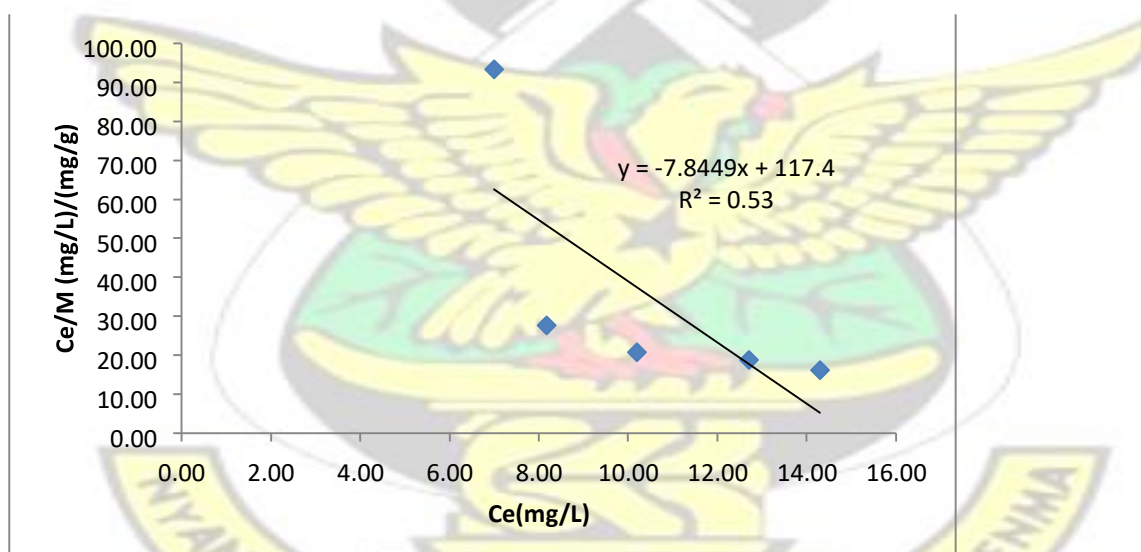


Figure 4.6: Langmuir model for SNC at varied concentrations of arsenic solution.

4.9.3 Langmuir Model for PKC at varying concentrations of Lead solution With a

fixed mass of PKC and varied concentrations of lead solution, the correlation

coefficient, $R^2 = 0.92$ which indicated that, the results fitted well into the Langmuir isotherm. The correlation is shown in Figure 4.7.

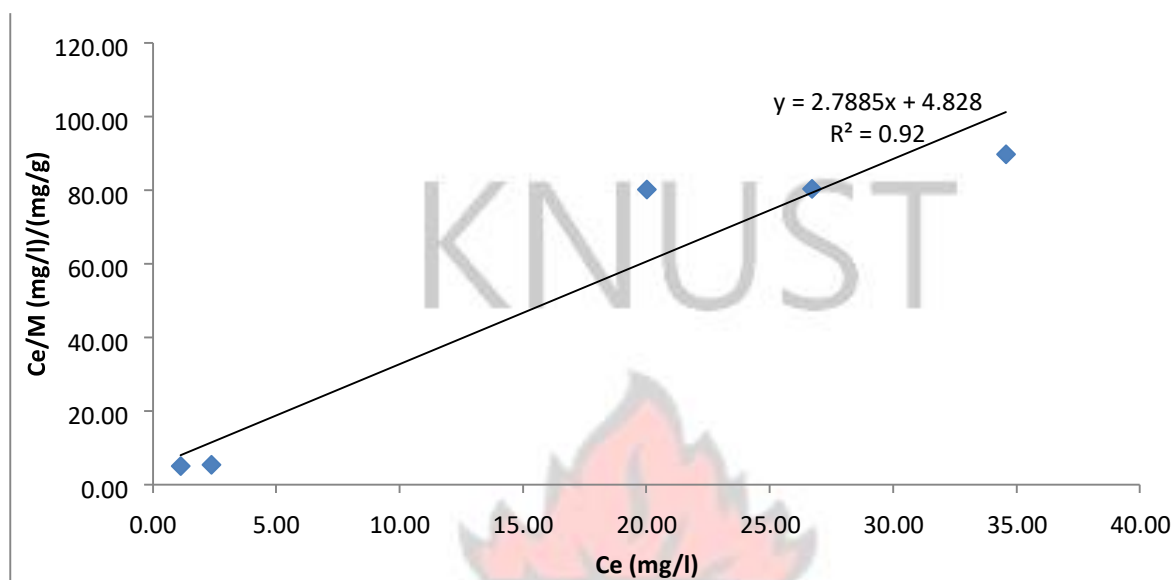


Figure 4.7: Langmuir model for PKC at varied concentrations of lead solution

4.9.4 Langmuir Model for SNC at varying concentrations of Lead Solution Results obtained for sorption by SNC when lead concentration in the solution was varied did not fit into this model since $R^2 = 0.41$ (Figure 4.8).

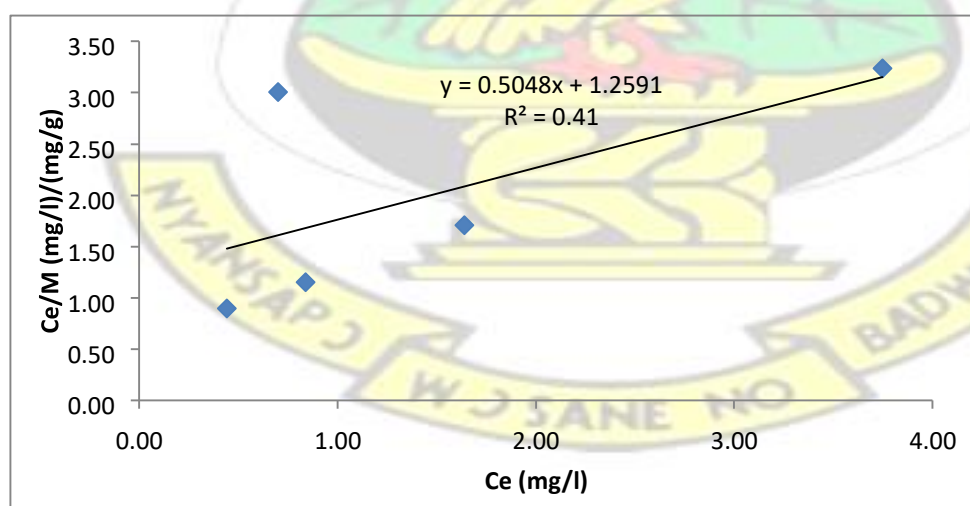


Figure 4.8: Langmuir model for SNC at varied concentrations of lead solution.

4.9.5 Langmuir Model for adsorption of Arsenic at varying masses of PKC R^2 value of 0.77 was obtained after results from this experiment were modeled along the

Langmuir isotherm (Figure 4.9). The value of 0.77 obtained implied that results from this experiment fitted moderately well into the Langmuir isotherm.

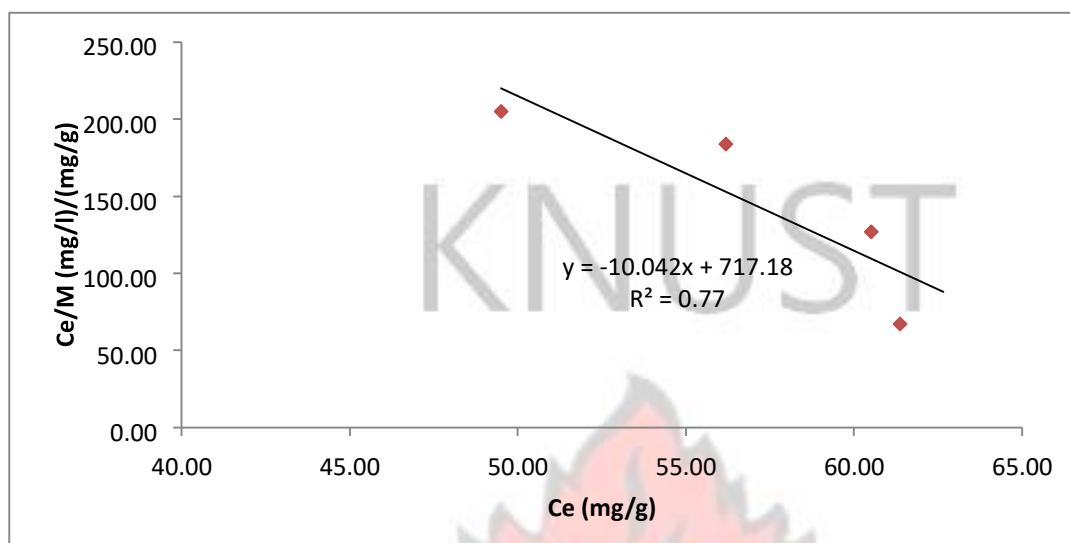


Figure 4.9: Langmuir model for adsorption of arsenic by varied masses of PKC.

4.9.6 Langmuir Model for Adsorption of Arsenic at varying masses of SNC

According to Figure 4.10, $R^2 = 0.91$ proved that the results from this experiment fitted well into the Langmuir isotherm.

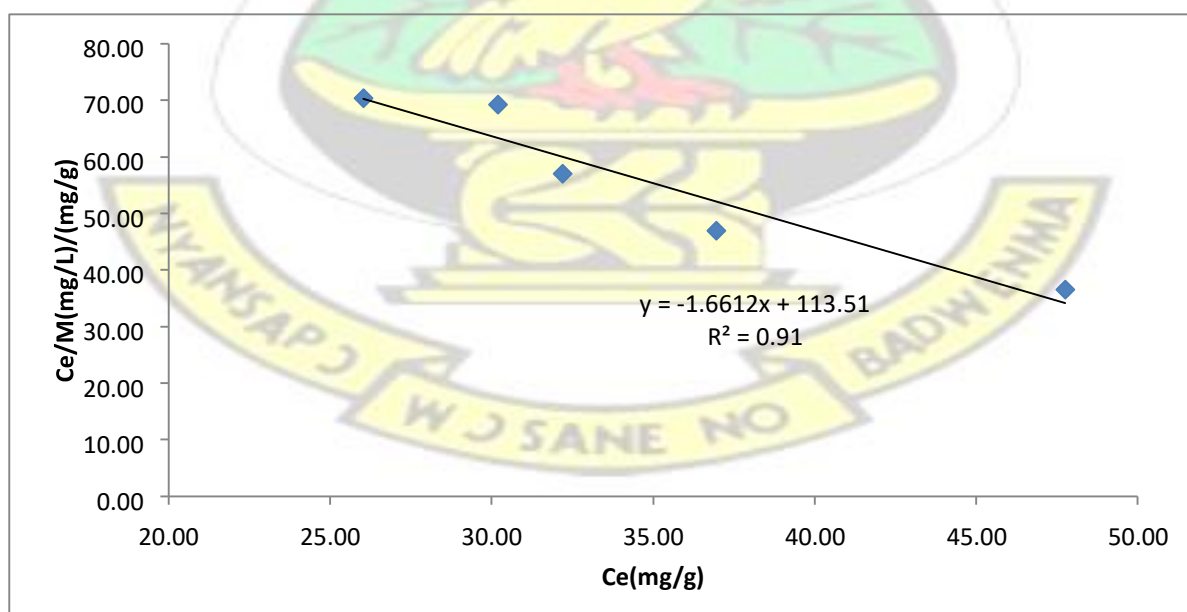


Figure 4.10: Langmuir model for adsorption of arsenic by varied masses of SNC.

4.9.7. Langmuir Model for adsorption of Lead at varying masses of PKC When the mass of the PKC was varied at fixed concentration of lead, and the results modeled along the Langmuir isotherm, the value obtained for the correlation coefficient R^2 was 0.10, indicating this experiment completely violated this model. The extent of this deviation is shown in Figure 4.11 below.

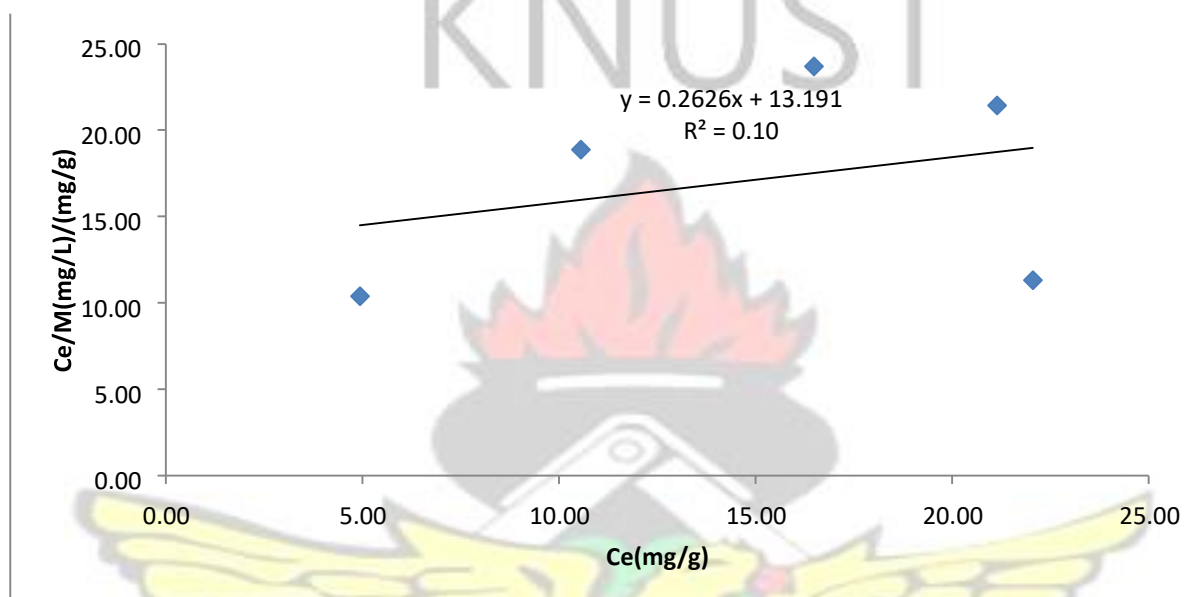


Figure 4.11: Langmuir model for adsorption of lead by varied masses of PKC

4.9.8 Langmuir Model for Adsorption of Lead at varying masses of SNC Results from this experiment produced an approximated perfect fit into the Langmuir isotherm producing a R^2 value of 0.99. All data points were approximately linearly aligned as illustrated in Figure 4.12.

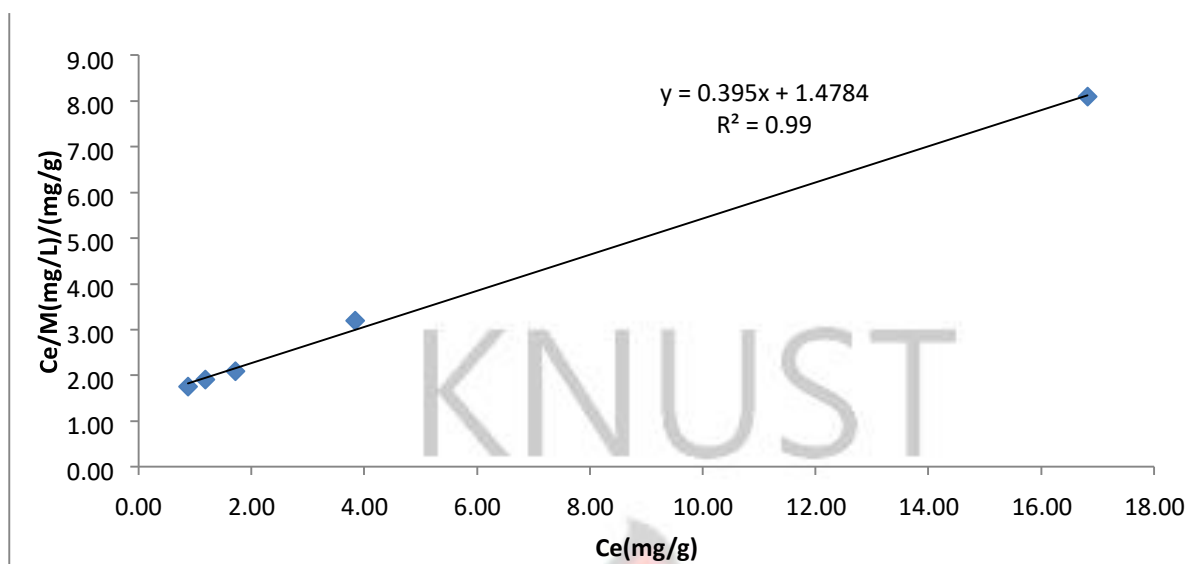


Figure 4.12: Langmuir model for adsorption of lead by varied mass of SNC.

4.10 Freundlich Sorption Models for PKC and SNC

Quantitative evaluation of biosorption using Freundlich sorption model shows that some of the results performed poorly while others fitted well into the isotherm. For the Freundlich isotherm model, R^2 should be approximately equal to 1.

4.10.1 Freundlich Model for PKC at varying concentrations of Arsenic solution

Results from this experiment strongly fitted into the Freundlich isotherm producing a R^2 value of 0.99 as illustrated in the Figure 4.13.

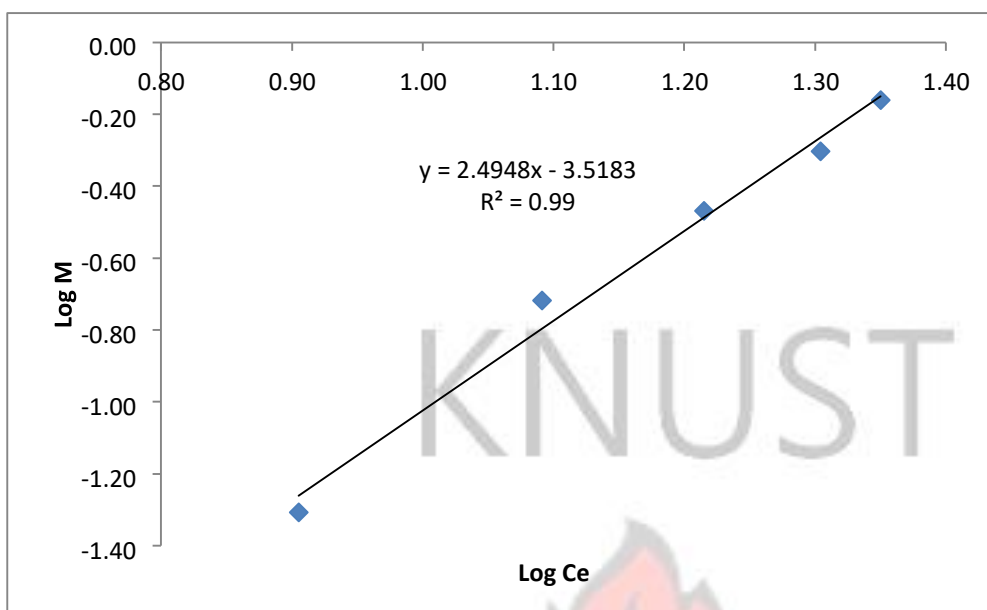


Figure 4.13: Freundlich model for PKC at varied concentrations of arsenic solution.

4.10.2 Freundlich Model for SNC at varying concentrations of Arsenic solution

Results from this experiment fitted moderately well into the Freundlich isotherm producing R^2 value of 0.87 as illustrated in the Figure 4.14.

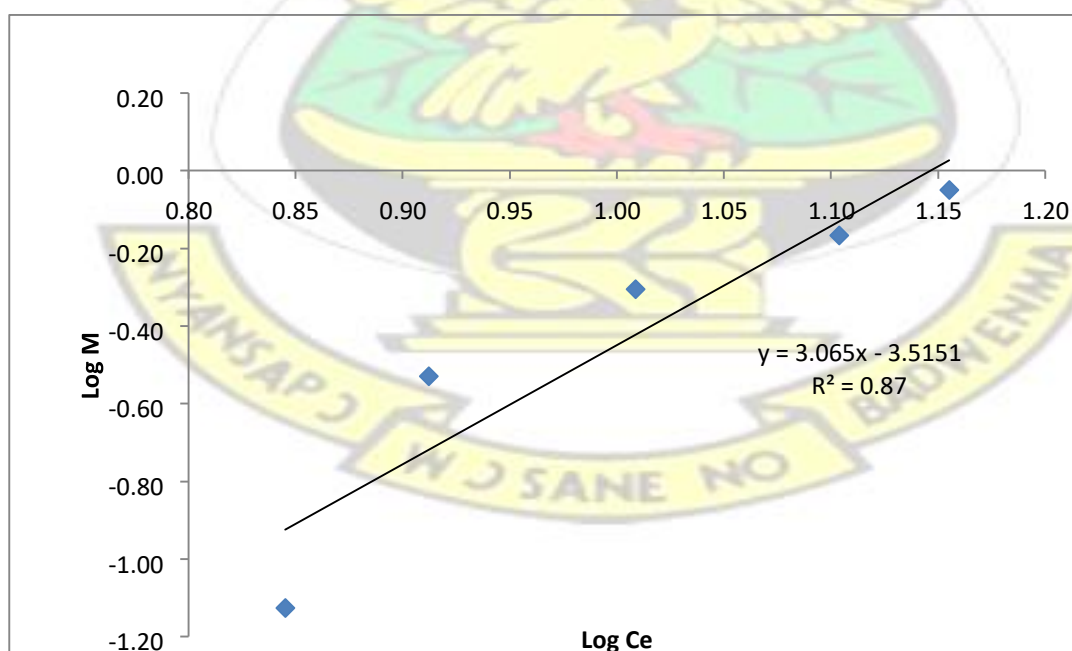


Figure 4.14: Freundlich model for SNC at varied concentrations of arsenic solution.

4.10.3 Freundlich Model for PKC at varying concentrations of Lead solution.

Results from this experiment did not fit into the Freundlich isotherm with R^2 value of 0.06 Figure 4.15.

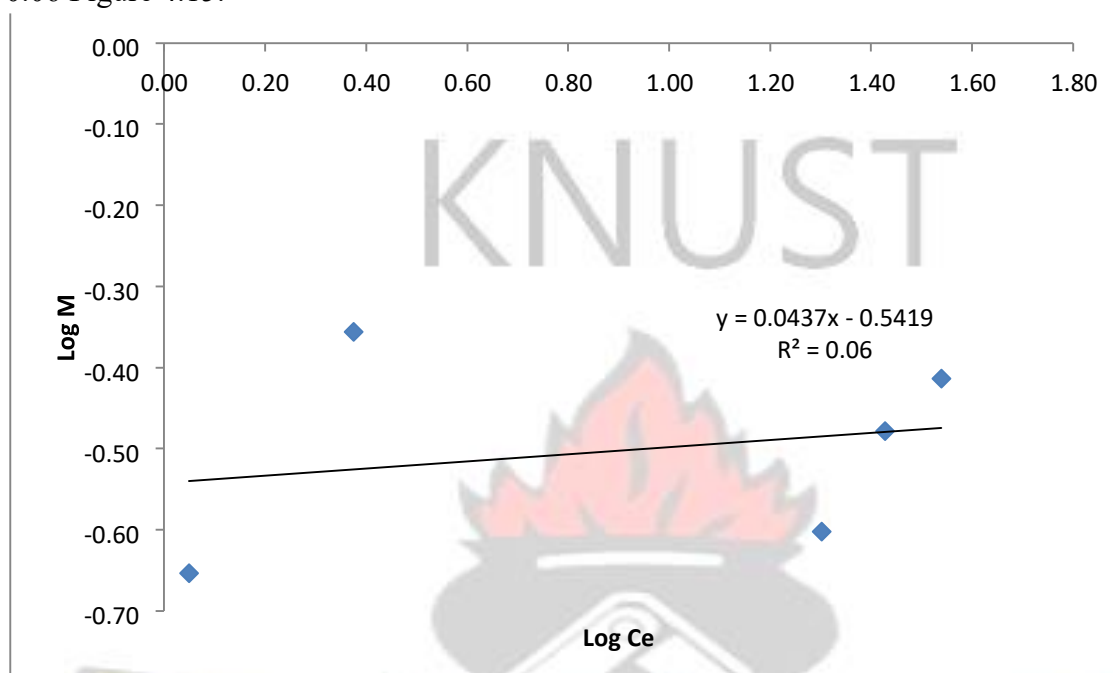


Figure 4.15: Freundlich model for PKC at varied concentrations of lead solution.

4.10.4 Freundlich Model for SNC at varying concentrations of Lead solution

Results from this experiment modeled well along the Freundlich isotherm with R^2 value of 0.92 as indicated in Figure 4.16.

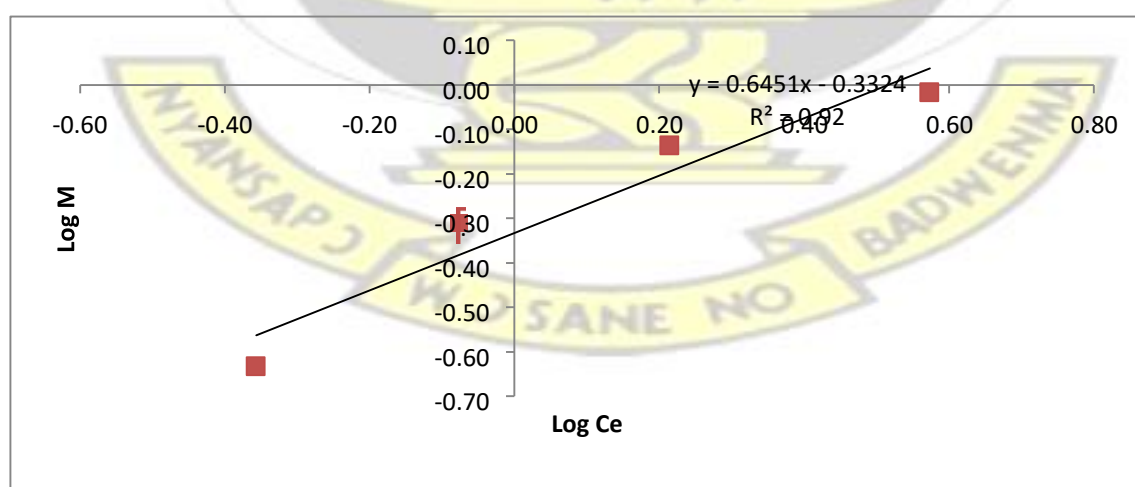


Figure 4.16: Freundlich Model for SNC at varied concentrations of lead solution

4.10.5 Freundlich Model for Adsorption of Arsenic at varying Masses of PKC

Results from this experiment moderately fitted into the Freundlich isotherm producing a R^2 value of 0.55 as illustrated in Figure 4.17.

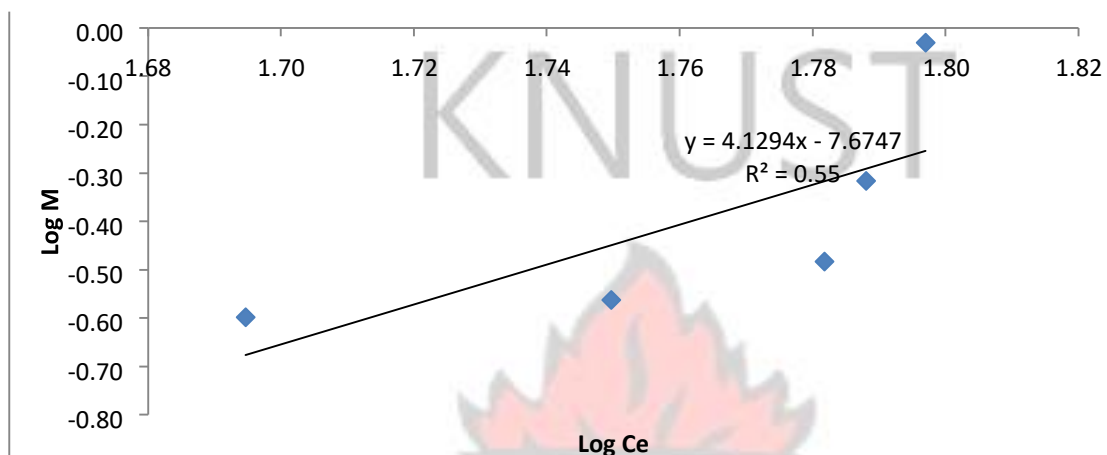


Figure 4.17: Freundlich model for adsorption of arsenic at varied masses of PKC.

4.10.6 Freundlich Model for adsorption of Arsenic at varying masses of SNC

Results from this experiment showed $R^2=0.98$, which implied a good fit into Freundlich isotherm (Figure 4.18).

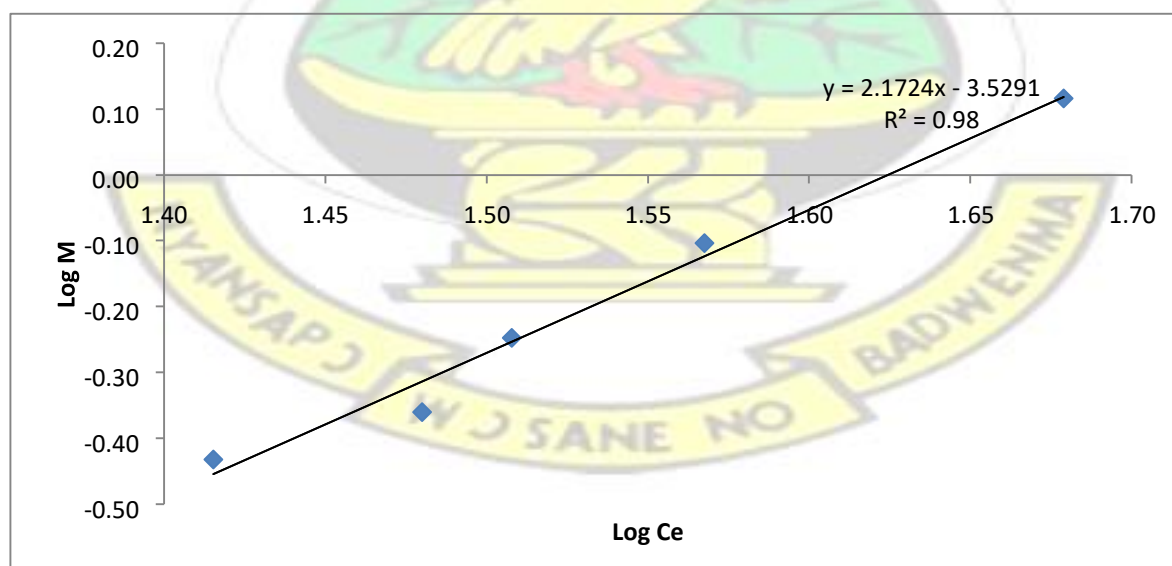


Figure 4.18: Freundlich model for adsorption of arsenic at varied masses of SNC

4.10.7 Freundlich Model for adsorption of Lead at varying masses of PKC By keeping the concentration of lead constant and varying the mass of PKC, a value of 0.64 was obtained for R^2 which indicates a moderate fit into the Freundlich isotherm (Figure 4.19).

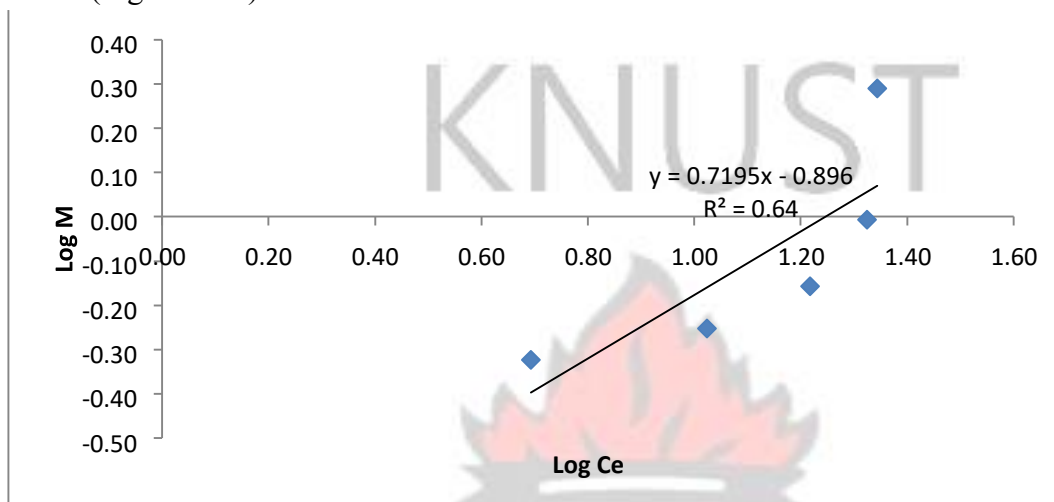


Figure 4.19: Freundlich model for adsorption of lead at varied masses of PKC.

4.10.8 Freundlich Isotherm for Adsorption of Lead at Varying Masses of SNC

Results from this experiment modeled well along the Freundlich isotherm. It produced R^2 value of 0.98. The correlation is illustrated in Figure 4.20.

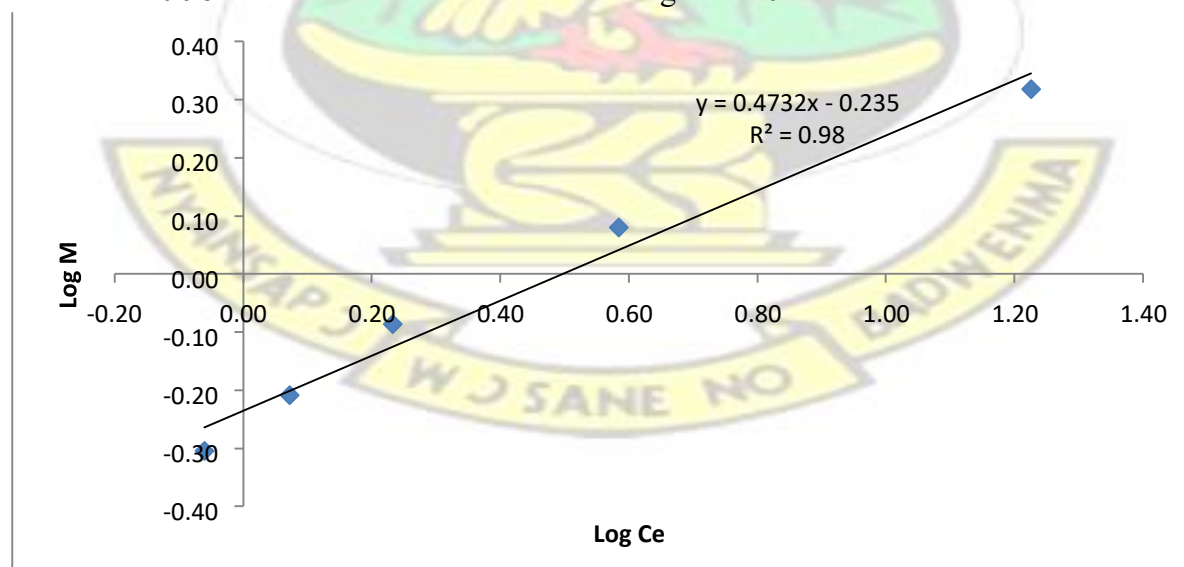


Figure 4.20: Freundlich model for adsorption of lead at varied masses of SNC

CHAPTER FIVE

DISCUSSION

5.1 Adsorption Capacity of PKC and SNC in varying Concentrations of as and Pb Solutions

The adsorption efficiency of both palm kernel cake (PKC) and shea nut cake (SNC) adsorbents increased as the concentration of arsenic in the solutions increased. For 2 g of each adsorbent, the mean sorption efficiency ranges were 19.7-55.2% and 30-71.4%, respectively, for PKC and SNC in arsenic wastewater.

With a fixed mass of 2 g of PKC as adsorbent, percentage sorption of between 30.0 and 88.8% was achieved in various concentrations of simulated lead wastewater. The rate decreased as lead concentration in the solution increased. For the SNC adsorbent, mean percentage sorption ranged from 93% and 98%, and sorption generally appeared to decrease slightly with concentration.

The sorption efficiency of the materials investigated in this study was attributed to the electrostatic forces of attraction between the positively charged ions of the metal species (Arsenic and Lead) and the positively and negatively charged protein ends of the PKC and SNC adsorbents. Pushpa *et al.* (2005), made similar observation when they used *Moringa oleifera* seeds as adsorbents.

The general rise in adsorption with an increase in the concentration of arsenic contaminant could be associated with the increasing availability of metal ions in solution, and possibly suitable experimental conditions (pH=2.67 and temperature=25°C). This conclusion on adsorption efficiencies has also been made by Afidenyo (2011), and Hunter *et al.* (1993).

However, the general observation that increasing the concentration of lead, unlike arsenic led to reductions in the adsorption efficiency in both PKC and SNC is attributed to the dissociation of so much ions in solution, which probably could have resulted in a state of super-saturation. Super-saturation reduces the mobility of ions and, thus hinders adsorption processes. Another possible cause for this observation is the probability that the electrostatic forces of attraction between lead and the adsorbents were weak due to the low valence of lead. Also, clogging at binding sites could have contributed to the observed trends in these experiments.

The findings that no statistical significant differences existed among the adsorption efficiencies of PKC and SNC when concentrations of the metal ions were varied implied that the adsorption efficiencies of both adsorbents were roughly the same. That is, in both cases, almost the same amounts of dissociated ions were available and that both PKC and SNC could be used to achieve similar results in remediating a contaminated medium of arsenic and lead pollutants.

5.2 Adsorption Capacity of varying Masses of PKC and SNC adsorbents in Arsenic and Lead Solutions

The adsorption efficiency of the adsorbents (PKC and SNC) depended on the mass of the adsorbents in fixed concentration (100 mg/l) of arsenic or lead solution. While the efficiency decreased as the mass of PKC was varied from 2 g through 10 g in 100 mg/l simulated arsenic solution, the opposite was observed when SNC was used as the adsorbent, i.e. the efficiency of sorption increased as the mass of SNC was increased from 2 g to 10 g.

However, the efficiency of both adsorbents increased with increasing adsorbent mass in 100 mg/l of simulated lead "wastewater". Differences in adsorption capacities of the

PKC and SNC as their masses were varied was attributed to the availability of binding sites (surface area). The larger the mass, the more are binding sites available and the better the adsorption capacity of the sorbent. Hence, the highest sorption efficiency being observed in the 10 g of both adsorbents.

No significant statistical difference in mean percentage sorption of lead was observed for PKC and SNC at varied masses ($p > 0.05$). This implies that, the adsorption capacities of PKC and SNC with respect to lead were not too different, and that these adsorbents provided almost the same amount of active binding sites for adsorption. Thus, in the application of PKC and SNC to clean up lead pollution, similar results can be expected from both sorbents.

However, in the case of mean percentage sorption of arsenic by PKC and SNC at varied concentrations, significant statistical differences were observed for the sorbents ($p < 0.05$). The implication of this is that, the adsorption efficiencies of PKC were very different from that of SNC, probably due to differences in the amount of dissociated ions in solution.

5.3 Comparison of Adsorption Efficiencies of PKC and SNC in varying concentration of Arsenic and Lead Solutions

Results from the experiments show that SNC performed better than PKC in adsorbing arsenic and lead from wastewater. For both adsorbents, efficiency of adsorption rose with increasing concentration of arsenic solution. The efficiency of adsorption depends on the amount of protein in the sorbents. Sharma *et al.* (2007), explain that amino acids, depending on pH, possess both negative and positive charged ends, and thus, capable of generating the appropriate binding sites for attracting anionic or cationic metal ions.

Therefore, given the fact that SNC contains 40% crude protein mainly of saponine, theobromine, and tannins

(Hassan and Yeong, 1999), it provided more adsorption sites for the metal ions of the contaminants to be adsorbed. The crude protein content of PKC is 14-21%, and mainly comprises of arginine, serine, valine and leucine (Hair-Bejo and Alimon, 1995), and provided fewer binding sites for adsorption and this could have accounted for the lesser efficiencies of adsorption observed at all concentrations of arsenic and lead in the wastewater.

Another reason which could have accounted for these results is the acidic medium (pH=2.67) in which the experiments were performed. Protein works better in acidic media than in basic or neutral media. This means that adsorption would be higher for the adsorbent with higher protein content as compared with one with lower protein content. Hence, the SNC performing better as adsorbent than the PKC.

Again, another reason which could have accounted for the lower efficiencies of PKC was the presence of some oily substances on the surface of the filtrate seen after the mixing and vacuum filtration. This oily substance possibly could have hindered the drying of the PKC when both sorbents were subjected to the same conditions of drying in the Sun and the electric oven.

5.4 Ease of Adsorption between Arsenic and Lead

In each experimental set-up, higher efficiencies were achieved for lead than for arsenic. This means that lead ions were much more readily adsorbed than the arsenic ions in solution. This observation was true for both adsorbents. The observed differences in the adsorption efficiencies could be attributed to fact that, at a low pH (2.67), lead most

possibly dissociated much more easily in solution than arsenic, and therefore produced more free ions to interact with the adsorbents. It can therefore be concluded that, PKC and SNC are good adsorbents of lead than arsenic. This conclusion was also reached by Pushpa *et al.* (2005), in their experiment involving the use of *Moringa oleifera* seeds to adsorb arsenic and lead from wastewater.

5.5 Langmuir and Freundlich Models

Sorption models take into account sorption levels at different concentrations and do not solely depend on the optimum sorption. Therefore, a very high optimum sorption may not guarantee a very good model.

5.5.1 The Langmuir Model

According to Jaynes and Boyd (1991), adsorption conforms to the Langmuir Adsorption Model when the value of the correlation coefficient is greater than 0.89. The results of the models indicate that some of the experiments fitted very well with the Langmuir isotherm while others did not. When the concentrations of arsenic and lead in the wastewater were varied, the PKC adsorbent produced a moderate fit for arsenic ($R^2 = 0.77$) and a perfect fit for lead ($R^2 = 0.92$) while the SNC adsorbent did not fit at all for both arsenic and lead ($R^2=0.53$ and $R^2=0.41$, respectively).

Again, with different masses of both adsorbents, PKC produced a moderate fit for arsenic ($R^2=0.77$) and no fit for lead ($R^2=0.1$). However, the SNC conformed to the Langmuir isotherm for both metals (i.e., $R^2=0.91$ and $R^2=0.99$, respectively for As and Pb). According to Atef (2009), a major requirement for the Langmuir model to be adhered to is that, a monolayer of adsorbates should saturate on the surface of adsorbents. This probably explains why some of the experiments produced data that conformed to the Langmuir isotherm.

Also, given the results obtained, it can be deduced that Langmuir isotherm can be used to model the trend of adsorption for these experiments under controlled pH and temperature. Though Langmuir isotherm is less sensitive to data error, it has some bias towards aligning the data in the middle at high concentration ranges (Journal of American Chemical Society). Therefore, this model is not conclusive in itself and should be complemented with other models for better predictability of adsorption trends of adsorbents.

Results obtained also indicate that SNC conformed better to the Langmuir model than PKC when the mass of the adsorbents were varied, and this again, can be related to the presence of larger surface area and active binding sites in SNC than PKC. Larger surface areas enhance the formation of monolayers.

5.5.2 The Freundlich Model

Results of some of the experiments followed the Freundlich Model. The import derived from these particular experiments was that, possibly there was an initial surface adsorption followed by a condensation effect resulting from extremely strong solutesolute interactions. This relation was made based on the assertion of Atef (2009) that Freundlich model often represents an initial surface adsorption followed by a condensation effect resulting from extremely strong solute-solute interaction.

What could also be said about these experiments is that, the Freundlich isotherm under controlled conditions of pH and temperature can be used to predict the adsorption outcomes of these experiments. In this model too, results from SNC modelled better

than those of PKC and this observation was also associated with the presence of numerous binding sites in SNC than PKC.

KNUST



CHAPTER SIX

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

In this research work, Palm Kernel Cake (PKC) and Shea Nut Cake (SNC) were used as adsorbents to remediate arsenic and lead contaminated wastewater. The results of the experiments indicate that both adsorbents were effective in removing arsenic and lead ions from the wastewater. The sorption efficiency of both the PKC and SNC depended on the mass of the adsorbent material, as well as the concentration of the metal (As or Pb) in solution.

With a fixed mass of PKC or SNC at varying concentrations of as (10-50 mg/l) in wastewater, maximum sorption efficiencies of 55.20% and 71.40% were attained with PKC and SNC, respectively. The efficiencies rose with increase in concentration of as in the wastewater. However, the efficiencies of sorption decreased with increase in concentration of Pb in the wastewater. Maximum efficiencies attained with PKC and SNC were 88.80% and 98.00%, respectively.

With varying mass of PKC and SNC in 100 mg/l solution of as, maximum sorption efficiencies were 62.70% and 74.00%, respectively for PKC and SNC. Sorption efficiency increased with mass of SNC whereas it decreased with that of PKC. Again, sorption efficiency increased with mass of PKC and SNC in 100 mg/l solution of Pb. Maximum efficiencies were 95.10% and 99.10%, respectively for PKC and SNC.

In general, the results of the study indicates that shea nut cake (SNC) performed better than palm kernel cake (PKC) in adsorbing arsenic and lead from wastewater due to the higher protein content in the shea nut cake.

The results of the models also indicate that, generally, the experiments performed with SNC provided better fit than the PKC for the Langmuir and Freundlich isotherms for both metals.

Finally, it can be concluded that both shea nut cake and palm kernel cake can be used to effectively clean up arsenic and lead contaminated water since appreciable sorption efficiencies were attained in the study.

6.2 Recommendations

The following are recommended:

- Further research should be conducted to determine the influence of pH on the sorption efficiencies of PKC and SNC.
- The performance of a combination of SNC and PKC was not studied in this work, thus I recommend it for further research
- The effects of temperature on the adsorption efficiencies of SNC and PKC should also be investigated
- Research to compare the performance of SNC and PKC against Activated Carbon (AC)

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APPENDICES A. AVERAGE SORPTIONS BY PKC AND SNC

A1. Sorption of Arsenic by 2 g PKC in varying conc. of as solution

Sample code	Initial conc. (mg/l)	Mass Of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
AP ₁	10	2	1.97	19.70
AP ₂	20	2	7.67	38.35
AP ₃	30	2	13.60	45.33
AP ₄	40	2	19.87	50.00
AP ₅	50	2	27.60	55.20

A2. Sorption of Arsenic by 2 g SNC in varying conc. of as solution

Sample code	Initial Conc. (mg/l)	Mass of SNC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
AS ₁	10	2	33.0	30.0
AS ₂	20	2	11.83	59.17
AS ₃	30	2	19.8	66.0
AS ₄	40	2	27.3	68.25
AS ₅	50	2	35.7	71.4

A3. Sorption of Arsenic by varying masses of PKC in fixed conc. of arsenic solution

Sample Code	Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
APM1	100	2	62.66	62.66
APM2	100	4	61.37	61.37
APM3	100	6	60.51	60.51
APM4	100	8	56.19	56.19
APM5	100	10	49.51	49.51

A4. Sorption of Arsenic by varying masses of SNC in fixed conc. of arsenic solution

Sample Code	Initial conc. (mg/l)	Mass of SNC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
ASM1	100	2	52.25	52.25
ASM2	100	4	63.05	63.05
ASM3	100	6	67.81	67.81
ASM4	100	8	69.8	69.8
ASM5	100	10	73.98	73.98

A5. Sorption of Pb by PKC in different concentrations of Pb solution

Sample Code	Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
LP1	10	2	8.88	88.80
LP2	20	2	17.63	88.0
LP3	30	2	20.01	33.0
LP4	40	2	26.71	32.0
LP5	50	2	34.57	31.0

A6. Sorption of Pb by SNC in different concentrations of Pb solution

Sample Code	Initial Conc. (Mg/l)	Mass of SNC (g)	Average Sorption(Mg/l)	Percentage Sorption (%)
LS1	10	2	9.30	93.0
LS2	20	2	19.60	98.0
LS3	30	2	29.16	97.0
LS4	40	2	38.36	96.0
LS5	50	2	46.25	93.0

A7. Sorption of Pb by varying masses of PKC in fixed concentration of Pb solution

Sample Code	Initial Conc. (mg/l)	Mass of PKC (g)	Average Sorption (mg/l)	Percentage Sorption (%)
LPM1	100	2	22.05	77.96
LPM2	100	4	21.14	78.87
LPM3	100	6	16.48	83.52
LPM4	100	8	10.55	89.45
LPM5	100	10	4.94	95.06

A8. Sorption of Pb by varying masses of PKC in fixed concentration of Pb solution

Sample Code	Initial Conc. (Mg/l)	Mass of SNC (g)	Average Sorption (Mg/l)	Percentage Sorption (%)
LSM1	100	2	83.18	83.18
LSM2	100	4	96.16	96.16
LSM3	100	6	98.29	98.29
LSM4	100	8	98.79	98.79
LSM5	100	10	99.13	99.13

B. Mean + Standard deviations

B1. Arsenic in PKC at varying concentration

Concentration (mg/l)	Mean ± SD
10	1.97 ± 0.06
20	7.67 ± 0.12
30	13.60 ± 0.00
40	19.87 ± 0.12
50	27.60 ± 0.00

B2. Arsenic in SNC at varying concentration

Concentration (mg/l)	Mean ± SD
10	3.0 ± 0.00
20	11.83 ± 0.06

30	19.80 ± 0.00
40	27.30 ± 0.00
50	35.70 ± 0.00

B3. Arsenic in PKC at varying masses

Mass (g)	Mean ± SD
2	37.34 ± 0.01
4	38.64 ± 0.01
6	39.50 ± 0.01
8	43.81 ± 0.00
10	50.50 ± 0.01

B4. Arsenic in SNC at varying masses

Mass (g)	Mean ± SD
2	52.25± 0.01
4	63.05 ± 0.00
6	67.81 ± 0.00
8	69.80 ± 0.00
10	73.98± 0.00

B5. Lead in PKC at varying concentration

Concentration (mg/l)	Mean ± SD
10	8.88± 0.00
20	17.63 ± 0.00
30	9.99 ± 0.00
40	13.29 ± 0.00
150	15.43± 0.00

B6. Lead in SNC at varying concentrations

Concentration (mg/l)	Mean ± SD
10	9.30± 0.17
20	19.60 ± 0.00
30	29.16 ± 0.00
40	38.36 ± 0.00
150	46.25 ± 0.00

B7. Lead in PKC at varying masses

Mass (g)	Mean ± SD
2	77.76± 0.00
4	78.87 ± 0.00

6	83.52 ± 0.00
8	89.45 ± 0.00
10	95.06 ± 0.00

B8. Lead in SNC at varying masses

Mass (g)	Mean ± SD
2	83.18 ± 0.00
4	96.16 ± 0.00
6	98.29 ± 0.00
8	98.79 ± 0.00
10	99.13 ± 0.00

C. STATISTICAL ANALYSIS

C1. Arsenic in PKC and SNC at varying concentration

Column A PKC
Vs Vs
Column B SNC

Unpaired t test with Welch's correction

0.4834

P value summary Ns

Are means signif. different? (P < 0.05) No

One- or two-tailed P value? Two-tailed

Welch-corrected t, df t=0.74 df=7

How big is the difference?

Mean ± SEM of column A 14 ± 4.5 N=5

Mean ± SEM of column B 20 ± 5.7 N=5

Difference between means -5.4 ± 7.3

95% confidence interval -23 to 12

R squared 0.073

F test to compare variances

F,DFn, Dfd 1.6, 4, 4

P value 0.6521

P value summary Ns

Are variances significantly different? No

C2. Arsenic in PKC and SNC at varying masses

Table Analyzed

Data 1

Column A

Vs
Column B

Unpaired t test with Welch's correction

P value 0.0018
P value summary **
Are means signif. different? ($P < 0.05$) Yes
One- or two-tailed P value? Two-tailed
Welch-corrected t, df $t=5.290$ df=6

How big is the difference?

Mean \pm SEM of column A 41.96 ± 2.396 N=5
Mean \pm SEM of column B 65.38 ± 3.723 N=5
Difference between means -23.42 ± 4.427
95% confidence interval -34.25 to -12.59
R squared 0.8234

F test to compare variances

F,DFn, Dfd 2.415, 4, 4
P value 0.4140
P value summary Ns
Are variances significantly different? No

C3. Lead in PKC and SNC at varying concentrations

Table Analyzed Data 1
Column A
Vs
Column B

Unpaired t test with Welch's correction

P value 0.0837
P value summary Ns

Are means signif. different? ($P < 0.05$)	No
One- or two-tailed P value?	Two-tailed
Welch-corrected t, df	$t=2.291$ $df=4$

How big is the difference?

Mean \pm SEM of column A	13.04 ± 1.635 $N=5$
Mean \pm SEM of column B	28.53 ± 6.560 $N=5$
Difference between means	-15.49 ± 6.760
95% confidence interval	-34.26 to 3.276
R squared	0.5676

F test to compare variances

F,DFn, Dfd	16.10, 4, 4
P value	0.0197
P value summary	*
Are variances significantly different?	Yes

C4. Lead in PKC and SNC at varying masses

Table Analyzed	Data 1
Column A	
Vs	Vs
Column B	

Unpaired t test with Welch's correction

P value	0.0562
P value summary	Ns
Are means signif. different? ($P < 0.05$)	No
One- or two-tailed P value?	Two-tailed
Welch-corrected t, df	$t=2.285$ $df=7$

How big is the difference?

Mean \pm SEM of column A 84.97 ± 3.243 N=5

Mean \pm SEM of column B 95.11 ± 3.027 N=5

Difference between means -10.14 ± 4.436

95% confidence interval -20.63 to 0.3533

R squared 0.4273

F test to compare variances

F,DFn, Dfd $1.148, 4, 4$

P value 0.8969

P value summary ns

Are variances significantly different? No



Oil palm plantation



Ripped oil palm fruit



Transverse section of oil palm fruit



palm kernel cake



Shea plantation



Ripped shea nut fruit



Transverse section of shea nut fruit



Shea nut cake

