IDENTIFICATION OF ELEMENTAL COMPOSITION OF PALM KERNEL SHELL

BY

YAKUBU HAMZAT

(2007/2/27744EH)

DEPARTMENT OF CHEMICAL ENGINEERING

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

NIGERIA

NOVEMBER, 2011

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A PROJECT SUBMITTED TO THE

DEPARTMENT OF CHEMICAL ENGINEERING,

FEDERAL UNIVERSITY OF TECHNOLOGY,MINNA

NIGERIA

IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF

BACHELOR OF ENGINEERING (B. ENG) DEGREE IN CHEMICAL

ENGINEERING

NOVEMBER, 2011

DECLARATION

I declare that the work in the project report entitled identification of elemental composition of palm kernel shell has been carried out by me under the supervision of Dr D. O. Agbajelola. No part of this project report was presented for another degree or diploma elsewhere at any institution to the best of my knowledge.

Hamzat Yakubu		
Student Name	Signature	Date

CERTIFICATION

This is to certify that this project report entitled IDENTIFICATION OF ELEMENTAL COMPOSITION OF PALM KERNEL SHELL by Hamzat Yakubu meets the requirements for the partial fulfilments of the award of Bachelor of engineering (B.Eng) degree in Chemical Engineering, Federal University of Technology, Minna.

.... 05/12/11 ....

Dr D. O. AGBAJELOLA
Project Supervisor

DATE

....

Engr. Dr. M. O. EDOGA
Head of department

DATE

....

External Examiner

DATE

DEDICATION

This research work is dedicated to the Almighty Allah for His guidance and protection and to the memory of my late mother.

ACKNOWLEDGEMENT

In the name of Allah the most merciful.For blessing and giving me strength and ability to complete this project. Firstly, I would like to acknowledge my deepest gratitude to my supervisor Engr. Dr. D.O Agbajelola for his constant attention and valuable suggestion enthusiastic support and understanding. Furthermore, my appreciation goes to Dr Ndamitso Mohammed and Nasiru Yahaya Saraki for his support and personal concern during the research. Furthermore, my appreciation also goes to my entire family for their prayer and support. I would also extend my sincere appreciation to my brothers Mohammed Raji, Nma, Aliyu, Nmandako, and others too numerous to mention. My deepest gratitude goes to U.U Alkali, Manmasun, Yahaya, Ibrahim, Ahmed, Bello, Nmanda, Kudu, Suleman, and Mustapha Idrisu, Faruk. My deepest appreciation also goes to Ahmed, Yahaya, Ibrahim and Musty. Special appreciation goes to my brothers and friends Umar, Yahaya, Ahmed, Ibrahim Mustapha who has always on my side, riding along with me on my ups and downs as well as given me encouragement to pursue my dream. Enormous appreciation to Mallam Umar Musa, Mallam A.D. Abdullah, Abdulmalik Mohammed for supporting me academically and always give me strength who always be there for me through my thick and thin and always love me. Not to forget my brothers. To my dear friends Kabir Danladi, Isah Dubagari, Abdulhafiz, Kure, Bashiru, and all the members in school of Engineering. Thanks for making my stayed in Minna so colourful and enjoyable; the memory of your friendship will forever stay inside my heart. My Allah blesses all of us and only He, the Almighty Allah could repay all debts to all of you.Wassalamualaikun warahmatullah wabarak.

ABSTRACT

This research work involved the identification of important elemental composition of palm kernel shell. The palm kernel shell was burned into the ashed in the furnace at the temperature of 700 ^{o}C for four hours twenty minutes. Precipitation method was used for the identification of each element present in the ashed palm kernel shell. The elements determined qualitatively were calcium, nickel, zinc, copper, iron, manganese, barium, lead, potassium and beryllium. Quantitatively, the concentrations of the constituent element in the sample were determined using Atomic Absorption Spectrometer (AAS) and from the results obtained the composition of alkali metals Ca, Cr, Mn, Fe, Cu, Zn, Ni, Pb, Mg and K were obtained after different runs of experimentation. The potassium (K), calcium (Ca) and magnesium (Mg) contents of the sample were generally higher than those of other elements. The concentration of potassium which was the highest was 78.1038 mg/l while that of manganese which was the lowest was 0.0011 mg/l.

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Chapter One

1.0 INTRODUCTION

The profession of Chemical Engineering has to do with the technology of the Chemical and process industries. Thus, the conversion of most useless materials in to highly demanding useful products is the aim of Chemical Engineer. For instance, as a result of technological advancement and research, waste in our society regarded useless which to some extent might sometimes cause environmental pollution. Example, kernel shell which is a waste from agricultural product. Palm kernel shell are the byproducts obtained from plant, it is obtain after the palm oil and nut have been carefully removed or used. These shells can serve as a raw material for many industrial processes because of its elemental composition, hence the project topic: identification of elemental composition in palm kernel shell (Bradshaw, 1993).

Palm kernel shell is a virgin biomass with high calorific value. It has a very low ash and sulphur content.

It is generally believed the no part of a palm tree is useless. After the processing of the fresh fruit to yield palm oil, the product that are left is the shell palm nut which can be cracked to yield the palm kernel nut and the shell. These shells can be used as fuel, road construction, raw material for potassium production, fertilizer production and in the manufacture of car brake pads.

Palm kernel shell is mostly used as Sources of fertilizer because the main ingredients are calcium, iron, magnesium, and potassium. Large amount of these material are present in palm kernel shell hence it is used as manure and raw material for fertilizer industries (Kreppler, 2006).

Palm kernel shell can be considered like a natural pellet and a high grade solid renewable fuel for burning, it is usually blended with other grade of biomass like wood chips. It is also used as a raw material for car brakes. It could help in providing raw material for soap manufactures. It could be used as a base for potassium production since it contains higher percentage composition of potassium. The potassium and other trace element contained in the shell could serve as a raw material in pharmaceutical (Akinyeye *et al*, 2011).

Palm kernel cakes are the leftovers after kernel oil is pressed out from the nut in the palm fruit. Palm kernel cake is commonly used as animal feed for dairy cattle because of its high protein content. If not, it is usually treated as biomass to fuel up boilers to generate electricity for use at palm oil mills and surrounding villages (Aletor, 1999).

There are two types of palm kernel cake, depending on the process to get it either through mechanical or solvent process. Mechanical extraction by screw press is the most widely used. The solvent extraction process is not generally used because it is more expensive. The incombustible residue is known as "Ash" potash an oxide of the element potassium (K) and other trace elements can be obtained from the ashed, leached, decanted and evaporated solution of palm kernel shell (Aletor, 1999).

1.1 Aim and Objectives

The aim of this research is to identify the elemental composition of agricultural waste which is palm kernel shell. To achieve these, a study was carried out with the following objectives:

i. To evaluate various operating parameters such as ashing temperature/time for palm kernel shell.

ii. To determine the concentration of various elements present in the ashed palm kernel shell produced (i.e.) elemental composition analysis.

1.2 Scope of Work

The scope of this study is to use qualitative analysis to identify the constituent that are contain in ashed palm kernel and to use quantitative analysis to determine the concentration of each components present in the ash palm kernel shell.

1.3 Justification of the Project

The importance of palm kernel shell cannot be overemphasized due to the fact that it contains many chemical compositions which could be of benefit to the man and society. Some of these component include Potassium, calcium, Barium, Iron, Nickel, Copper, Manganese, Vanadium and Zinc.

Chapter Two

2.0 LITERATURE REVIEW

2.1 Ashed Palm Kernel Shell

Ashed palm kernels form a large and important class of porous solids, which have found a wide range of technological applications. As a consequence, the porous structures of these materials and their adsorption of vapours, and liquids have been extensively studied. In this section the micro-structural and porous properties of the principal classes of ashed palm kernel shell are reviewed. It is outside the scope of this contribution to consider in detail the very many industrial applications and processes that employ ashing of carbonated materials. Ashed palm kernel shells have been explained in different ways. On the other hand, some of the principal methods used to characterize the pore structure in ashed palm kernel shell are outlined. Furthermore, the pore structure is having been reviewed. In order to understand the porous structure of ashed palm kernel shell, it is first necessary to give an outline review of the carbonization processes that convert the organic precursors to the carbon product. This provides a basis for understanding the relationship of the pore structure of activated carbons to the fine structure of the solid carbon materials. An appreciation of the fine structure of PKS leads to an account of the surface forces in pores that give rise to the powerful adsorptive properties of activated carbons. The processing method involved two types of analysis, physical and chemical analysis. The development of porosity in carbons by "physical", i.e., by reaction of the carbon with oxidizing fluid (steam, carbon-dioxide and air) is reviewed. Chemical analysis i.e. by reaction *of* carbon precursors with reagents, such as phosphoric acid and zinc chloride is considered. The review continues with palm kernel shell and some applications that summarize the salient points to emerge from the review (Guo and Lua, 2003).

Ashed Palm Kernel Shell (APKSC) as many known as a solid, porous, black carbonaceous material and tasteless. Marsh (1989) defined APKS as a porous carbon material, usually chars, which have been subjected to reaction with gases during or after carbonization order to increase porosity. APKS is distinguished from elemental composition and the oxidation of the carbon surface. While according to Norlia Baharun, (1999) APKS is an organic material that has an essentially graphitic structure. The main features common to all APKS are; graphite like planes which show varying degrees of disorientation and the resulting spaces between these which constitute porosity, and the unit built of condensed aromatic rings are referred to as Basic Structure Units (BSU) also stated that APKS is predominantly an amorphous solid with a large internal surface area and pore volume. Cokes, chars and activated carbon are frequently termed amorphous carbon (Norlia, 1999).

X-ray studies have shown that many so-called amorphous substances have crystalline characteristics, even though they may not show certain features, such as crystal angles and faces, usually associated with crystalline state. Although interpretation of the X-ray diffraction patterns is not free from ambiguities, there is general agreement that amorphous carbon consists of plates in which the carbon atoms are arranged in a hexagonal lattice, each atom, except those at the edge, being held by covalent linkages to three other carbon atoms. The crystallites are formed by two or more of these plates being stacked one above the other. Although these crystallites have some structural resemblance to a larger graphite crystal, differences other than size exist (Hassler, 1974).

From all the definition, it can be summarized that APKS is black, amorphous solid containing major portion of fixed carbon content and other materials such as ash, water vapour and volatile matters in smaller percentage. Besides that, APKS also contain physical characteristic such as internal surface area and pore volume. The large surface area results in a high capacity for absorbing chemicals from gases or liquids. The adsorptive property stems from the extensive internal pore structure that develops during the activation process (Guo and Lua, 2003).

2.2 The Palm Tree

Palm trees are a family of plants called Arecaceae. Most of them are trees but some are shrubs. They grow in hot climates although some have been planted as far north as Britain. Some of the well known palm trees include Date palm and Coconut palm but over two thousand kinds of this plant are found in many kinds of places ranging from rainforests to the deserts (Hartley, 1988).

Most palms have a straight, unbranched stem, but sometimes a branching stem, or even a creeping vine such as the rattan. They have large evergreen leaves that are either 'fan leaved' or 'feather-leaved' arranged in a spiral at the top of the trunk. The leaves have a tubular sheath at the base that usually splits open on one side when it is growing. The flowers grow on an inflorescence, a special branch just for carrying the great number of tiny flowers. The flowers are generally small and white, and are star-shaped. The sepals and petals usually number three each. The fruit is usually a single seed surrounded by flesh and some kinds may contain two or more seeds in each fruit. The wax palm, the tallest palm in the world, reaches heights of 70 meters (Hartley, 1988).

Palms are one of the most well-known and widely planted tree families. Palms first appear in the fossil record around 80 million years ago, during the late Cretaceous Period. Some kinds from that period are still to be seen today, such as the nipa palm or mangrove palm. They have played many important roles on humans producing many common products and foods apart from the fact that they are also used in a lot of parks and gardens in areas which do not have heavy frosts. Historically, these plants have been symbols of victory, peace and fertility and up until today, they remain a popular symbol for the tropics and vacations (Kreppler, 2006).

Over two-thirds of palms are found in tropical forests, where some species grow tall enough to form part of the canopy while other shorter ones form part of the understory. Some kinds form pure stands in areas with poor drainage or regular flooding. Other palms live on tropical mountains above 1000 meters, Palms may also live in grasslands and scrublands, usually where there is water and in desert oases. A few ones are adapted to extremely basic lime soils while others are similarly adapted to very acidic soils. They are also found growing alongside streams running through the deserts (Manocha, 2003).

Like many other plants, some palms are in danger of dying out because of human activity. The greatest dangers are from increasingly large cities, mining, and turning forests into farmland. The harvesting of heart of palm for food also poses a threat, because it comes from the inner core of the tree, and harvesting it kills the tree. The use of rattan palms in furniture has caused a big fall in the number of rattan palms, as they are collected from the wild instead of being farmed. The sale of wild seeds to growers and collectors is another threat. It is very hard to save palm seeds because they are killed by cooling them which is the normal way of keeping rare seeds for the future. Also, planting rare kinds in parks can never truly recreate the wild areas from where they come and they may not do well in these parks. Many plants and trees like olive, soybean, canola, sunflower and coconut palm produce one type of oil. The fruits of the oil palm tree, however, yield two distinct oils - palm oil and palm kernel oil (Zeven, 2000).

Oil palms were introduced by java to Malaysia (then the British Colony of Malaya) in 1910 by Scotsman William Simi and English banker Henry Darby. The first plantation companies remained listed in London until the Malaysian government engineered their "Malaysianisation" throughout the 1960 s and 1970 s. Federal Land Development Authority is the world's biggest oil palm planter with planted area close to 900,000 hectares in Malaysia and Indonesia. Felda was formed on July 1st, 1956 when the land Development Act came into force with the main aim of eradicating poverty. Settlers were each allocated 10 acres of land (about 4 hectares) planted either with oil palm or rubber, or given 20 years to pay off the debt for the land (Dutch, 1848).

After Malaysia achieved independence in 1957, the government focused on value adding of rubber planting, boosting exports, and alleviating poverty through land schemes. In the 1960s and 1970 s, the government encouraged planting of other crops, to cushion the economy when world prices of tin and rubber plunged. Rubber estates gave way to oil palm plantations. In 1961, Felda's first palm oil settlement opened with 3.75 km^2 of land (Scotsman, 1987).

2.2.1 The Products of Palm Tree

2.2.1.1 Palm Kernel Cakes

These are the leftovers after kernel oil is pressed out from the nut in the palm fruit. Palm kernel cake is commonly used as animal feed for dairy cattle because of its high protein content. If not, it is usually treated as biomass to fuel up boilers to generate electricity for use at palm oil mills and surrounding villages.

There are two types of palm kernel cake, depending on the process to get it either through mechanical or solvent process. In Malaysia, mechanical extraction by screw press is the most widely used. The solvent extraction process is generally not used because it is more expensive.

The incombustible residue is known as "Ash" potash an oxide of the element potassium (K) and other trace elements can be obtained from the ashed, leached, decanted and evaporated solution of palm kernel shell (Yang, 2003).

2.3 Factors Affecting Ashed Palm Kernel Shells Production

2.3.1. Raw Material

Most organic materials rich in carbon that do not fuse upon carbonization can be used as raw material for the manufacture of AC (Rodriguez-Reinoso, 2002). The selection of raw material for preparation of porous carbon, several factors are taken into consideration.

The factors are:

i. High carbon content

ii. Low in inorganic content (i.e. low ash)

iii. High density and sufficient volatile content

iv. The stability of supply in the countries

v. Potential extent of activation

vi. Inexpensive material

vii. Low degradation upon storage

Lignocelluloses materials constitute the more commonly used precursor and account for around 45 % of the total raw materials used for the manufacture of potassium and other elements. Low content in organic materials is important to produce potassium and other element with low ash content, but relatively high volatile content is also needed for the control of the manufacturing process. Raw material such as coconut shell and fruit stones are very popular for many types of AC, because their relatively high density, hardness and volatile content are ideal for manufacture of hard granular potassium , calcium and other element. Coconut shells, together with peach and olive stones are used commercially for the production of micro porous products, useful for a very wide range of applications (Alimon, 2004).

2.3.2. Temperature

Temperature, particularly the final ashing temperature, affects the characteristic of the product produced. Generally, for commercial end product usually conducted at temperature above 800 °C in a mixture of steam.

(Miguel *et al.*, 2003). Recently, the researchers have been working out on optimizing the final ashing temperature to economize the cost of production and time. As reported by several authors, Ashing temperature significantly affects the production yield of APKS and also the surface area of products. The temperature used as low as 200 °C (Haimour and Emeish, 2005) and up high to 1100 °C (Miguel, *et al.*, 2003). The optimum temperatures have been reported to be between 400 °C to 500 °C by most the earlier researchers irrespective of the time of activation and impregnation ratio for different raw material (Srinivasakannan and Zailani, 2003). The increasing of ashing temperature reduces the yield of the elements continuously. According to Guo and Lua (2003), this is expected since an increasing amount of volatiles is released at increasing temperature from 500 °C to 900 °C. The decreasing trend in yield is paralleled by the increasing ashing temperature due to the activation reaction. These phenomena are also manifested in the decreasing volatile content and increasing fixed carbon for increasing ashing temperature. Previously, Haimour and Emeish (2005) suggested that the percentage of volatile matter decreased with an increased of carbonization temperature and the variation of this parameter was at maximum between 200 °C and 800 °C due to rapid carbonization occurring in this region. It is also unsuitable to prepare activated carbon when carbonization temperature was more than 800 °C since the successive decreased in volatile matter is minimal above this range.

This was accompanied with an increased of fixed carbon and ash content which may be attributed to the removal of volatile matter in the material during carbonization process. Thus, leaving behind the more stable carbon and ash-forming mineral (Haimour and Emeish, 2005). Another notable feature that showed the effect of ashing temperature on the APKS properties is the BET surface area. As the ashing temperature increased, the BET surface area also increased

(Haimour and Emeish, 2005). This may be attributed to the development of new pores as a result of volatile matter released and the widening of existing ones as the ashing temperature become higher.

2.3.3 Ashing Time

Besides ashing temperature, the ashing time also affects the carbonization process and properties of PKS. From previous study, the ashing times normally used were from 1 hour to 3 hour for palm shell and coconut shell (Srinivasakannan and Zailani, 2003). As the time increased, the percentage of yield decreased gradually and the BET surface area also increased. This result is possibly due to the volatilization of organic materials from raw material, which results in formation of ashed PKS. The extent of decrease in product yield is observed to be reducing when excessive activation occurs (Kim *et al.*, 2001).

2.4 Uses of Palm Kernel

Palm kernel oil is thick, reddish oil derived from the kernels of palm trees. Native to Africa, the use of palm oil has spread beyond the continent's borders.

1. Africans have used palm kernel oil for centuries. The applications vary, from cooking and frying oil to the manufacture of soap.
2. Palm kernel oil is rather thick or semi-solid and can last for long periods of time. Because of this, it is used as a substitute for cacao, or fats in milk.
3. Palm kernel oil is used in biscuits, cakes and certain types of bread to give them a softer texture and sweeter taste as well as making certain kinds of margarines.
4. Palm kernel oil was successfully manufactured as biodiesel fuel for vehicle, years after inventor and engineer Rudolf Diesel (1858 to 1913)--inventor of the diesel engine--used vegetable oil for his car.
5. Palm kernel oil is used to manufacture items such as detergents, candles, cosmetics, several types of greases, glue and printing inks.
6. Due to high calorific value of palm kernel shell, this commodity has been one of the key biomass material in order to replace fossil fuel for steam power plant, however due to more and more study conducted, it value has been elevated.

7. Carbonize Palm Kernel Shell can be use as charcoal which can be pressed into bio-fuel briquette, these form of charcoal could be directly sell to consumer for family use. Part of the carbonize Palm Kernel Shell can also be processed into activated carbon which is used in liquid and gaseous phase filtration or adsorption.

Although palm kernel cake supplies both protein and energy, it is looked upon more as a source of protein. Palm kernel cake is a reasonably good economic feed for cattle, both for fattening and supplementary feeding. It has effectively reduced the cost of milk production in Malaysia because it is a substitute of costly imported feedstuffs like soybean meal and ground maize.

3.1.6 Identification of Ni^{2+}

To the solution of group IV metal ions drop of 1.5M NH_3 drop wise solution were added to the portion till the solution was basic, then another 2 drops of Dimenthylgloxime (DMG) was added. A bright red precipitate confirmed the presence of Ni^{2+}.

3.1.7 Identification of Ca^{2+}

To the solution of group metal ions drop of 1.5M NH_3 solution was added till the solution is basic, then 0.5cm^3 saturated $(NH_4)_2$ C_2O_4 (Ammonium oxalate) was also added and heated in a boiling water bath for 5 minutes to increase particles size, then the solution was allowed to cool to room temperature. A white precipitate of calcium oxalate (CaC_2O_4) confirmed the presence of Ca^{2+}.

3.2 The List of Equipment / Apparatus

Table 3.1 List of Equipment and Apparatus used in the Experiment

S/n	Equipments	Source	Remark
1	Thermometers	Technico Ltd,Germany	0-100, glass
2	Beakers	Technico Ltd, Germany	0-250 ml, Pyrex
3	Conical flask	Technico Ltd, Germany	0-250 ml, Pyrex
4	Measuring cylinder	Vuline Ltd, England	0-100 ml, Pyrex
5	Weighing balance,	OHAUS, England	Digital
6	Hot plate	Technico Ltd. England	Electronic
7	Mortar and pestle	Bosso Market, Minna	Wood
8	Magnetic stirrer	Gallen Kamp, England	500 rpm, electronic
9	Crucible	Bida market, Niger	25-50 g
10	Centrifuge	Technico Ltd, England	Electronic
11	Dropping pipette	Technico Ltd, England	25 ml, pyrex
12	Filter paper	Whatman, England	125 mm, paper
13	Volumetric flask	Technico Ltd, England	0-250 ml, Pyrex
14	Furnance	Gallen Kamp, Egland	750-1200 ^{0}C,2.5 KW, electronic
15	Atomic Absorption Spectrometer	Zeal, England	Cathode lamp, electronic

3.3 The List of Chemicals Used are Presented Below

Table 3.2 List of Chemicals Used in the Experiment

S/n	Chemicals	Source	Remark
1	Hydrogen sulphide	BDH limited, Britain	Analytical reagent, 0.5 M
2	Ammonium sulphide	BDH limited, Britain	Analytical reagent, 6 M
3	Ammonium carbonate	BDH limited, Britain	Analytical reagent, 1.5 M
4	Hydrogen chloride	BDH limited, Britain	Analytical reagent, 6 M
5	Silver Nitrate	BDH limited, Britain	Analytical reagent, 1.5 M
6	Ethanol	BDH limited, Britain	Analytical reagent, 1.5 M
7	Calcium oxale	BDH limited, Britain	Analytical reagent, 1.5 M
8	Dimethylaloxine	BDH limited,Britain	Analytical reagent,1.5 M
9	Sodium bismuthate	BDH limited,Britain	Analytical reagent, 1.5 M

3.4 Experimental Work

3.4.1 Sample Collection and Treatment

The palm kernel shell is the by-product obtained from plant, it is obtained after the useful palm oil has been removed and used. The palm kernels were purchased from Market in Bida, Niger State and sun dried for 48 hours in order to reduce their moisture content. They were cracked and the shells were then stored in dried waterproof polyethene bags for further use.

3.4.2 Analysis of Sample

For each analyte of interest, samples were taken and analyzed according to the standard methods of the Analytical Chemist and some other standard procedures.

3.4.3 Drying

Drying is a physical separation process where an objective is the removal of liquid phase from a solid phase by means of thermal energy. The net wet of palm kernel shell of 120 g was dried to constant weight of 100 g. The drying was done in the sun for three (3) days to reduce the moisture content of the palm kernel shell and prepare the shell for ashing process. Proper drying enhances ashing. The average ambient temperature at this period is approximately 35 °C.

The drying rate is rapid as the moisture content was negligible.

3.4.4 Crushing

In crushing, the weighed sample of dried palm kernel shell (PKS) was crushed (Breaking into smaller pieces) with mortar to produce size integration i.e. reducing their total surface area and making them easily peaceable in the crucible ready for ashing.

3.4.5 Weighing

The weight of the sample (PKS) to be processed was taken using an electronic weighing balance for sensitivity purpose.

3.4.6 Ashing

Ashing implies the burning of combustible material at controlled temperature in limited supply of oxygen. The dried (PKS) sample was ashed in a furnace at a constant temperature for 260 minutes and the resultants (end product).

3.5 Experimental Procedure

50 g of ashed (PKS) was weighed and poured in 1 litre beaker containing 800 cm^3 of boiled distilled water. The resulting mixture was boiled using the hot-plate with magnetic stirrer at 100 °C with maximum stirring for one hour. It was then allowed to settle overnight. The residue obtained from the filtration the second day was added to solid residue left in the beaker after settling. It was found that the sample contains several elements. The sample was further analyzed to determine the contained element present.

3.6 Determination of Concentration of Elements in Palm Kernel Shell Using Atomic Absorption Spectrometer

One gram of the sample was weighed into a digestion flask. 5 ml of digestion mixture (nitric acid) was introduced in to the flask. The sample was digested for three hours at 200-250 ^{0}C and filtered in to 100 ml standard volumetric flask with whatman No.1 quantitative circle 125 mm filter paper. The filtrate was made up to the mark with distilled water.

Since each element has a characteristics wave length that will be absorbed, the specific hollow cathode lamps were selected accordingly. The slit width of each element was also identified. The sample was aspirated in to the flame; the elements present in the sample absorbed some of the light hence reducing the intensity of the light. The computer data system converts the change in intensity of light into an absorbance which is directly proportional to the concentration of the elements present in the sample.

The concentration of elements present in the sample was determined from the standard calibration curve for various elements present.

Chapter Four

4.0 RESULT AND DISCUSSION

4.1 Results

Table 4.1: Identification of Components in Palm Kernel Shell (PKS) Sample

S/N	Test	Observation	Inference
i.	Sample + 1.5 M NH_3, then in excess.	No precipitate was formed with excess NH_3 (aq.)	Ca^{2+} and Ni^{2+} were confirmed.
ii.	Above sample + excess NH_3.	White gelatinous precipitate formed. Precipitate soluble in excess NH_3 (aq.)	Zn^{2+} was confirmed.
iii.	Sample + 1.5 M NH_3 then Heated.	Pale blue precipitate formed. Precipitate dissolved in excess NH_3 (aq.) to form a deep blue solution.	Cu^{2+} was confirmed.
iv.	Sample + Excess NH_3 +Heated.	Reddish brown precipitate formed. Precipitate insoluble in excess NH_3 (aq.)	Fe^{2+} confirmed.
v.	Sample + 6 M Acidified HNO_3 then followed by $AgNO_3$.	White precipitate formed. Precipitate dissolved in excess NH_3 (aq.) to form a clear solution.	Mn^{2+} present. Mn^{2+} confirmed.
vi.	Sample +1 cm^3 of ethanol, +2 drops of 6 M H_2SO_4.	Precipitate is formed.	Ba^{2+} confirmed.
vii.	Sample + Dilute HNO_3, then $AgNO_3$ + Heated gently.	A cream precipitate formed.	Br^- confirmed.

Table 4.2: Determination of Concentration of Elements in Sample

S/N	Elements	Concentration (mg/l)			Average conc. (mg/l)
		Run 1	Run 2	Run 3	
1	Ca	61.8856	60.4440	61.6487	61.3261
2	Cr	ND	ND	ND	ND
3	Mn	ND	ND	0.0033	0.0011
4	Fe	0.4706	0.4765	0.3941	0.4471
5	Cu	1.8900	2.0000	1.9000	1.9300
6	Zn	0.6216	0.5405	0.5784	0.5802
7	Ni	0.4000	0.2890	0.3000	0.3297
8	Pb	0.2374	0.1872	0.1749	0.1998
9	Mg	11.4548	12.4933	12.7300	12.2260
10	K	78.5200	78.0877	77.7037	78.1038

4.2 Discussion of Results:

In Table 4.1 above, the result of identification of component minerals in the palm kernel shell (PKS) using ethanol, heat and dilute solutions of H_2SO_4, $AgNO_3$ and aqueous ammonia showed the presence of calcium, nickel, zinc, copper, iron and manganese. Barium and beryllium were also confirmed.

In Table 4.2 above, the results of concentrations of various elements in the sample showed that the concentrations (mg/l) of potassium (K) and calcium (Ca) were relatively higher in the three (runs) than the concentrations of other trace elements. Calcium (Ca) is a soft silvery-grey metal with melting point of 815 °C and a boiling point of 1487 °C. It has a density of 1.55 g/cm^3 and it is a good conductor of heat and electricity. It is malleable and ductile and a good metal used in the extraction of uranium. Calcium is used as a deoxidant in steel castings and copper alloy and specifically in the manufacture of calcium fluoride and calcium hydride. This element is also used as a dehydrating agent in the preparation of pure ethanol and removal of impurities from petroleum. From the values obtained in this work one can say that the raw material can serve as a good source of calcium that can be used for the purposes enumerated above thus converting the undesirable or waste palm shell to a desirable end product using economically viable principles.

Potassium is an alkali metal which is strongly electropositive and ionizes to form univalent positive ions by the loss of the only one electron in the outermost shell. The conductivity of potassium is reduced if impurities are present. At low temperature, the conductivity is high, because the atom vibrates very little and there is no resistance. Potassium is a good reducing agent. Being potential donor of electrons, its ions give characteristic flame colours which are used to identify it. The relative reactivities of the potassium are reflected by their reactions with water. Thus it reacts explosively with water.

The result in the table above reflects the high composition of alkali metals in the waste material analyzed hence, it can serve as good raw material in our manufacturing companies. Magnesium (Mg) also had a high concentration in the sample. Magnesium is used in preparing tough alloys such as duralumin and magnalium. It is also used in photographic flash bulbs, military flames and fireworks. Copper (Cu) level in the sample was also appreciable which was indicative of the fact that this waste material could be used as a source of iron which is used in making wires and cables for conducting electricity because of its high electrical conductivity. It is also used to make water pipes and boilers because of its resistance to chemical attack. However, iron (Fe) contains many impurities like phosphorous, sulphur, silicon and manganese which lower the melting point of iron from 1530 ^{0}C to about 1200 ^{0}C. This impure iron is hard and brittle and at 1200 ^{0}C it can be melted and used to produce castings which do not need machining and can be used to produce materials which do not bear much strain such as kitchen stoves, drain pipes, Bunsen burners as well as radiators. The result of this analysis also indicated the presence of zinc which is used for galvanizing iron and steel to prevent rusting. It is also used in making dry cells and alloys such as brass and coinage bronze. Zinc is also used for roofing and as a reducing agent. Table 4.2 also showed the concentration of lead in the palm kernel shell (PKS). Lead is used as roof flashings and for tanks and living vessels to store acids. It is also used for making bullets, lead shots and weights because of its high density. In general, however, the results of this analysis showed that the manganese (Mn) content of the sample was low. From the results also, the concentrations of iron (Fe), zinc (Zn), nickel (Ni) and lead (Pb) were relatively low showing that the palm kernel shell is probably not a good source of these metals. Also, from the results, chromium (Cr) was not determined in the analysis which showed that the palm kernel shell is probably not a good source of this metal either.

Chapter Five

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the result of chemical analysis of palm kernel shell it can be concluded that these waste material which are discarded after the removal of palm kernel for the oil industry can serve as a good source of potassium (K), calcium (Ca) and magnesium (Mg), hence serving as a good source of potassium for the soap industry and calcium serve for the dental industry or company. Therefore it is suggested that instead of discarding this material it should be exploited for other industrial purposes.

5.2 Recommendation:

For further research work on this subject matter, the following recommendations are made.

1. Other methods of solid-liquid extraction such as steam distillation should be used to compare the yields in the concentrations of analytes of interest.
2. Other schemes should be drawn in order to determine and compare the quantities of these analytes in natural deposits and other materials like the palm kernel
3. The use of the ashing period (for more than 3 hrs) or increase in the ashing temperature (greater than 700 ^{o}C) should be investigated to find the limit of operating conditions.
4. Measures should be taken in order to convert the raffinate from the ashed palm kernel into desired and profitable products so as to avoid wastage.

Finally, further work should be on the effects of ashing time in the production of potassium hydroxide (KOH) from palm kernel shell.

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Chapter Three

3.0 METHODOLOGY

3.1 Identification of Elements Present

3.1.1 Identification of Cu^{2+}

To the solution of group II metal ions, drops of 1.5 M NH_3 solution were added till basic (tested with litmus). Five (5) more drops were added and a deep blue colour of Cu^{2+} $(NH_3)_4$ confirmed the presence of Cu^{2+}.

3.1. 2 Identification of Fe^{3+}

To the solution of group III the group metal ions, 1cm^3 of 2M NH_3 and 2 drops of 0.2M Potassium thiocyanate (KSCN) solution were added and a deep red colour confirmed Fe^{3+}.

3.1.3 Identification of Cr^{3+}

To the solution of group III 1cm^{3}of 0.3 M $BaCl_2$ solutions was added and a creamy yellow precipitate of $BaCrO_4$ confirm the presence of Cr^{3+}. The solution centrifuged and decanted and the solution then dissolved the precipitate in 3 drops 6M HNO_3, a yellow solution of $Cr_2O_7^{2-}$ confirmed the presence of Cr^{3+}.

3.1.4 Identification of Zn^{2+}

The solution of group IV metal ions was boiled to remove any H_2O_2, checked to see that it is still slightly basic, and then 5 drops of H2/water was added. A white/grey precipitate of ZnS confirmed the presence of Zn^{2+}.

3.1.5 Identification of Mn^2To the solution of group IV metal ions of small amounts of Sodium bismuthate were added to one portion. The appearance of a purple colour due to the formation of MnO_4 confirmed the presence of Mn^{2+} (although the colour may take a while to appear).