OkPRODUCTION OF AFRICAN BLACK SOAP USING COCOA POD HUSK AND PALM KERNEL OIL AND IMPLICATION FOR ENTREPRENEURSHIP

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CERTIFICATION

This is to certify that this project work, titled Production of African Black soap Using Cocoa Pod husk and palm kernel oil and implication for Entrepreneurship, was carried out by **CHUKWUDUMEBI M. NDUKA** with matriculation number **PSC1808542** of the department of Chemistry, faculty of physical sciences, University of Benin, Benin City.

DEDICATION

This project work is dedicated to my Elder brother Mr. Stephen Ikechukwu Stefano Nduka for his continuous and unending love and support.

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ABSTRACT

Production of African Black soap using cocoa pod husk ash (alkali base) and palm kernel oil is essentially crude soap obtained from the process of Saponification. The cocoa pud husk were carbonized (burnt to ashes) and the Alkali was extracted from it by dissolving in water and later filtered. The suitability of the Cocoa Pod husk ash as an alkali source for soap production was evaluated, the Alkali extracted was made to react with hot palm kernel oil and the resultant mixture is the African Black soap. The soap formed was analysed. The values of the pH test, foamability test and Lather volume analysed were 9.0, 300ml and 6.33 minutes. The analysis however has revealed that the African black soap can compete favourably with other toilet soaps in the market and can also be improved on. So therefore, using cocoa pod husk for the production of the African black soap, can be modelled to preserve this age-old craft and

guarantee that future generations continue to use the traditional methods of creating soap which serve as job opportunities for local and modern communities and women's cooperatives.

CHAPTER ONE

1.1 INTRODUCTION

African black soap, sometimes referred to as African soap or black soap, is a traditional soap that comes from numerous West African nations, including Ghana, Nigeria, and Mali. Various African cultures have been using it for millennia for its cleaning and skincare benefits. Locally produced natural materials used in the soap's creation include plantain peels, cocoa beans, palm kernel oil, shea butter, and medicinal herbs. (Barker, Elizabeth, 2014).

The substances utilized gave the soap its distinctive black color and texture. Cocoa and plantain peels are dried in the sun, roasted, and finally burned to ash. A lye solution is created by combining the ash with water and filtering the mixture. According to the particular recipe, this solution is blended with oils like palm kernel oil and shea butter, as well as optional components like honey or essential oils (Boakye et al., 2018).

Heating the mixture causes it to thicken and solidify. The soap is made into bars or balls and allowed to cure before use. The natural and gentle washing qualities of African black soap are well known. All skin types can use it, and it is frequently used to treat a variety of skin issues such acne, eczema, psoriasis, and oily skin. The soap is thought to have antibacterial and antifungal qualities that efficiently cleanse the skin and remove pollutants without destroying natural oils. Additionally, it promotes exfoliation, lessens the visibility of scars, and enhances skin texture.

African black soap can exhibit variations in color, texture, and scent based on the specific recipe and ingredients used. Traditionally handmade black soap may have an earthy or smoky aroma.

Over time, African black soap has gained global popularity, leading to commercial production and variations that may incorporate additional ingredients or modifications to cater to different markets.

African black soap has cultural importance in various African communities, being regarded as a cherished beauty secret passed down over generations. It has also gained global recognition and popularity in the beauty and skincare industry as a natural and ethical substitute for mainstream soaps. ago by local women not only for effective cleansing but for maintaining healthy skin and hair. It is known by many names based on the local dialect of the area where it is produced. The recipe has not changed much since it was formulated. However, it has been constantly re-engineered to make it more suitable for use.

At Cocoa Research Institute of Ghana, the soap has been re-formulated by mixing the Potassium Hydroxide with either cocoa butter or shea butter to enhance its nourishing effects on the skin. Liquid soap has also been developed from Cocoa pod husk potash. Quality assessment conducted by the Ghana Standards Authority on both the solid and liquid soap made from Cocoa pod husk potash has revealed that they contain no free caustic alkali and have lather volumes ranging from 200-300 ml. Total fatty matter, which is an indication of good quality or free fatty acids in the soap, also ranged from 84-87%. Any soap with fatty matter above 70% is considered to be a good soap. The average pH of both the liquid and solid soaps made from Cocoa pod husk potash is 9.

However, pH of the skin is 4.7 [Ayeni, L.S.et Al, 2008) indicating that the skin is slightly acidic (Agbeniyi, S.O., K.A. 2016) Although soap made from Cocoa pod Husk potash is highly alkaline, it is found to be soft on the skin because potassium is known to keep skin cells hydrated and moisturized by absorbing water molecules from the atmosphere.

The pH value of 10 in the soap is an indication of the absence of free caustic alkali. Soap made from Cocoa Pod Husk potash and Potassium Hydroxide 7 mixed with cocoa butter or shea butter is unique in that it has the combined properties of all the ingredients used. It has been found to reduce skin inflammation and irritations such as acne, moisturize both dry and normal skin, and clear blemishes, spots and other skin diseases such as eczema and psoriasis. It also has anti-bacterial and anti-fungal properties. The potash used is also produced from a natural source, making the soap soft on both sensitive and normal skin. The soap also contains glycerin, which is a natural by-product of the soap making process. Unlike other commercial soaps, the glycerin produced is retained and this enhances the moisturizing effect of the soap. The natural ingredients used make the soap non-toxic to the environment because they are biodegradable, making the disposal of unwanted soap easy.

1.2. Other unique qualities and benefits of African Black soap.

- Natural Source of Potash: Cocoa pod husk ash contains high levels of potassium hydroxide, also known as potash. Potash is a crucial alkali used in the saponification process, which converts oils or fats into soap. The potassium hydroxide in cocoa pod husk ash helps break down the oils and fats and facilitates soap formation.

- Exfoliating Properties: Cocoa pod husk ash acts as a mild exfoliant due to its slightly abrasive texture. It helps remove dead skin cells, unclog pores, and promote smooth and healthy skin.

- Darkening Effect: Cocoa pod husk ash contributes to the characteristic dark brown or black color of African black soap. The ash contains natural pigments that add color to the soap, distinguishing it from other types of soaps.

- Palm Kernel Oil: Palm kernel oil is another essential ingredient in African black soap and is derived from the seeds of the oil palm tree. It is widely used in soap making due to the following reasons:

- High in Lauric Acid: Palm kernel oil is rich in lauric acid, a fatty acid that produces a rich lather and provides cleansing properties. Lauric acid helps remove dirt, oil, and impurities from the skin, making it an effective ingredient in soap formulations.

- Moisturizing and Nourishing: Palm kernel oil contains natural emollients that help moisturize and hydrate the skin. It can leave the skin feeling soft, supple, and well-nourished.

- Sustainable and Economical: Palm kernel oil is readily available in regions where oil palm trees are grown, such as various parts of Africa. It is a cost-effective ingredient, making it suitable for local soap production.

Overall, the use of cocoa pod husk ash and palm kernel oil in African black soap production reflects a combination of traditional knowledge, local ingredient availability, and their specific properties that contribute to the unique qualities and benefits of this culturally significant soap.(Mensah, E. A., 2016)

1.3 IMPLICATION FOR ENTREPRENEURSHIP

The production of African Black soap has significant implications for entrepreneurship, particularly in the context of West Africa where this traditional soap has its origins. Here are some key implications for entrepreneurs: (Boateng, S. O., & Moser, C. (2016)

Cultural Preservation: Opportunities for the preservation of traditional knowledge and practices are provided by the manufacturing and sale of African Black soap. To preserve this age-old craft and guarantee that future generations continue to use the traditional methods of creating soap, entrepreneurs can work with regional craftsmen and communities.

Natural and Sustainable Product: African Black soap is in line with the worldwide trend toward natural and sustainable products. Entrepreneurs can benefit from the rising demand for chemical-free, eco-friendly skincare.

Market Expansion: By exporting African Black soap, business owners can look into foreign markets. Due to its distinct cultural identity and all-natural composition, the soap has the potential to become an export-friendly niche product, boosting trade and economic opportunities.

Product diversification: In addition to the conventional soap bars, business owners can develop derivative goods like liquid African Black soap, skincare items, or spa and beauty lines. The product line can serve a wider spectrum of customers by being more diverse.

Economic empowerment: By creating employment opportunities and assisting women's cooperatives, which are frequently involved in the soap-making process, entrepreneurship in the manufacturing of African Black soap can benefit nearby communities. In order words entrepreneurship in Black soap production provides jobs and benefits local communities and women's cooperatives.

Quality Control: To guarantee consistency in product quality, business owners can put quality control procedures in place. This can improve the soap's standing and dependability in both domestic and foreign markets.

Marketing and branding: Powerful marketing and branding techniques can promote African Black soap as a high-end, authentically cultural product with environmental considerations. Conscious consumers may be drawn in by tales about its history and advantages (Djameh, G. I. A., & Wang, Y. (2017)

1.4. STATEMENT OF PROBLEM

The properties of soap are determined by the amount and composition of the fatty acids and the oil blend used. However some soap cause problem to the user such as irritation to the skin, bleaching on the skin, inefficient lathering etc. The research work is aimed at producing good quantity soap from ash extract of cocoa pod husk ash and palm kernel oil

1.4.1 JUSTIFICATION/RELEVANCE OF THE STUDY

According to research, African black soap has been created using alkalis derived from various agricultural waste and palm kernel oil. However, little is known about the manufacture of soap using cocoa pod husk ash as an alkali source and palm kernel oil. The soap made from Cocoa pod husk and palm kernel oil research work is also aimed at producing good quantity African black soap from the alkali obtained from cocoa pod husk ash and palm kernel oil.

1.4.2 SCOPE OF RESEARCH WORK

The scope of this work covers the preparation of African black soap from palm kernel oil and a local source of alkali (Cocoa Pod husk) to form black soap. Some natural additives were added to the process. Some characteristics of the soap were further determined such as foamability test, ph and efficiency in cleaning

1.4.3: AIMS AND OBJECTIVES

The aim of this research work is to produce African black soap using cocoa pod husk and palm kernel oil as the locally sourced materials and implication for entrepreneurship.

OBJECTIVES.

1. To locally source for cocoa pod husk as the agricultural waste material which serve as the alkali Base for the production of the African black soap

2. To determine whether or not the soap can compete with other toilet soap by carrying out some characterization e g pH test and foaming Efficiency

1.5 LITERATURE REVIEW

1. Cocoa Pod Husks

Cocoa (Theobroma cacao L) is an important and economic crop in developing countries. The production of cocoa bean in 2016-2017 was 4.7 million tonnes worldwide (Icco Quarterly bulletin of cocoa statistics. 2017) Cote D'Iviore, Ghana and Indonesia are the top three producers of cocoa beans, contributing to 67% of the global production. Large quantities of underexploited by-product, including, cocoa pod husk (CPH) and pulp, are generated by removing the beans from the cocoa pods (FAOSTAT.2015) Cocoa pod husk weighs about 75% of the of the whole fruit and it's the main by-product (Daud, Z., et al. 2013) (Adjin-Tetteh, M., et al., 2018) (Perez, E., et al.2015)

Figure 1: Fresh cocoa pod fruits with illustration showing the cocoa pod husk and cocoa bean.

After removal of the cocoa beans, CPH is usually discarded on the farm and can function as an organic fertiliser, a practice that adds organic matter to soil and enables the return of nutrients to the soil and their recycling to plant-available forms after decomposition (Syamsiro, M., et al.,2012) (Aboyeji, et Al, 2016) However, untreated CPH left on the soil surface may act as a source of inoculum for plant diseases such as black pod rot due to the presence of Phytophthora spp. [Yapo, B.M., et,, 2013.). Black pod rot causes an annual yield loss from 20% to 30% worldwide, while individual farms may suffer an annual yield loss from 30% to 90% (Acebo-Guerrero, Y., et 2011) Cocoa Pod Husk is under-exploited as a renewable resource that is rich in dietary fibre, lignin and bioactive antioxidants such as polyphenols (Yapo, B.M., et, 2013) Recovering these lignocellulosic fractions and bioactive compounds may lead into the development of a profitable commodity and subsequently this could bring revenue to farmers, thus promoting economic development (Omobuwajo OR, et al, 2011) Bioconversion of Cocoa Pod Husk to added-value products, such as biomaterials for food and non-food uses, it is also a potential approach to maintain the sustainability of cocoa production. The opportunity of valorising Cocoa Pod Husk towards added-value applications is enormous, given its high abundance and the fact that cocoa is mainly cultivated in developing countries. This review will evaluate existing low-value applications of Cocoa Pod Husk and the value-added potential of Cocoa pod Husk and its fractions in food applications based on Cocoa Pod Husks chemical composition

1.5.1 Chemical composition of cocoa pod husk

Cocoa pud Husk comprises the epicarp, mesocarp, sclerotic part and endocarp. Table 1 shows the chemical composition of an example of a CPH from Ghana. The CPH consists primarily of fibrous materials including 19.7-26.1% cellulose, 8.7-12.8% hemicellulose, 14-28% lignin and 6.0-12.6 % pectin. The epicarp is enriched with lignin, while the mesocarp contains mainly (~50%) cellulose and the endocarp is rich in pectic substances [Ugbogu OC, Onyeagba RA, Chigbu OA 2006]. Xylan, arabinoxylan and arabinan are the main hemicellulose in Cocoa Pod husk) that have been deduced from the high amount of isolable arabinose and xylose [Bashua OC 2017). Other hemicelluloses such as xyloglucans, galactomannans or (galacto) glucomannans can also be found in Cocoa Pod Husk [Gunathilake R 2009].

Lignin is a complex aromatic heteropolymer, made from phenylpropane units (p-coumaryl, coniferyl and sinapyl alcohols) and is strongly attached to cellulose and hemicellulose, providing rigidity to the plant cell wall [Aliyu MS, Hanwa UA1990) (Adelakun N. et al 2009) Condensed tannins have highly polymerized structures and could be bound to lignin in CPH with a dry weight content of 5.2%. Pectin that is associated with cellulose and hemicellulose is determined as uronic acids, contributing to 6.7-12.4% of Cocoa Pod Husk (Man MQ, Lin TK, Santiago JL, et al 2014). The ash content of CPH ranges from 6.4-8.4% w/w with a variety of minerals. Significantly high amounts of K (2.8-3.8% w/w) are observed, followed by Ca, Mg and P [Ugbogu OC, Onyeagba RA, Chigbu OA 2006]. Cocoa Pod Husk is also a source of phenolic acids, ranging from 4.6 to 6.9g GAE/100g.

Table 1: Chemical Composition of cocoa pod husk [9, 17-19]

Composition.		Amount (%, w/w, dry weight)
Protein.		7-10
Fat		1.5-2
Carbohydrates.		32-47
Cellulose.		19.7-26.1
Hemicellulose (Xylan & arabinoxylan)		8.7-12.8
Lignin.		14-28
Pectin.		6.0-12.6
Ash.		6.4-8.4
Minerals		
	K	2.8-3.8
	Ca.	0.25-0.46
	Mg.	0.11-0.25
	P.	0.19
	Na.	0.01-0.02
	Fe	0.003-0.006

Phenolic content (g GAE1/100g).   4.6-6.9

Gallic acid equivalent

1.5.2 Low value applications of Cocoa Pod Husk

1. Fertiliser and soil organic matter

A study in Nigeria has compared the gross margin of cocoa production between matter. The high mineral content of Cocoa pod Husk, particularly in K, Ca and P, offers the possibility for partially substituting conventional fertilisers, based on research from Nigeria and Ghana (Bella, O., 2011, African black soap). One study in Nigeria reported that combining Cocoa Pod Husk powder with basal phosphorus fertiliser could achieve similar plant quality, seed yield and harvest index of black benniseed cultivation, compared to NITROGEN, PHOSPHORUS AND POTASSIUM fertiliser (mixture of $(NH_4)_2SO_4$, P_2O_5 and K_2O) [Misra M, 1997]. Other studies have focused on the use of Cocoa Pod Hush ash as a fertiliser; replacing up to 50% conventional NITROGEN, PHOSPHORUS AND POTASSIUM fertiliser with CPH ash had a positive effect on grain yield and nutrients uptake in maize production and on fruit growth, yield and soil fertility in tomato Onifade KR. Onifade 1994]. Such eco-friendly applications could potentially replenish the shortage of expensive NITROGEN, PHOSPHORUS AND POTASSIUM fertiliser due to limited distribution and marketing for fertiliser procurement in developing countries such as Ghana. Burning Cocoa Pod Husk to produce ash might also have additional benefits in terms of improved farm sanitation and control of a potential source of inoculum for black pod rot disease [Nwoko V.O. (1980). To demonstrate the economic feasibility of using Cocoa pod husk fertiliser in cocoa farming,

2. Soap making

Cocoa pod Husk could also be a starting material for soap making in western African countries, through a process that includes steps of ashing, leaching, filtration and concentration, saponification, cooling and cutting. After burning CPH in air into an ash containing K_2O, the ashes were leached with water and correspondingly generated Cocoa Pod husk potash (potassium hydroxide, KOH), each tonne of fresh pod husk would generate about 6 kg potash]. The potash could be further filtrated, concentrated and used in saponification with oil to produce soaps (Ikotun AA, Ogundele OF 2017). CPH derived soaps have been successfully produced in pilot plant scale with detailed market research in Ghana and other African countries. Soaps made with Cocoa Pod Husk potash have great solubility, consistency, cleansing and lathering ability, compared to soaps made with chemical KOH (Ikotun AA, Olalere C, 2017). Results also showed that the commercialization of soap with Cocoa Pod Husk potash is highly feasible, and mainly driven by the demand for natural and less harsh toiletry products. As Cocoa Pod Husk is abundant and readily available in these countries, using natural raw materials could also ease the financial pressure of importing chemicals for developing countries. A market report in Ghana showed that 3 tonnes of liquid soap made by Cocoa pod Husk potash could bring the profit of 63,000 Ghanaian cedi. The successful usage of Cocoa pod Husk as a starting material for commercial soap making would also be an alternative way to generating additional income for farmers.

3. **Activated carbon**

Activated carbon may also be produced from Cocoa Pod Husk through physical or chemical activation. Carbonisation and activation involve physical/thermal processing. In a recent study, CPH was heat dried at 500^{o}C with a N2 flow (carbonisation), followed by applying a CO_2 flow at 650-850^{o}C for 30 min (activation). This process yielded 18-38% (w/w) of activated carbon with a surface area of 1.1 m2/g. However, pre-treatment of CPH with hydrochloric acid (HCl) significantly reduced the ash content of the CPH, resulting in the production of activated carbon with higher surface area (356 m2/g) using lower temperatures (650^{o}C vs 900oC) (Adewusi AO 2020) Chemical activation of CPH involves the usage of agents such as K_2CO_3, KOH and $ZnCl_2$ under nitrogen at temperatures of 500°C - 800°C, yielding 13.5-47.2% of activated carbon with a much larger surface area (780 m2/g) (Mishra N, Rajput PS 2017). The above-mentioned studies indicate that CPH could be utilised as an alternative source for activated carbon production, delivering a high surface area and high adsorption capacity given proper treatment. The low price of CPH, compared to traditional precursors such as anthracite, coal or peat, could enhance its economic potential with a reduction in overall production costs (Underwood, 2008)

Combustion of plant biomass into ashes

Ash is formed from mineral matter during the process of combustion and gasification. Plant biomasses are usually made up of cellulose which consists of 99.5 % combustible materials and 0.5 % of incombustible materials. The combustible materials are usually converted to water vapour and carbon dioxide whiles the incombustible minerals are converted to ashes. The ashes, however consist of 80 % water insoluble components whiles the 20 % water-soluble components are potash (K_2CO_3), arcanite (K_2SO_4) and soda ash (Na_2CO_3) with potassium carbonate (K_2CO_3) forming the major component (Thoburn, 2008). The combustion of agricultural wastes (plant biomass) causes the oxidation of mineral ions such as potassium to potassium oxide which then combines with the carbon dioxide resulting in the formation of K_2CO_3 (Kumar ,2013).

$4K(s) + O_2(g) \rightarrow 2K_2O(s)$ …………………………………. (1)

$K_2O(s) + CO_2(g) \rightarrow K_2CO_3(s)$ …………………………….. (2)

Effects of temperature on the yield of ash

(Babayemi et al. (2011)).observed that the incomplete combustion or ashing of plant biomass results in the formation of a blackish residual substance. (Onyegbado et al. (2002) in an attempt to produce potassium hydroxide resorted to drying of waste plantain peels and grinding of the dried peels into fine powder instead of combustion. The yield of KOH was insignificant. Kumar (2013) also studied the effect of the ashing temperature on the yield of the ash from the heating of the coffee husk in muffle furnace. The maximum yield of the ash obtained was 18 % between the temperature ranges of 400^oC to 500^oC. However, the yield of the ash decreased at temperatures above 500^oC. This was due to the fact that the coffee husk becomes completely combusted at temperatures above 500^oC oxidizing the carbon present to carbon dioxide. Subsequently, at temperatures beyond 700^oC the potassium metal salts and the other solid components present in the ash disintegrates into gaseous components. (Ofori-Boateng and Lee (2013); Kamalu and Oghome (2011) share the same view that the extent of combustion of plant materials into ashes had an influence on the quality of the potassium carbonate (K_2CO_3). This was observed in their attempt to produced caustic potash for the manufacturing of soap using cotton seed hulls. The yield of the potassium carbonate (K_2CO_3) was very minimum since a low temperature of ashing causes incomplete combustion leading to the formation of black residual particles which can impart negatively on the colour of the K_2CO_3 produced. This presupposes that the temperature at which plant biomass are combusted into ashes has an influence on the quality of the ash produced.

Elemental composition of ashes of plant biomass

Zekri and Obreza (2013); Schwarzkopf (1972) in their publications made known that the element potassium is one of the major nutrient that plays a very significant role in plants physiological processes such as protein synthesis, rate of respiration, enhancement of the transport of nutrients to the parts of the plant and enhances the thickening of the plant cell wall, helping plants to remain upright. Potassium (K) forms a major component of the ashes obtained from plant biomass such as cocoa husk, plantain peels, cassava peels and coffee husks, since potassium (K) is found in nature usually embedded in the earth crust. Kumar (2013), in his work analyzed the ashes from the combustion of coffee husk by Atomic Absorption Spectrophotometry (AAS) and found the presence of the elements: potassium, sodium, manganese, calcium and iron. The concentrations in terms of composition were 41.93 ppm, 5.48 ppm, 9.71 ppm, 13.75 ppm and 4.04 ppm respectively. Potassium was the highest accounting for about 71.5 % w/w. Onyegbado et al (2002), in their work on the production of soap using plantain peels as the source of alkali analyzed the presence of metallic ions by dissolving 0.15 kg of the plantain peels ash in 2.50 dm3 of deionized water at a temperature of 60^{o}C and then leaving the mixture to stand for about 8 hours. AtomicAbsorption Spectrophotometric (AAS) analysis of the filtrate of the mixture showed that potassium was the highest with a concentration of 126.1 ppm followed by sodium – 24.4 ppm, calcium – 1.04 ppm.

Caustification of potassium carbonate (K2CO3)

This involves a metathesis reaction between potassium carbonate, (K_2CO_3) and calcium hydroxide causing the precipitation of calcium carbonate ($CaCO_3$) which is filtered off as the residue to give potassium hydroxide (KOH) as the filtrate. This method can also be used to produce sodium hydroxide (NaOH) from sodium carbonate (Wikipedia, 2016).AE

Potassium hydroxide (KOH)

Potassium hydroxide (KOH) can be produced by the caustification of potassium carbonate (K_2CO_3) from the ashes of plant biomass (cocoa pod husks and plantain peels) and is the main product of interest in this study.

1.5.3 The nature and properties of potassium hydroxide (KOH)

Potassium hydroxide (KOH), commonly known as caustic potash, lye or potassium hydrate is an inorganic chemical compound which has the chemical formula KOH, a molecular weight of 56.11 g/mol and a melting temperature of 380°C. It is odourless, usually appears as a yellowish white lumpy solid in the solid state and colourless when in the aqueous state and is usually hygroscopic in nature (Patnaik, 2002). It has a refractive index of 1.409 at 20°C. Potassium hydroxide is a strong base, has a bitter taste, a slippery or soapy feel between the fingers and turns red litmus paper to blue (Kamalu and Oghome, 2011). Potassium hydroxide has a pH of 13.0 - 13.5 (0.60 %) and undergoes complete dissociation in an aqueous solution:

$$KOH\ (aq) \rightarrow K+(aq) + OH-(aq)$$

Potassium hydroxide has a density as well as relative density of 2044 kg/m3 and 2.0 respectively at a temperature of 20^oC (LabChem, 2012). Potassium hydroxide is not a flammable substance but when in contact with reactive metals can generate the flammable hydrogen gas (comet, 2014)

1.5.4 Importance and uses of potassium hydroxide (KOH)

Potassium hydroxide (KOH) is an important chemical substances that has numerous uses as a result of its characteristic property as a strong base and its ability to degrade substances. It is a very vital resource used in the petroleum industry for the refining of petroleum fractions, for the production of soaps and detergents by the soap manufacturing industries, an active component in the manufacturing of fertilizers, a dehydrating agent for drying gases, as a stabilizer in most of the synthetic lubricants, serves as electrolyte in alkaline batteries because it is more conductive (OxyChem, 2000). Potassium hydroxide is a caustic reagent that is used widely to neutralize acids and prepare potassium salts of reagents. It is used in the mercerizing of cotton, electroplating, photoengraving and lithography as well as the analysis of bone and cartilage samples by histology (Sigma, 2003). Potassium hydroxide is used as a food additive in the processing of food to prolong the shelf-life. Potassium hydroxide is used to correct the acidity of soils and also it is used in the manufacturing of fungicides and herbicides. In veterinary medicine it is employed in disbudding the horns of calves (Wasserman, 2013).

1.5.4 Chemical reactions of potassium hydroxide (KOH)

Potassium hydroxide reacts very vigorously with many organic and inorganic substances.

1.5.4.1 Reaction as a base

Potassium hydroxide is a base which forms an alkaline solution when dissolved in water and other polar solvents. It reacts with acids in neutralization reactions to produce salt and water:

$$KOH\ (aq) + HCl\ (aq) \rightarrow KCl\ (aq) + H_2O\ (l)$$

1.5.4.2 Saponification of ester

Potassium hydroxide hydrolysis a carboxylic acid ester to form a carboxylic acid salt and an alcohol (Olabanji et al., 2012) and in order to convert the salt back to the corresponding carboxylic acid, an acid hydrolysis is required. An example of a saponification reaction under basic condition is shown below:

$$KOH + CH_3CO_2Et \rightarrow EtOH + CH_3CO_2K$$

1.5.4.3 Reaction of KOH with carbon dioxide to form bicarbonates

Potassium hydroxide (KOH) reacts with carbon dioxide to form bicarbonates:

$$KOH\ (aq) + CO_2\ (g) \rightarrow KHCO_3\ (aq).$$

1.5.4.4 Transesterification reaction

Potassium hydroxide is used as a catalyst in the transesterification of vegetable oils for the production of biodiesel (Anastopoulos et al., 2009).

1.5.4.5 Hazards involved in the usage of potassium hydroxide (KOH)

Potassium hydroxide is a corrosive substance when concentrated and on contact with water generates a high amount of heat energy which may be sufficient to cause the ignition of some nearby materials which are capable of undergoing combustion and can cause severe skin burns, permanent eye injury and can cause irritation to the respiratory tract if inhaled and also when ingested can cause gastrointestinal burns (Comet, 2014). However, potassium hydroxide is not a carcinogenic substance since there has been no report of such incident but prolonged exposure to the dilute solutions may have damaging effects on the tissues of the body and can be fatal.

Potassium hydroxide is very toxic to aquatic organisms when disposed of indiscriminately into water bodies. This is because when it dissociates in the water, it elevates the pH of the water making aquatic life unpleasant for its inhabitants (OxyChem, 2013).

1.5.4.6 Safety precautions for usage of potassium hydroxide (KOH)

It is very prudent to be careful when using potassium hydroxide (KOH) by observing basic safety precautions which includes the wearing of protective clothing such as lab coat or an apron, nose mask to avoid inhalation of the fumes, the use of eye protective goggles is also very much recommended to protect the eyes in case of any possible splash in the cause of usage not forgetting the wearing of gloves as well as safety boot or foot wear to protect the feet. However, in the circumstance where there is a splash of caustic potash on one’s eyes, it is recommended that the victim flood the eyes with lots of water continuous for about 15 minutes after which medical attention must be sought for (OxyChem, 2013).

1.5.5 Storage materials for potassium hydroxide (KOH)

According to Daniyan et al. (2014), the materials for the storage of KOH should not be corroded by KOH. The use of mild steel tank as storage vessel is recommended, since potassium hydroxide does reacts with the iron in steel , the appropriate material for storage should be a tank made from PVC, polyethene, polypropylene and must be stored at ambient temperature. Storage materials made from copper, aluminum, zinc, bronze, brass, etc. are readily attacked by potassium hydroxide solution and are therefore not suitable for use as storage vessels for potassium hydroxide. Sigma (2003), one of the leading potassium hydroxide manufacturing companies , in their Material Safety Data Sheet do not recommend the use of glass materials as storage vessel since potassium hydroxide solution does each glass over a period of just a few days of storage in a glass vessel. LabChem (2012), in their Material Safety Data Sheet made known that potassium hydroxide must , however , be kept in a tightly closed container, stored in a cool, dry, ventilated area since it is deliquescent. Potassium hydroxide must be isolated from incompatible substances to protect it against physical damage. Potassium hydroxide on contact with water releases heat which can result in violent boiling and spattering and so must be protected from moisture.

Soap

Soap is a surfactant used together with water for washing, bathing, and cleaning that is available in solid bars and in the form of viscous liquid (Willcox, 2000). In a chemical term, soap is referred to as the salt product of a fatty acid (Stroebinger et al., 2021). It is equally used as an agent of textile spinning and is a key component in lubricants. Soap is generally obtainable by reacting vegetable/animal oil and fat with alkaline solution like NaOH, KOH or soda ash (sodium bicarbonate), (Willcox, 2000). The aqueous alkaline solution, is usually termed as lye, and give rise to saponification reaction as presented below (Figure 2). In saponification reaction, triglyceride fats are hydrolyzed at the beginning to give free fatty acids, which later react with the aqueous alkali to yield crude soap (Willcox, 2000).

$$(C_{17}H_{35})_3C_3H_5 + 3NaOH \rightarrow 3C_{17}H_{35}COONa + C_3H_5(OH)_3$$

Fat **soap.** **Glycerol**

Figure 2: Saponification reaction

The precise origin of soap remains obscure, although the Romans reported that it started about 600B C, when Phoenicians prepared it from goats tallow and wood ash (Jimoh and Jimoh, 2021). Soap was used by Celts, from Britain. Soap was used widely throughout the Roman Empire. Soap was not identified as a cleanser until second century A. D. By the eight century, soap was common in Italy, France, and Spain, but it was rarely used in the rest of Europe until as the 17th century (Jimoh and Jimoh, 2021).

Soap is made up of the following reagents:

- **Fats and Oils:** These are the primary ingredients and can be sourced from animals or plants, like tallow, palm oil, or coconut oil. They consist of glycerol and fatty acids.
- **Lye**: Either sodium hydroxide (for solid soap) or potassium hydroxide (for liquid soap) is a critical component. It reacts with fats and oils in a process called saponification, transforming them into soap and glycerin.
- **Water**: Water is essential for dissolving and diluting the lye, making it easier to mix and homogenize the soap mixture.
- **Additives**: Soap formulations may include various additives such as fragrance, colorants, exfoliants, moisturizers, and preservatives to achieve desired properties and characteristics (Haslinger, W. (2015).)

Soap is classified based on the nature of oil employed in its production. Based on this, soap can be classified into drying (manufactured using drying oils like cotton seed oil, coconut oil, Palm Kernel Oil etc.) and non-drying soap manufactured in the form of liquid using non-drying oils. Sometimes drying oils can equally be used (Gupta, 1990).

Methods of manufacturing soap

Soap is produced using either cold processes or hot process. In the cold process, soap is made by reacting fatty acid and NaOH or KOH together. The acid can generally be any oil. Cold process of soap making combines both art and science into action. The shortened form of this process is that there is a certain ratio of lye (NaOH) and water to fatty acid that forms the reaction known as saponification.

In the course of saponification, the oil and lye solution mixed to produce soap. It takes this whole process not less than six weeks to complete. The following safety equipment like hand gloves, goggles, nose mask etc are required while using cold process. Soap produced using this process are known for their hardness and their quality depends on the type of oil used (Garzena, 2004; Willcox, 2000; Jimoh and Jimoh, 2021). While, in the hot process the whole materials used for the soap making are added together in a pot or any container on which heat is applied like stove. The sample on the fire is stirred frequently until the whole stages of soap making are undergone. The excess water is dried off and the soap is cooled before use (Garzena, 2004; Willcox, 2000; Jimoh and Jimoh, 2021).

The cleansing property of soap is defined by its polar and non-polar structures together with principle of solubility, the hydrocarbon part is non-polar and hydrophobic while, the salt is ionic and hydrophilic as shown in the structure below

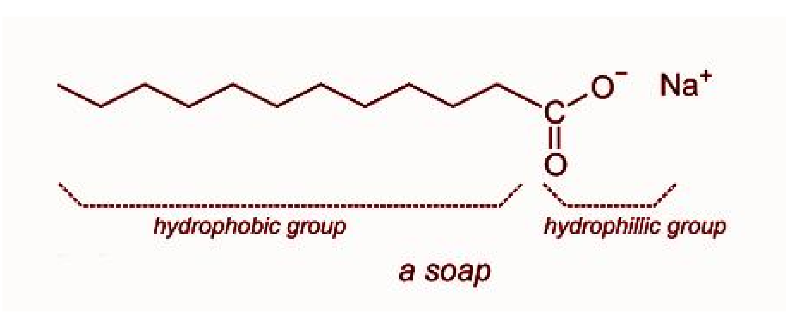

Figure 2: Soap Structure

When oil (that is, a non-polar hydrocarbon) is mixed with a soap solution, the soap molecule works as an intermediate between water (polar) molecules and non-polar molecule. This is because, soap molecule has both polar and non polar properties and therefore acts as an emulsifier. An emulsifier has the ability of diffusing one liquid to another immiscible liquid (David, Encyclopedia Britannica "Soap and Detergent" 2009).

Palm Kernel Oil

Palm Kernel Oil is extracted from oil palm fruits(from its kernel). It is in the lauric oils family just like coconut oil. Meaning, it possess a high concentration of lauric acid, which is a saturated fatty acid. Initially, the oil is yellowish-brown in color, but after purification becomes white-yellowish fat. Palm Kernel Oilis solid at normaltemperature, but at body temperature it melts rapidly with a nice cooling effect.This is the reason why it is commonly found in ice confection, ice cream coatings, cocoa glazes, and cool-melting confectionery fillings. It is equally used in margarine production.Palm Kernel Oilis the primary raw material for making intermediate materials that are used in cosmetics and cleansing (Liu et al., 2019).

Table 1: Approximate percentages of individual fatty acids in Palm Kernel Oil.

Acids	Number of Carbon.	Percentage(%)
Lauric saturated.	C_{12}	48.2
Myristicsaturated	C_{14}	16.2
Palmiticsaturated	C_{16}	8.4
Capricsaturated	C_{10}	3.4
Caprylicsaturated	C_{8}	3.3
Stearicsaturated	C_{18}	2.5
Oleicmonounsaturated	$C_{18:2}$	15.3
Linoleicpolyunsaturated	C_{18}	2.3

(Jimoh and Jimoh, 2021).

The Properties of soaps are determined by the amount and composition of the components fatty acid in the starting fat mixture. The usefulness of fatty acids in soap making are limited by the chain length and degree of unsaturation.

Generally, chain length of less than 12-carbon atoms produce undesirable soaps because their soaps are irritating to the skin. Conversely, saturated chain lengths greater than 18-carbon atoms form soap too insoluble for ready solution. Also too, a large proportion of unsaturated fatty acids yeilds soaps susceptible to undesirable atmospheric oxidation change. The most important soaps are the sodium and potassium salts of fatty acids. The harder sodium salts are commonly used.

PROPERTIES OF SOAP

- It serves as a surface-active agent, capable of reducing water's surface tension.
- Soap molecules have both water-attracting and water-repellent components, making them amphiphilic.
- When soap is introduced into water, it forms micelles, where the hydrophobic tails group together internally, while the hydrophilic heads interact with the surrounding water.
- Soap's ability to emulsify hydrophobic substances, like oils and dirt, results from its hydrophobic tails surrounding these substances, allowing them to be suspended in water.
- Soap's cleaning action relies on its capacity to surround and dislodge dirt and oils from surfaces, making them easy to wash away when rinsed with water (Adamson, A. W., & Gast, A. P. (1997).)

HYDROLYSIS OF SOAP

The term "hydrolysis" is applied to any resolution of a body into it's constituents where the decomposition is brought about by the action of water, hence when soap is treated with cold water, it is said to undergo hydrolysis, the reaction taking place being represented in it's simplest form by the equation;

$2C_{17}H_{35}COONa + H_2O$ ——-------$2C_{17}H_{35}COOH + 2NaOH$.

The actual reaction which occurs has been subject of investigation by many Chemist and very diverse conclusions have been arrived at. Chevreaul, the pioneer in the modern chemistry of oils and fats found that a small amount of alkali was liberated, as appears in the above equation, together with the formulation of an acid salt (Katz, D.A. 2000)

The extent of dissociation occuring when a soap is dissolved in water depends upon the nature of the fatty acids from which the soap is made and also on the concentration of the solution. The Hydrolytic action of water on soap is affected. Also very considerably, by the presence of certain substance dissolved in the water particularly salts of calcium and magnesium. (Warra et Al., (2010)).

CLEANSING ACTION OF SOAP

The cleansing action of soap is a result of its surfactant properties. Soaps are substances that lower the surface tension of water by forming unique structures in aqueous solutions. These soap molecules have two distinct parts: a water-repellent tail and a water-attracting head. When soap is mixed with water, these molecules come together to form micelles.

Inside these micelles, the hydrophobic tails of soap molecules gather in the center, while the hydrophilic heads point outward, interacting with the surrounding water. This micellar structure enables soap to envelop and disperse hydrophobic substances like oil and dirt, which do not readily mix with water. As a result, these oily particles are effectively trapped within the micelles, making it possible to wash them away when rinsed with water. (Rosen, M. J. (2004)).

POST-SAPONIFICATION PROCESS

- Neutralization: To ensure the soap is gentle on the skin, any excess alkali (sodium hydroxide or potassium hydroxide) left from saponification is neutralized using an acid like citric acid or borax.

- Additives and Fragrance: Fragrances, colorants, and other additives are introduced at this stage to enhance the soap's scent, appearance, and properties. They are mixed into the soap base for desired characteristics.

- Aging: Some soaps benefit from a resting period, typically several weeks, to improve their hardness, longevity, and quality. This is known as aging.

- Cutting and Shaping: Solid soap is poured into molds, allowed to solidify, removed, and cut into bars or shapes. Liquid soap is prepared for dispensing.

- Packaging: The finished soap products are packaged for sale, using materials such as wrappers, boxes, or bottles, depending on the type of soap and its intended use. (Smith, R. L., & Montgomery, L. F. (2019))

TYPES OF SOAPS

- Bar Soap: The classic solid soap used for general cleansing.

- Liquid Soap: A convenient option available in various formulations and container types.

- Glycerin Soap: Transparent soap with added glycerin to retain skin moisture.

- Castile Soap: Made from vegetable oils, known for its mild properties.

- Antibacterial Soap: Contains active antibacterial agents, though regular soap is often recommended for handwashing.
- Medicated Soap: Designed to treat specific skin conditions like acne or fungal infections.
- Exfoliating Soap: Contains abrasive particles for removing dead skin cells.
- Herbal or Natural Soap: Made from natural ingredients, often free from synthetic additives.
- Baby Soap: Gentle and mild, formulated for delicate infant skin with a lower pH (Lambers, et Al (2006))

CHAPTER TWO

2.0 MATERIALS AND METHODS

2.1 Materials and Reagents

- Cocoa pod husk
- Palm kernel oil
- Shell butter
- Distilled water
- 1M Hydrochloric acid
- 0.5M Hydrochloric acid
- 4% Ethanoic potassium hydroxide
- Phenolphthalein indicator

2.2 APPARATUS USED

- Filter paper
- Conical flask
- Measuring Cylinder
- Weighing Balance
- Stirrer
- Thermometer

- Hot plate
- pH Meter
- Burette
- Pipette
- Retort stand
- Sand bath
- Round bottom flask
- Beakers
- Oven
- Muffle Furnace
- Mortal and pestle
- Stop watch
- Test tubes
- Reflux condenser

2.3. Methodology

2.3.1 Sample collection

Cocoa pod fruits, palm kernel oil and shell butter were purchased from New Benin market in Benin city, Edo state. Nigeria.

2.3.2 Sample Preparation

The cocoa pod husk were sun-dried for 5-7 days and was pounded with the mortal and pestle so as to increase the surface area. The pounded cocoa pod husk were further oven dried using the oven dryer. The dried pounded cocoa pod husk was carbonized (burnt to ash) using the muffle Furnace at a temperature of about 750°C. The ash sample was allowed to cool and packed in a rubber for storage.

2.3.3 Extraction of Alkaline Solution from the carbonized sample

Distilled water was added to the ash in a plastic rubber without measuring, after stirring for 20 minutes, the mixture was left to settle for 3 days. After settling, the mixture was filtered using the filter paper and plastic funnel to obtain the Alkali Solution

2.3.4 Preparation of African Black Soap

Soap making is very versatile because of its wide demand and so are the Methods. In choosing materials and methods in this work, consideration is made of various methods already carried out in research. (E.G. Olumayede and B.F. Adeosun (2008). R.N. Shreve (1967).

Saponification Procedure

100ml of the extract (Alkali) was added to a 250ml beaker containing 30g of thoroughly heated palm kernel oil. The mixture was then heated using a sand bath with stirring occasionally into the hot soap solution. Calculated amount of Shell butter was added with continuous stiring of the soap mixture. The heating continued for about 40 minutes until a homogeneous soap solution was obtained. The Soap formed was allowed to cool into a solid mass which was collected as black soap.

2.7. DETERMINATION OF THE CONCENTRATION OF POTASSIUM HYDROXIDE IN THE ALKALINE SOLUTION USING TITRATION

Materials used for the determination of potassium hydroxide in the alkaline solution

- Sample (Alkaline Solution)h
- 1M conc, Hydrochloric acid
- Phenolphthalein indicator

- Filter paper
- Titration apparatus
- Burette
- Pipette
- Tripod stand
- Beaker
- Plastic funnel

Preparation of 1M concentration of Hydrochloric acid:

1M concentration of Hydrochloric acid was prepared by diluting 16.6ml 12M hydrochloric acid in 190ml distilled water and an additional distilled water was added to the mark to obtain 200ml 1M concentration hydrochloric acid.

Titration

50ml 1M concentration of hydrochloric acid was filled in the burette to the meniscus, 2 drops of Phenolphthalein indicator was added to 10ml of the alkaline solution in a beaker and titrated against the hydrochloric acid. The Titration continues until a white colour or clear solution was seen, indicating an end point for the titration. The titration was repeated 5 extra times and an average Value was obtained.

CALCULATIONS

Calculating for volume of acid:

$$C V = C V$$

$$12 \times V = 1 \times 200$$

$$V = \frac{120}{12} = 8.33ml$$

2.8 DETERMINATION OF SAPONIFICATION VALUE

Saponification value is defined as the number of milligrams of potassium hydroxide required to completely hydrolysed (saponify) one gram of the oil/fat. In practice a known amount of the oil or fat is refluxed with excess amount of standard alcoholic solution and the unused Alkali is titrated against a standard acid (O. P. Agarwal; Advanced Practical Organic Chemistry; Page No. 275)

Requirements:

Sample:

Oil/fat

Apparatus:

Conical flask

Reflux and condenser water bath

Burette and pipette

Chemicals:

0.5N hydrochloric acid

Phenolphthalein indication

0.5N alcoholic Potassium hydroxide

1. Preparation of 4% Ethanoic potassium hydroxide solution.

1.67g of potassium hydroxide pellets was weighed and taken into a round bottom flask. 200ml of ethanol was measured and poured in the same flask, a reflux condenser was attached to the flask, heated and refluxed for 30 mins.

10ml of ethanol was then collected. Potassium hydroxide pellets were taken and crushed to fine powder without measuring. 6g of the grinded Potassium hydroxide pellets was weighed, the potassium hydroxide pellets was then transferred into a flask to which 4% Ethanolic potassium hydroxide solution was prepared. 150ml of ethanol was measured and collected from the distillation. The ethanol was later poured in a flask containing potassium hydroxide pellets, it was mixed well and kept in a cold water to regulate the temperature.

2. Preparation of 0.5N HCL

0.5N Hydrochloric acid was prepared by diluting 4.1ml of 12M hydrochloric acid in 100ml distilled water and an additional distilled water was added to the mark to obtain 100ml 0.5N hydrochloric acid.

3. TITRATION FOR SAMPLE

1g of palm kernel oil sample was measured and poured in a sample flask, 50ml of 4% Ethanoic potassium hydroxide was measured and added in the sample flask. A condenser was attached with the sample flask and reflux for 30minutes. During the process of refluxing, the cold water supply was kept on and running.

After refluxing, 2 drops of phenolphthalein indicator was added into the solution while still hot and shaken properly for proper mixture. On addition of the phenolphthalein, the solution turned dark brown. 0.5N HCl was poured in the burette to the meniscus point titration of the solution with phenolphthalein was done against 0.5N HCl while still hot. The titration continues until the disappearance of the dark brown colouration which indicates the end point (Low et al., 2023).

4. TITRATION FOR BLANK

50ml of 4% Ethanolic potassium hydroxide was measured and poured in a sample flask. A condenser was attached with the sample flask and reflux for 30minutes. During the process of refluxing, the cold water supply was kept on and running.

After refluxing, 2 drops of phenolphthalein indicator was added into the solution and shaken properly for proper mixture. On addition of the phenolphthalein, the solution turned Dark brown. 0.5N HCl was poured in the burette to the meniscus point, titration of the solution with phenolphthalein was done against 0.5N HCl while still hot, until the disappearance of the dark colouration which indicates the end point.

5. CALCULATION FOR SAPONIFICATION VALUE

Saponification value = $\frac{56 X 0.5 (V - X)}{W}$

V (Volume of acid used for blank) = Final burette reading – Initial burette reading.

X (Volume of acid used for sample) = Final burette reading – Initial burette reading.

W = weight of sample

2. pH Determination

2g of the finished soap was dissolved in 10ml distilled water and stirred till sample dissolved. The pH was determined with pH meter, the electrode of the pH meter was inserted into the solution. The pH reading was recorded. The step was repeated three times for accurate pH reading.

3. Lather Volume/Foamability Test

2.0g of the soap was added to a 500ml measuring Cylinder containing 100ml distilled water as reported by Isah (2006). The mixture was shaken vigorously so as to generate forms, after shaking for about 2 minutes, the cylinder was allowed to stand for about 10 minutes, the volume of the foam or lather was measured and recorded.

4. Time for Lather to subside

The soap produced was used to form lather in water and the time taken for the lather to subside was determined using a stopwatch.

CHAPTER THREE

3.0 RESULTS AND DISCUSSIONS

3.1 RESULTS

TABLE 3.1: Determination of Potassium hydroxide in the Alkaline solution

No of readings	Initial	Final	Final - Initial
1st	27.5	45.5	18
2nd	30.5	48.4	17.9
3rd	29.6	48.5	18.9
4th	25.0	44	19.0
5th	26.0	45	19.0

$$\text{The mean} = \frac{18.9+19.0+19.0}{3}$$

$$= \frac{56.9}{3}$$

$$= 18.9 \sim 19\text{cm}$$

TABLE 3.2 TIME AND FOAMING EFFICIENCY

Number of reading	1st	2nd	3rd
Volume of lather formed (ml)	310	330	350
Time taken to subside (min)	6.05	6.30	6.40

Average volume of lather formed

$$= \frac{310+330+350}{3}$$

= 330ml

Average time taken for the form to subside

$$= \frac{6.05+6.30+6.40}{3}$$

$$= \frac{19.0}{3}$$

= 6.33mins

TABLE 3.3 pH DETERMINATION

Number of reading	1st	2nd	3rd
pH	9.57	9.88	9.91

The mean $= \frac{9.57+9.88+9.91}{3}$

$$= \frac{29.36}{3}$$

pH = 9.7

TABLE 3.4 Summarizes the results obtained from the analysis of the African Black soap

TABLE 3.4 SUMMARY ANALYSIS OF AFRICAN BLACK SOAP

PARAMETERS	COMPOSITION
pH	9.7
Lather Volume	330ml
Foam efficiency	6.33mins

3.2 DISCUSSION

Table 3.6 contains the analysis results of the African black soap from cocoa pod husk (alkali extract) and palm oil.

The foamability test carried out on the African Black soap showed that the African black soap has a moderate foaming stability and can compete favourably with other toilet soap.

From the result, the pH of the African black soap was 9.7 which is consistent with the normal pH range for soap 8 - 10.5. Soap being salt of strong base and weak acids should be weakly alkaline in aqueous solution. But Soap with pH range 11 - 14 can cause damage to the skin, hence pH of Soaps should be in the range of 8 - 10.5 as recommended by NAFDAC a regulatory agency in Nigerian. (Umar, 2002)

3.3 CONCLUSION

The above data on the African black soap shows that the African black soap being produced, can compete favourably with other toilet soaps, and the soap can be further improved upon by purification of the finished soap through the post saponification process. The forming efficiency can also be improved by addition of water softeners and scum dispersant, this addition plays an important role in lowering the surface tension of water. Therefore, using cocoa pod husk for the production of the African black soap, can be modelled to preserve this age-old craft and guarantee that future generations continue to use the traditional methods of creating soap which can serve as job opportunities for local and modern communities and women's cooperatives.

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