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PRODUCTION OF BLACK SOAP USING ALKALI SOURCE PLANTAIN PEEL BIOCHAR AND ASH

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Ash, Biochar, Black Soap, Characterization, Plantain Peel, Saponification, Shea butter

ABSTRACT
This study explores the characterization of alkaline extracts from plantain peel biochar and ash, alongside the analysis of Shea butter oil's physiochemical properties, and their application in black soap formulation. The plantain peel biochar extract exhibited a pH of 11.5 and a concentration of 0.14M, whereas the ash extract displayed a higher pH of 13.6 with a concentration of 0.21M. This difference in alkalinity was substantiated through titration results. A comparative physiochemical analysis of Shea butter oil was conducted against other oils like coconut, Aegyptian Balanite, and palm kernel oils, following FAO standards. Shea butter oil demonstrated a relative density of 0.91±0.01, saponification value of 180±1.2 mgKOH/g, %FFA of 9.61±0.23 mgKOH/g, and an acid value of 10.57±0.87 mgKOH/g. These results highlighted the unique properties of Shea butter oil, particularly in saponification and free fatty acid content. Furthermore, the study included a comparative analysis of black soaps formulated from plantain peel biochar (APPB) and ash (APPA). Both soaps adhered to FAO's acceptable pH range, with APPB at 8.70 and APPA at 9.93. The lathering ability of APPB soap was 720.00mL, and APPA soap was 703.00mL. The moisture content was 5.00% for APPB soap and 6.99% for APPA soap, indicating potential implications for shelf-life and microbial growth. The study's findings contribute valuable insights into the sustainable use of plantain waste products in soap production, emphasizing the potential for environmental and economic benefits. The physiochemical properties of Shea butter oil and plantain-derived extracts are crucial for the development of high-quality, environmentally friendly soaps, presenting a compelling case for further research and application in the soap industry.

1.0 INTRODUCTION

1.1 Background

Black soap, also known as African black soap or *alata samina,* is a traditional soap that has been used for centuries in various African countries (Igbashio *et al.,* 2022). It is renowned for its natural ingredients and numerous skin benefits. The soap is made from plantain peels, which are rich in essential nutrients and possess unique properties that make them ideal for skincare (Bernat *et al.,* 2021). The production of black soap from plantain peels involves a traditional and intricate process that harnesses the power of nature to create a gentle and effective cleansing product (Igbashio et al., 2022). More so, Soaps are typically fatty acid salts that can be firm or soft depending on the components utilized. They are made from fats/oils that have been hydrolyzed with an alkaline to produce fatty acid salts (soap) and glycerol as a by-product. This process is well known as saponification (Nnyia *et al.,* 2023). This soap, characterized by its dark color and natural ingredients, has gained international recognition for its unique properties and cultural significance (Goumbri *et al.,* 2021).

More so, the origins of black soap can be traced back centuries, with its production methods handed down through generations. The precise historical timeline of the black soap's creation is challenging to pinpoint, as it emerged organically within indigenous communities without extensive written records. However, it is believed to have been in use for several centuries, with oral traditions passing down the techniques involved in its production (Bernat *et al.,* 2021). It is crafted from locally sourced natural ingredients, reflecting the rich biodiversity of West African regions. The primary components include plantain skins which are heated to either become biochar or burn to ashes which serve as a derived alkali source. Also, palm kernel oil, cocoa pod ash, and Shea butter are other ingredients meticulously prepared and combined using traditional techniques, resulting in a bar of soap with distinctive properties.(Goumbri *et al.,* 2021).Moreover, it is globally recognition and its Commercialization graph hasn't plummeted since its advent. In recent years, black soap has gained popularity beyond West Africa, finding its way into international markets (Nnyia *et al.,* 2022). Its natural, eco-friendly, and chemical-free composition has resonated with consumers seeking sustainable skincare products. However, this increased demand has also led to challenges related to authenticity and quality control, emphasizing the need to support ethical and community-based production practices (Doris *et al.,* 2024).

In consideration of its cultural Significance which includes cleansing rituals, holding a significant cultural importance in various West African communities. It is used in daily cleansing rituals, serving as a symbol of purity and spiritual cleansing (Nnyia *et al.,* 2022). Its inclusion in traditional ceremonies, such as births, weddings, and coming-of-age rituals, underscores its role in cultural practices. Besides, its healing and medicinal properties make it renowned for its therapeutic benefits (Igbashio et al., 2022). It is believed to alleviate skin conditions like eczema, acne, and psoriasis due to its natural, non-irritating ingredients. Additionally, the high content of Shea butter provides moisturizing effects, making it an essential element in skincare routines (Okunola *et al.,* 2019).

In conclusion, black soap stands as a testament to the enduring cultural practices and indigenous knowledge of West African communities. Its history, deeply rooted in tradition, exemplifies the significance of natural resources and sustainable practices. As it continues to gain global recognition, it is essential to acknowledge and respect the cultural heritage and economic contributions associated with this remarkable soap. (Doris *et al.,* 2024).

1.2 Problem Statement

The production of black soap from plantain peels presents a unique opportunity to explore sustainable and eco-friendly alternatives in the skincare industry. Despite its potential benefits and growing popularity, there remains a lack of comprehensive research on the optimization of the soap-making process, the utilization of enzymes for saponification, and the evaluation of its performance in comparison to conventional commercial soaps. Addressing these knowledge gaps can further enhance the understanding and application of black soap as a natural and beneficial skincare product, contributing to advancements in chemical engineering and sustainable practices in the personal care industry (Igbashio *et al*., 2022).

1.3 Aim and Objectives

This study aims to produce black soap using alkali-derived Plantain Peel.

The specific objectives of this research are as follows:

1. Preparation and Characterization of the Plantain Peel biochar and Ash Extract.
2. Analyzing the physiochemical characterization of blended oil used in soap production.
3. Production of black soap using alkali-derived extract from the biochar and ash.
4. Analyzing the Physiochemical properties of the black soap.

2.1 METHODOLOGY

2.1.1 Extraction of Alkali from Plantain Peel Biochar (*PPB*)

Figure 3.1 reveals the step-step process involved in the extraction of *PPB* or *PPA*, strictly following Nnyia *et al.*, (2023) & Onyegbado *et al.*, (2002) protocols.

2.1.1.1 Preparation of Plantain Peel Biochar (*PPB*)

Adhering to Nnyia *et al.*, (2023) & Onyegbado *et al.*, (2002)s' protocols, to eliminate impurities, the fresh ripe plantain peels collected underwent a cleansing process using distilled water and were subsequently dried in an oven at 100°C for 2 days. The production of *PPB* involved cutting the plantain peels into small fragments and subjecting them to a 5-hour heating process in a furnace at 600°C. Afterward, the produced biochar was allowed to cool in a desiccator before being finely ground in a mortar and sifted through a mesh. Then it was divided into two equal parts labeled A and B.

2.1.1.2 Preparation of Plantain Peel Biochar Alkali Extract

100g of fine biochar powder labeled B was added to 200 mL of distilled water in a beaker. This blend was agitated and then left undisturbed for 72 hours at 60°C. Following this period, the alkaline solution was filtered using cotton wool and a funnel. Heat application was subsequently employed to concentrate the alkaline extract. Some parameters in Nnyia *et al.*, (2023) were adjusted.

2.1.2 Extraction of Alkali from Plantain Peel Ash

2.1.2.1 Preparation of Plantain Peel Ash (*PPA*)

To achieve a higher purity percentage of alkali, Onyegbado *et al.*, (2002)'s protocol was adhered to by subjecting the labeled A biochar to a higher temperature in an open combustion pan, heated until it ignited to become ash. The ashing lasted 3 hours. The ash sample was homogenized and crushed to achieve an average PSD of 0.106mm.

2.1.2.2 Preparation of *PPA* Alkali Extract

100g of the ash was leached in 200 distilled water and kept for 72 hours at 60°C. The solution was then filtered to have the extract collected. The pH of the two sample solutions was checked and recorded to determine the alkalinity of the potassium hydroxide solution.

2.1.3 Characterization of the *PPB* and Ash Extract

2.1.3.1 Determination of pH of *PPB* and *PPA* Extracts

pH meter was used to check the solution of both the alkaline extract from the biochar and the ash and were recorded.

2.1.3.2 Determination of the Concentration of the Extracts

Following the methodologies established by Onyegbado *et al.*, (2002), the concentration of the alkaline extract was measured by performing a titration with a 0.1M solution of hydrochloric acid (HCl) and using phenolphthalein as the indicator. The titration procedure was repeated twice to get an accurate result for both extracts. A series of volumes of acid used were recorded.

The equation of the reaction for which the experiment was based is shown in Eqn (3.1).

$$KOH_{(aq)} + HCl_{(aq)} \rightarrow KCl_{(aq)} + H_2O_{(aq)} \qquad 3.1$$

Eqn 3.2 was employed in the determination of the concentration of the extract.

$$C_aV_a = C_bV_b \qquad 3.2$$

According to Zauro *et al.*, (2019), the amount of KOH in the extract was calculated from equation (3.3).

$$V_{KOH} = \frac{FW_{KOH}N_{KOH}V_{ex}}{100ml} \qquad 3.3$$

2.2 Oil Blend Preparation and Characterization

2.2.1 Preparation of Oil Blend

The preparation of the oil blend began with selecting Shea butter oil and palm oil. 400g of palm oil and 200 kg of Shea butter oil, were mixed in an 80:20 weight ratio. This proportion aligns with those utilized by prominent Nigerian soap manufacturers like Unilever Plc and P.Z. Plc. A quantity of 30g from this oil mixture was warmed to 65 - 75°C and then kept for the saponification process (Onyegbado *et al.*, 2002).

2.2.2 Oil Bleaching Process

The bleaching process for the oil blend involved heating 100g of the oil to 50°C while stirring continuously. To this, 0.15 ml of 98% sulfuric acid was added to break down the oil's long molecular chains. Subsequently, 4 g of activated carbon was incorporated into the mixture, which was stirred for an additional ten minutes. A thermometer-equipped stopper was used, and the oil blend was heated to 85°C under a total pressure of 700 mm Hg, maintained using a vacuum pump for 30 minutes. After cooling to 80°C, the oil was neutralized with 0.05 g of sodium hydroxide (NaOH), then further cooled to 70°C, and finally filtered. Before saponification, other analyses conducted on the oil blend included determining its saponification value, titer value, percentage of impurities, and moisture content. These procedures are standard in oil processing and have been extensively described in other sources.

2.2.3 Physiochemical Characterization of Blended Oil

Characterizing the oil before saponification is crucial as it determines the quality and suitability of the oil for soap making, ensuring that the final product meets desired standards in terms of hardness, lather, and skin compatibility.

2.2.3.1 Determination of Relative Density of Oil

The density of the oil was estimated using this approach: First, 20 ml of the oil sample was measured and poured into a cylinder with a known weight. Then, the combined weight of the cylinder and the oil was recorded (Zauro *et al.*,2019). From these measurements, the density of the oil was calculated using the following formula:

$$\text{Relative Density of oil} = \frac{\text{weight of the sample (g)}}{\text{Volume of the sample (mL)}} \quad 3.4$$

2.2.3.2 Determination of Saponification Value

2g of the oil was measured and placed in a 300 ml conical flask. A 0.5 M solution of KOH was then added, and the mixture was heated to 55°C with continuous stirring. The temperature was subsequently increased to 100°C to complete the saponification process, allowing the mixture to boil for an hour. The excess KOH was titrated against the mixture using phenolphthalein as an indicator, and the Saponification value (SV) was calculated using the provided equation.

$$SV = \frac{\text{av.vol of KOH} \times 29.056}{\text{weight of the oil (g)}} \quad 3.5$$

2.2.3.3 Determination of free fatty acid (FFA)

Adhering to the protocol of Bello *et al.,* (2023), 2g of the oil sample was placed in a 250 mL conical flask and gently heated. Following that, 2.5 mL of methanol, two drops of phenolphthalein indicator, and a drop of 0.14M potassium hydroxide solution were added with thorough stirring. The mixture, containing the oil sample, was titrated against 0.14M potassium hydroxide solution while vigorously shaking until a consistent light pink color, lasting for 1 minute, was observed. The endpoint was then recorded, and the calculation of the Free Fatty Acid (FFA) value was performed using a specified equation (Afolabi, 2008).

$$\%\ FFA = \frac{\text{vol. of KOH(mL)} \times \text{Molarity of KOH} \times 28.2}{\text{Sample weight (g)}} \quad 3.6$$

2.2.3.4 Acid Value Determination

A 250 mL conical flask, previously dried, was used to hold 2.0 grams of oil sample. To this, 25 mL of absolute ethanol and 3 drops of phenolphthalein indicator were added. The mixture was then heated in a shaking water bath for 5 minutes. While still hot, it was titrated with 0.1 M KOH solution until a pink color developed. Vigorous shaking was performed as the endpoint approached to ensure complete mixing. The amount of 0.1 M KOH used by the acid was noted. The calculation of the acid value was done using a specific formula as documented by Bello *et al.,* (2023).

$$A.V = \frac{56.1 \times V \times M}{m} \quad 3.7$$

2.3 Black Soap Production

2.3.1 Sample Preparation

The native black soap was made by combining 100g of plantain skin with 500 mL of water in a beaker. The mixture was stirred and allowed to settle for 80 h. The alkaline extract was then filtered with cotton wool and a funnel. The heat was used to condense the alkaline extract, and Shea butter and palm oil were slowly introduced in a 4:1 ratio. These were further heated on a hot plate and stirred at 60oC until complete saponification occurred. The soap was then allowed to cure for 24 hours to obtain a local black soap.

2.3.2 Procedure for Saponification

A 150mL beaker was weighed and its mass was recorded as M_1. 5g of the blended oil was added into the 150 mL beaker and reweighed to determine the new mass (M_2). Then 15mL of ethanol and 15mL of 20% NaOH were further added to the solution in the beaker. A small magnetic stir bar was placed in the beaker while the mixture was heated for 30 minutes until the solution became homogenous. The transparency of the solution was noted as it is a requirement to adhere to. During heating, some of the solution evaporated then to prevent the high decrease of the heated solution, a more liquid solution containing equal volumes of ethanol and water was added.

2.3.3 Salting

After saponification, the beaker was removed from the heat source thereafter, A salt solution was prepared by mixing 30g of NaCl with 100mL of distilled water. When the salt dissolved, the solution was divided into two. 50mL of a saturated NaCl solution was measured using a graduated cylinder and put into a 400-mL beaker. The salt solution was gradually added to the solution that underwent saponification and stirred thoroughly. This process is called salting out.

2.3.4 Soap Curing

Salting out increased the density of the soap and this caused the soap to precipitate, floating on the surface of the solution. The hot solution was then put in an ice water bath, and left for some minutes until it reached a room temperature of 25°C. 20 mL of distilled water was chilled and used to rinse the solid soap in the funnel. The soap curds were then collected by vacuum filtration with an aspirator.

2.3.5 Physiochemical Properties of the Black Soap

The physical and chemical characteristics of the indigenous black soap produced were evaluated against the standards set by the Nigerian Industrial Standard (NIS). The properties include pH, Free Fatty Matter (FFM), Moisture Content, Water Insoluble Matter, and physical appearance even though there are still more properties required by NIS these properties checked in this work are the major properties many research work adhered to.

2.3.5.1 pH of Black Soap

The pH of the local black soap was determined by dissolving 2 grams of the soap in 200 milliliters of distilled water. Following this, the electrode of the pH meter was immersed in the solution to measure and record the pH value (Nnyia *et al.,* 2023).

2.3.5.2 Determination of Lathering Ability (Foam Height)

Measuring the lathering ability of the local black soap involved dissolving 2g of the soap in distilled water. The solution was then agitated in a clean blender with 200 mm of distilled water for 30s. Subsequently, the height of the resulting foam was gauged using a 1000-milliliter tube (Nnyia *et al.,* 2023).

2.3.5.3 Moisture Content

To determine the amount of moisture present in the black soap. This can be done by weighing the soap before and after drying it in an oven and the formula below can be used for estimation.

$$\text{Moisture Content (\%)} = \frac{w_1 - w_2}{w_1} \times 100\% \qquad 3.8$$

2.3.5.4 Physical Appearance of the Soap

The physical appearance such as color was seen without using any instrument.

2.4 Safety Precautions

2.4.1 Precautions during the Experiment

Adhering to the safety protocol set up in the lab manual, safety goggles were worn especially important in this experiment, since NaOH can cause permanent eye damage according to the research by (Bernat *et al.,* 2021). Likewise, gloves were worn to improve handling and prevent contamination. More so, the reaction mixture was watched at all times as it was heating, and boiling over was prevented while the volume of the heated solution was properly maintained in order not to decrease too much.

2.4.2 Safety Precautions during Titration

During titration, safety was prioritized by wearing personal protective equipment such as goggles, gloves, and lab coats. Chemicals were handled cautiously by adding acid to water. To minimize exposure to fumes, the experiment was carried out in a well-ventilated lab. Chemicals were disposed of properly as well.

3.0 RESULTS AND DISCUSSION

3.1 Result and Discussion

This study focused on the characterization of alkaline extracts from plantain peel biochar and ash, and their application in black soap formulation, along with an analysis of Shea butter oil's physiochemical properties. The biochar extract showed a pH of 11.5 and a concentration of 0.14M, while the ash extract exhibited a higher pH of 13.6 and a concentration of 0.21M. Titration results further emphasized the stronger alkalinity of the ash extract over the biochar extract.More so, in examining Shea butter oil, comparisons were made with other oils like coconut, Aegyptian Balanite, and palm kernel oils against FAO standards. Shea butter oil had a relative density of 0.91±0.01, a saponification value of 180±1.2 mgKOH/g, %FFA of 9.61±0.23 mgKOH/g, and an acid value of 10.57±0.87 mgKOH/g. These findings highlight the distinctive characteristics of Shea butter oil, particularly in terms of its saponification and free fatty acid content. The study thereafter included a comparative analysis of black soaps, including those made from plantain peel biochar (*APPB*) and ash (*APPA*). Both soaps fell within the FAO's acceptable pH range, with *APPB* at 8.70 and *APPA* at 9.93. In terms of lathering ability, *APPB* soap had a foam height of 720.00mL and *APPA* soap 703.00mL. The moisture content was 5.00% for *APPB* soap and 6.99% for *APPA* soap, indicating potential differences in shelf-life and microbial growth.

3.2 Physio-Chemical Analysis (*PPB* and *PPA* Extract)

Table 3.1: The physiochemical analysis of Alkaline Plantain Peel Biochar and *APPA*

Properties	**Plantain Peel Biochar Extract (/2160mL)**	**Plantain Peel Ash Extract(/2160mL)**
pH	11.5	13.6
Concentration (M)	0.14	0.21
Volume of KOH present (mL)	169.67	254.4

3.2.1 Titration Result

3.2.1.1 Concentration of the Alkaline Biochar Extract Result

Table 3.2: Titration result on reacting alkaline extract with acid.

	Alkaline Biochar Extract		
	Rough	Trial 1	Trial 2
Final Burette Reading (mL)	40.00	35.25	35.60
Initial Burette Reading	0.50	0.35	0.50
Amount of Acid used (mL)	39.50	34.9	35.10
Average of Best 2 result (mL)		35.00	

3.2.2.2 Concentration of the Alkaline Ash Extract Result

Table 3.3: Titration result of Alkaline Plantain Peel Ash

	Alkaline Ash Extract		
	Rough	Trial 1	Trial 2
Final Burette Reading (mL)	50.00	52.50	52.00
Initial Burette Reading	0.00	0.00	0.00
Amount of Acid used (mL)	50.00	52.50	52.00
Average of Best two results (mL)		52.25	

3.3 Physiochemical Parameters of Shea Butter Oil

The data for the parameters of Coconut Oil and Aegyptian Balanite oil in Table (4.3) were obtained from the research of Nnya *et al.*,(2023) and Bello *et al.*, (2018) respectively, and for PKO and FAO were affirmed by Zauro *et al.*, (2016).

Table 3.4: The physiochemical analysis of various oil

	Shea Butter	Coconut Oil	FAO Standard
Relative Density (g/ml)	0.91±0.01	0.902	0.89 – 0.91
SV (mgKOH/g)	180±1.2	259	189 – 199
%FFA (mgKOH/g)	9.61±0.23	2.60	< 0.5
Acid Value (mgKOH/g)	10.57 ± 0.87	1.240	≤ 30

3.4 Physiochemical Analysis of the Black Soap

Table 3.5: Physiochemical and comparative analysis of various soaps

	APPB Black	*APPA* Black Soap	Local Soap
pH	8.70	9.93	9.62
Foam Height(ml)	720.00	703.00	620.00
Moisture Content (%)	5.00	6.99	15.51
Color	Black	Black	Dark-brown

3.4.1 pH of the Black Soap

The pH of the soap is shown in Table 4.5

3.4.2 Determination of Lathering Ability (Foam Height)

Gauging the height of the resulting foam, the foam height determined was 720.00mL and 703.00mL for *APPB* Soap and *APPA* Soap respectively.

3.4.3 Physical Appearance

The colour of the soap is black

3.5 Discussion

3.5.1 The Biochar and Ash Extract

The physiochemical analysis of plantain peel biochar reveals an ash content of 11.3%, suggesting a significant mineral presence in the collected sample. Ash represents the inorganic residue remaining after the elimination of water and organic substances through heating processes. With a pH value of 13, the biochar exhibits strong alkalinity, indicating a high concentration of lye as also represented in Fig. 4.1.

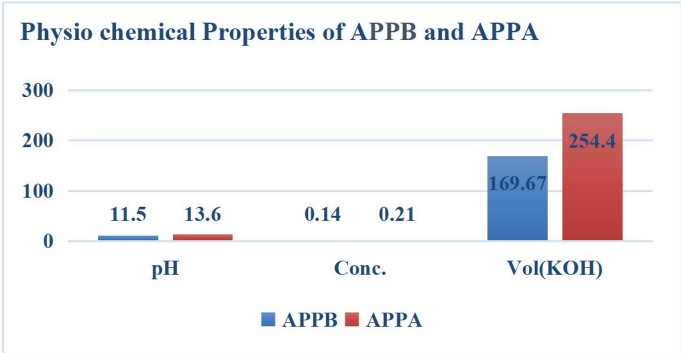

Figure 3.1: Bar chart showing the result of the properties of *APPB* and *APPA*

According to figure 3.1, the yield percentage of extract using plantain ash is higher which may reduce the cost of production of black soap and enhanced saponification although the cost of energy to produce ash may be higher.

Since both fall in line with the standard set by FAO, then economy analysis is recommended to decide which is more economical in the production of black soap.

3.5.2 Shea Butter and some common oils used in Saponification

Analyzing the data for Shea Butter, Coconut Oil, ABO, PKO, and their alignment with FAO standards, we observe distinct physiochemical properties among these oils and fats. Relative density, a measure of the oil's heaviness compared to water, shows Shea Butter and PKO aligning closely with the FAO standard range of 0.89 – 0.91 g/ml, indicating a standard oil density. Coconut Oil is slightly above this range, while ABO is notably less dense.

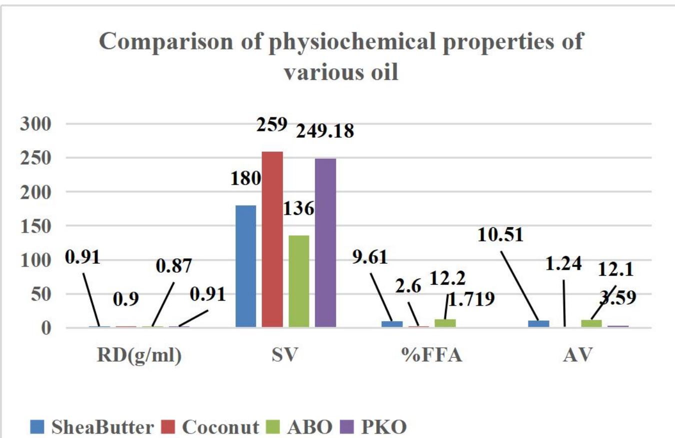

Figure 3.2: Bar chart showing the physiochemical properties of various oils using data from Table 3.4

In summary, Shea Butter and PKO fit well within FAO standards in terms of relative density, but their higher FFA and acid values indicate a need for more refining. Coconut Oil, with its higher SV and lower FFA and acid values, presents as a higher-quality oil for soap production. ABO, with its lower density and SV, may require blending with other oils for optimal soap-making properties.

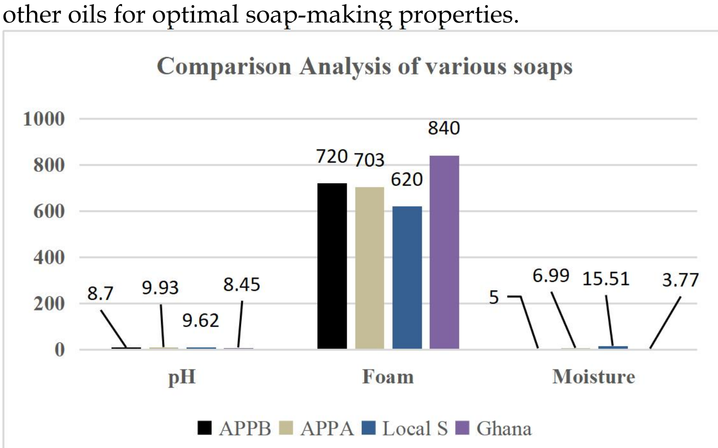

Figure 4.3: Comparison analysis of various soaps using data from Table 4.5

The comparative analysis of *APPB* Black Soap, *APPA* Black Soap, Local Soap, and Ghana Soap against FAO standards reveals each soap's unique characteristics. *APPB* and Ghana Soaps have pH levels within the FAO's recommended range, suggesting they are gentle on the skin, while *APPA* and Local Soaps have slightly higher pH values, indicating stronger cleaning properties but potentially increased harshness. In foam height, *APPB* and *APPA* Black Soaps exceed FAO minimum standards, suggesting luxurious lathering qualities, with Ghana Soap leading in foam production. Moisture content varies across the soaps, with *APPB* and Ghana Soaps having lower moisture levels, possibly indicating less hydration but longer shelf life, while *APPA* and Local Soaps offer more moisture. The soaps also differ in color, from traditional black to brown, catering to different aesthetic preferences. Overall, these soaps show a balance of effective cleansing, skin gentleness, and diverse consumer appeal in terms of hydration, longevity, and appearance.

4.0 Conclusion and Recommendation

4.1 Conclusion

This study embarks on an innovative journey to produce black soap using alkali derived from plantain peels, blending the art of traditional soap-making with modern scientific approaches. Central to this endeavor is the thorough collection and analysis of plantain peel biochar and ash, alongside a detailed examination of the physiochemical properties of Shea butter oil, a key ingredient in the soap. However, a potential drawback of this project lies in the high alkalinity of the plantain peel biochar and ash, which might affect the soap's suitability for sensitive skin, potentially limiting its appeal to a wider consumer base.

4.2 Recommendation

Considering the project's insights, it is recommended to conduct a comprehensive cost analysis to ensure the economic viability of using plantain peel biochar and ash in soap production. This analysis should include sourcing, processing, and transportation costs to maintain competitive pricing. Additionally, it is vital to assess the environmental impact of this production method. Exploring sustainable and eco-friendly practices, such as waste minimization and energy-efficient processes, can enhance the soap's appeal to environmentally conscious consumers. Implementing these recommendations could lead to a more sustainable, cost-effective, and marketable product, aligning with both economic and environmental goals lastly, a Calorimetric Analysis is required for oil blending.

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