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The Preparation and Physicochemical Analysis of Local Black Soap from Coconut Oil and Plantain Peel Biochar

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Abstract: Local black soap, produced from coconut oil and plantain peel biochar (alkaline source) was presented in this study since the agricultural raw materials gain significance in environmentally benign feedstocks for saponification reactions. The physicochemical analysis of the coconut oil and plantain peel biochar shows remarkable free fatty acid (FFA) and alkalinity contents respectively suitable for soap making. The physicochemical properties of the as-prepared local black soap demonstrated a good moisture content (15.1 %) showing that it can be stored for long periods without any water-associated deteriorations. The percentage of matter insoluble in water (4.4 %), matter insoluble in alcohol (12.3 %), FFA content (2.1 %), and pH (9) were found to be higher than the acceptable limit according to the Nigerian Industry Standards (NIS). Also, the total fatty matter (TFM) was higher (67.6 %) than the minimum acceptable level with a good foam height of 2.5 cm. The overall results show good soap properties and are suitable for domestic purposes.

Keywords: Local black soap, plantain peel, coconut oil, Agricultural feedstocks

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1. INTRODUCTION

The agrarian diversity in human and economic growth is a significant concern. Over the years, raw materials of agrarian origin have been sourced as benign feedstocks for many industrial activities because they are eco-friendly, cheap, and readily available. This has increased the awareness of agricultural production, necessary to meet the demand emanating from population increase and also for servicing commercial industries. Interestingly, large quantities of waste materials which are residues of agrarian practices, contribute significantly to the degradation of the environment and as such must be carefully handled to prevent any form of negative consequences such as pollution and economic loss (1,2).

Today, several researchers are beginning to focus on the recyclability of bio-waste materials, pollution control, and income generation. However, the conversion of agricultural wastes into new products or their use in the formulation of new products must certainly characterize the chemical composition, surface morphology as well as physicochemical properties of any of these materials (3). For instance, the shells from peanuts account for about 20% of the peanut with production amounting to 46 metric tons every year. The peanuts are majorly disposed of through burning which contributes to environmental pollution and in some cases could be used in the production of livestock feed which requires proper treatment and processing. Peanut shells contain antioxidants due to the presence of Phyto derivatives used relevantly in cosmetic industries and were reported to function as an absorbent to eliminate heavy metals (4,5). Hence, agro- and non-agro-waste residues have been extensively explored as sustainable unfired earth-building materials for blocks construction (6), waste ginger straw has been examined as a productive heterogeneous catalyst for biodiesel production (7), while Chickpea husk has shown huge prospects for textile coloring and operational finishing (8).

Nevertheless, the production of soap from natural products in place of synthetic chemicals is gaining attention in recent times. Agricultural by-products (wastes) are considered sources of organic materials in soap production because they have been reported to contain phytochemicals for moisturizing the human skin and treatment of skin-related infections (9,10). Organic compounds from agro-wastes have shown antibacterial activity against *Streptococcus pyogenes* and *Pseudomonas aeruginosa* as presented by Rambabu and colleagues (11). More also, agro-waste materials e.g. plantain peels have been considered a viable source of alkaline for saponification reactions because of the important class of phytochemicals (alkaloids, flavonoids, terpenoids, phenols, etc.) as well as vital minerals components (calcium, copper, potassium, phosphorus, zinc, etc.) (12). Besides, oils derived from agro-materials (e.g. palm oil and coconut oil) contain saponifiable free fatty acids as well as alkaloids, terpenoids, saponins, etc. (13–15). 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 (by-product) (Figure 1) (16). Negative ions comprising long hydrocarbon chains connected to a carboxyl group are common in the soaps that are being produced, giving it a cleansing action when used for bathing, washing, or general cleaning (17). Therefore, this paper examines the production of local black soap using two agro-feedstocks (coconut oil and plantain peel), and the physiochemical properties of the soap produced were also examined.

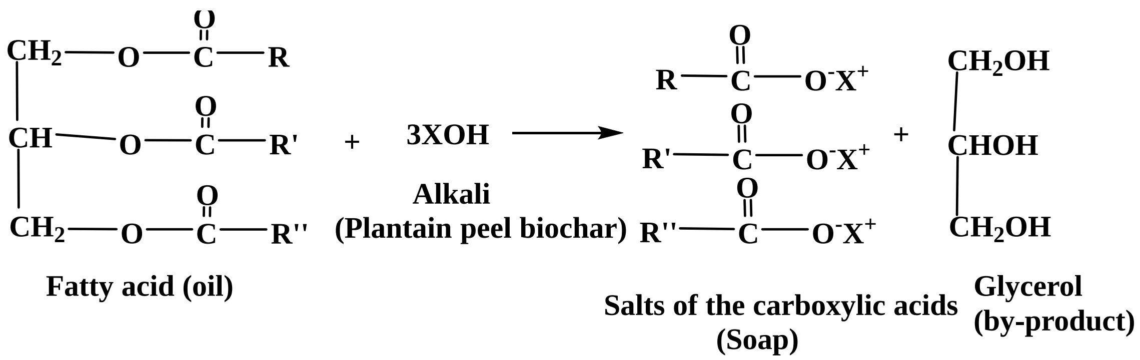

Figure 1: Saponification reaction of a triglyceride and alkaline to produce soap.

2. EXPERIMENTAL SECTION

2.1. Sample Collection and Materials Used

Waste plantain peels were collected from a local eatery. Pure coconut oil was bought from commercial vendors. The other analytical grade reagent used like Hydrochloric acid (HCl), Potassium Hydroxide (KOH), Phenolphthalein indicator, Ethanol (C_2H_5OH), Nitric acid (HNO_3), Neutralized isopropyl alcohol (IPA), Sodium hydroxide (NaOH) Sulfuric acid (H_2SO_4), Acetone, Diethyl ether, Methyl orange indicator, and Barium chloride ($BaCl_2$) were supplied by Maya Reagent, and Sinopharm Chemical Reagent Co. Ltd. For the production of aqueous solutions, distilled water was employed.

2.2. Preparation of Plantain Peel Biochar

To remove dirt, fresh plantain skins were washed with distilled water and dried in a slightly warmer setting. Plantain peel biochar was made by cutting the peels into little pieces and heated in a furnace for 5 h at 600 ºC. The resulting biochar was allowed to cool in a desiccator before being pulverized in a mortar and sieved with a mesh.

2.3. Production Process of Local Black Soap

The native black soap was made by combining 100 g of plantain skin biochar with 500 mL of water in a beaker. The mixture was stirred and allowed to settle for 72 h. The alkaline extract was then filtered with cotton wool and a funnel. The heat was used to condense the alkaline extract, and coconut oil was slowly introduced in a 4:1 ratio. These were further heated on a hot plate and stirred at 60 ºC until complete saponification occurs. The soap was allowed to cure for 24 h to obtain the local black soap (18) (Figure 2).

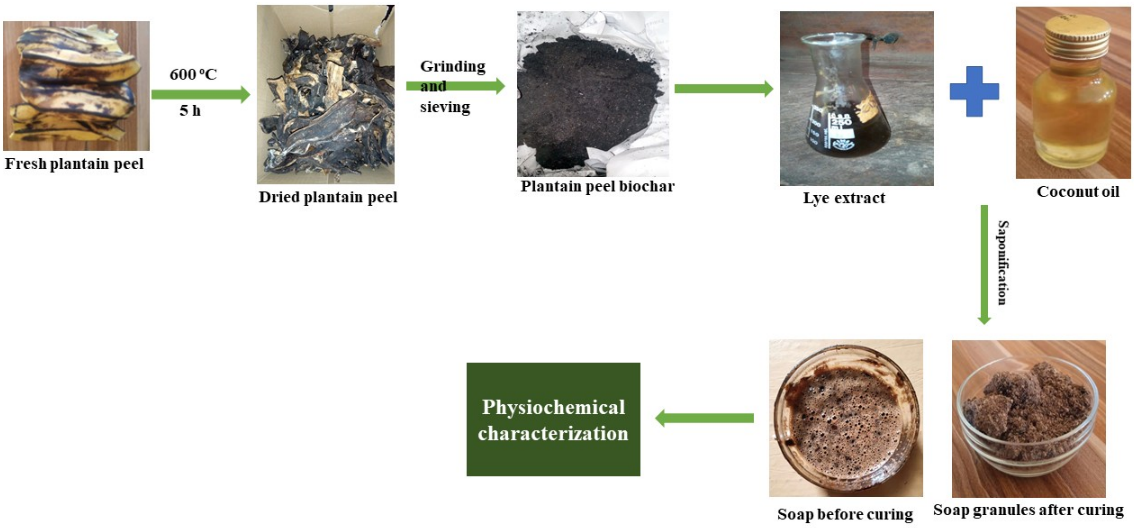

Figure 2: Schematic route for the preparation of local black soap.

2.4 Physicochemical analysis

The physicochemical analysis of the plantain peels biochar, coconut oil, and local black soap was performed according to established procedures (18,19).

2.4.1 Determination of moisture content

Weighing 2.15 g of black soap on a pre-weighed crucible and keeping it in an oven at 100°C - 105°C for 1 h was used to determine the moisture content of the soap. The soap was allowed to be cooled before being weighed once more. The given equation was used to compute the percentage moisture content (Eq. 1).

$$\text{Percentage Moisture Content} = \frac{w_3 - w_2}{w_1} x\, 100\,\% \qquad \text{(Eq. 1)}$$

Where W_1=sample weight; W_2=weight of crucible; W_3=weight after drying.

2.4.2 Determination of water-insoluble matter

Weighing 5.06 g of black soap into a 250 mL beaker, yielded the amount of water-insoluble materials in the soap. The soap was then dissolved in 100 mL of distilled water by heating on a hotplate. The soap solution was filtered and rinsed with distilled water three times. Further, the residue was kept in an oven (100 °C – 105 °C) for 1 h. After cooling in a desiccator, the residual weight was calculated. The following formula was used to compute the proportion of water insoluble-matter (Eq. 2):

$$\text{Percentage Moisture Insoluble Matter} = \frac{w_3 - w_2}{w_1} x\, 100\,\% \qquad \text{(Eq. 2)}$$

Where W_1=weight of sample; W_2=weight of filter paper; W_3=weight of filter paper residue after drying.

2.4.3 Determination of free fatty acid (FFA)

Firstly, 10 g of the black soap was weighed into a conical flask, followed by the addition of 100 mL of neutralized isopropyl alcohol (IPA) to determine the amount of FFA in the soap. The solution was allowed to boil on a hot plate until the soap was dissolved. Next, 10 mL of barium chloride was added to the mixture and titrated against NaOH using phenolphthalein as the indicator. The percentage FFA was calculated by Eq.3:

$$Percentage\ FFA = \frac{endpoint\ x\ Normality\ of\ base\ x\ 200\ x\ 100}{1000\ x\ weight\ of\ sample} \qquad \text{(Eq. 3)}$$

2.4.4 Determination of alcohol-insoluble matter
In a beaker, 2 g of the black soap was heated with 100 mL of neutralized IPA to estimate the proportion of the alcohol-insoluble matter. The soap solution was filtered and the insoluble matter was transferred using a hot neutralized IPA until all the soap entrained within the filter paper has been removed. The residue was baked for 30 min and then cooled in a desiccator. The percentage of the alcohol-insoluble matter was calculated according to Eq. 4.

$$\text{Percentage Moisture Insoluble Matter} = \frac{w_3 - w_2}{w_1} x\, 100\,\% \qquad \text{(Eq. 4)}$$

Where W_1=weight of sample; W_2=weight of filter paper; W_3=weight of filter paper + residue after drying.

2.4.5 Determination of pH
By dissolving 2 g of local black soap in 200 mL of distilled water, the pH of the soap was calculated. The pH meter's electrode was then dipped into the solution to record the pH.

2.4.6 Determination of total fatty matter (TFM)
By heating 5 g of the local black soap with 100 mL of distilled water, the total fatty matter was measured. For facile precipitation of the fatty components from the soap, the soap solution was transferred into a separating funnel, followed by the addition of methyl orange indicator and sulfuric acid. Thereafter, the soap solution was cooled, and diethyl ether (100 mL) was introduced to allow phase separation. The solution was further separated by shaking until the aqueous layer became clear and allowed to stand. The solution was re-washed with diethyl ether and with water until methyl orange was neutralized. The set-up was allowed to stand for another 5 min and run off any separated water. The solvent was then distilled off by adding 5 mL of acetone to the clean solution and heating it to evaporate the diethyl ether. Also, the acetone was removed under the steam of dry air. The beaker was placed in the oven for 30 min and allowed to cool and the weight was taken. The percentage of the total fatty matter was collected as calculated according to Eq. 5.

$$\text{Percentage Moisture Insoluble Matter} = \frac{w_3 - w_2}{w_1} x\, 100\,\% \qquad \text{(Eq. 5)}$$

Where W_1=weight of sample; W_2=weight of beaker; W_3=weight after drying.

2.4.7 Determination of foam height (lathering ability)
By dissolving 2 g of local black soap in distilled water, the foam height of the soap was determined. This was agitated in a clean blender with 200 mL of distilled water for 30 s. Thereafter, the height of the foam formed was measured on a 1000 mL tube.

3. RESULTS AND DISCUSSION

3.1 Physicochemical and Structural Properties of Plantain Peel Biochar

Physicochemical properties of plantain peel biochar show an ash content of 11.3% which indicates the presence of sufficient minerals in the sample collected. In the presence of oxidizing agents, ash is the inorganic residue left after the water and organic matter have been removed by heating (20). The value of pH was 13 which indicates a strong alkaline content and a lye concentration of 4.5 which makes it fit for the production of soft soaps.

Furthermore, the characterization of the plantain peel biochar with Fourier transform infrared (FT-IR) reveals a prominent peak at 3413 cm^{-1} which could be due to OH stretch (alcohol and carboxylic acid) (Figure 3). The weak peak at 2923 cm^{-1} confirms the presence of OH from carboxylic acid which could be due to the presence of phytochemicals like tannins (e.g. gallic acid), cellulose, and hemicelluloses in the plantain peels (21). More also, the peak at 1620 cm^{-1} could be ascribed to C=C vibrational stretch. The Peaks observed at 1387-1246 cm^{-1} can be attributed to the C-H bending of cellulose, hemicelluloses, or lignin polymer. The peak at 1100 cm^{-1} could be associated with the =C-O-C symmetric and asymmetric stretch in ether linkages of polyphenols (22); while the weak peaks appearing at 841-624 cm^{-1} might be linked to the presence of amine groups and other mineral elements (23).

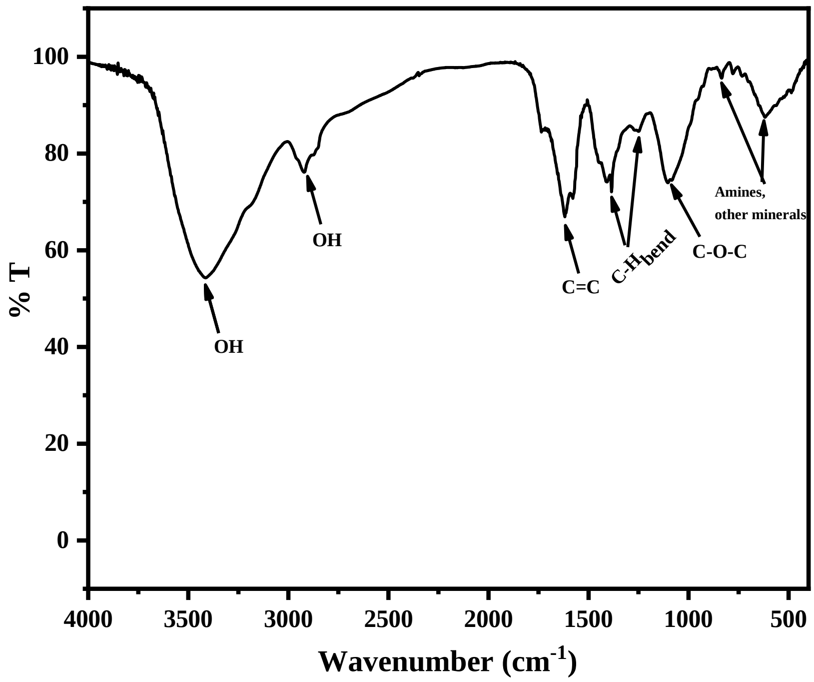

Figure 3: FT-IR spectrum of plantain peel biochar.

In addition, the structural morphology of the plantain peel biochar shows porous and thick fibrous-like nature with a cluster of small particles on the surface (Figure **4**). The morphological structure observed usually exhibits a higher surface area, with promising prospects to make a good soap material (24). The porous nature of the material will enable good interaction with the chemical content in the oil to give a good soap formulation.

3.2 Physicochemical Properties of Coconut Oil

The results in Table 1 show the physicochemical features of the coconut oil utilized in the local black soap production. The density of the coconut oil was 0.902 g/mL. The saponification value is the amount of KOH necessary to saponify 1 g of oil in milligrams. It is a measure of the free acid and saponifiable ester groups. This important parameter helps to determine the amount of salt to be formed. Substantial saponification and ester values of 259.00 mgKOH and 228.3 mgKOH respectively were obtained in the coconut oil revealing promising prospects for soap making. More also, coconut oils contain significant saturated and unsaturated fatty acids which contribute to the hardness, aroma, and cleansing properties of soaps. The coconut oil had a free fatty acid (FFA) concentration of 2.60, with a corresponding FFA composition (Table 2). The titer value provides information on the actual value of the melting point of the soap during saponification. The result obtained shows a titer value of 20.4ºC. This implies that the coconut oil possesses has a low melting point with good soap-forming properties. The coconut oil demonstrated a low acid value of 1.240 mg KOH and a low moisture content of 0.4%. This indicates that the oil will remain stable for a long time and will effectively preserve the soap against rancidity. The physiochemical parameters obtained for the coconut oil are within acceptable standards and results from other studies (25).

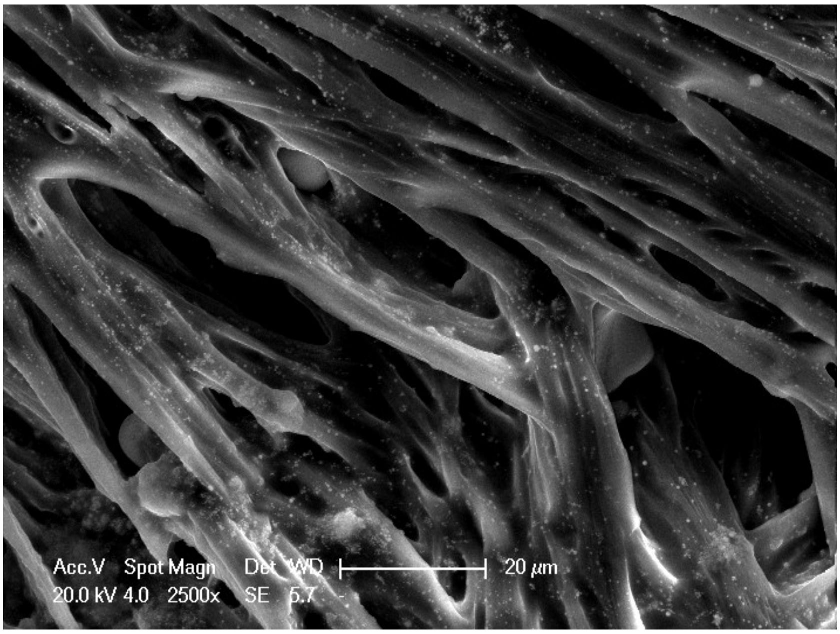

Figure 4: SEM micrograph of plantain peel biochar.

Table 1: Physicochemical properties of the coconut oil for local black soap production.

Property	Value
Density (g/mL)	0.901
Saponification value (mgKOH)	259.00
Ester value (mgKOH)	228.3
FFA (%)	2.60
Titre value (ºC)	20.4
Acid value mgKOH	1.240
Moisture content (%)	0.4

Table 2: Coconut oil's approximate fatty acid content.

Type of fatty acid	Composition (%)
Lauric acid	48.00
Myristic acid	18.49
Palmitic acid	8.80
Oleic acid	7.81
Stearic acid	3.00
Caprylic acid	5.55
Caproic acid	0.50
Linoleic acid	1.35
Capric acid	6.50

3.3 Physicochemical Properties of Local Black Soap

The physicochemical parameters of the native black soap obtained were compared with the Nigerian Industrial Standard (NIS) (Table 3). The reaction of unsaponified fat with excess water in the soap to form glycerol and FFA, assisted by soap hydrolysis, represents the FFA levels in soaps. The obtained black soap displayed a moisture level of 15.1%, which was insignificantly higher (0.1%) than the NIS model. The good moisture content of the black soap shows that it can be stored for 12 - 24 months without any water-induced deterioration. Thus, the black soap can be considered harmless for domestic and commercial applications (26). The percentage of black soap that was insoluble in water was 4.4%, which was more than the allowed standard of 2.5%. The soap's lather will be affected by the excessive amounts of insoluble particles in the water. Similarly, the percentage of matters insoluble in alcohol of the local black soap was 12.3%, higher than the acceptable limit of 5.0%. This reveals that there are plausibly insoluble fats and sodium silicate insoluble in alcohol in the local soap. This could indicate that the black soap included waxes and lipids that are water-insoluble (27).

Table 3: Comparison of the physicochemical parameters of the native black soap produced (in this study) to the Nigerian Industrial Standard (NIS).

Parameter	Local black soap	NIS
Physical appearance	Blackish brown	-
Moisture content (%)	15.1	15 max.
Matters insoluble in water (%)	4.4	2.5 max.
Matters insoluble in alcohol (%)	12.3	5 max.
Free fatty acid content	2.1	0.2 max.
pH	9	6-10
Total fatty matters (%)	67.6	60 min.
Foam height (cm)	2.5	-

The FFA content of the local black soap was 2.1%. This value was higher than the specified limit and implies poor transparency of the black soap (28). The black soap shows a pH of 9 since soaps are alkaline substances thereby functioning as barriers against dangerous organisms such as bacteria and viruses, this can counteract the body's protective acid mantle. The obtained pH of the soap shows that the saponification process is completely hydrolyzed, and may not impact corrosive action when applied to the skin. On the other hand, the corrosive nature of local black soaps is solvable by increasing oil content (29). Thus, the local black soap may be suitable for domestic use.

The soap's cleansing ability is proportional to its total fatty matter (TFM). The TFM of the local black soap was 67.6%, higher than the minimum of 60 % set by the NIS. This suggests that the local black soap possesses high cleansing properties. The foam height is an attribute associated with oil composition used in black soap production. Lauric acid, the major FFA in coconut oil has a remarkable foaming property. Thus, the foam, high of 2.5 cm shows the good formability of the local black soap (30).

4. CONCLUSION

The production of local black soap from agro-based feedstocks of coconut oil (triglycerides) and plantain peel biochar (alkaline source) is hereby presented in this study. The physicochemical characterization of the plantain peel biochar demonstrated good alkalinity which makes it fit for making soft soaps. The physicochemical characterization of the coconut oil extracts, indicates good fatty acid content and remarkable oil properties within acceptable standards. Thus, the physicochemical parameters of the black soap displayed good formability and harmless properties for domestic and commercial applications. However, the results reveal the presence of some sort of waxes and fat components in the soap reducing the transparency of the black soap. Nevertheless, local black soap manufacturing should be encouraged since it has good quality for commercial consumption and thereby reduces the waste generated from plantain peels.

5. CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

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