Production and Characterization of Soap using Alkali from Agricultural Waste

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ABSTRACT

This study evaluated the potential of agricultural wastes, namely yam (Dioscorea rotundata), cassava (Manihot esculenta), and plantain (Musa paradisiaca) peels, as sustainable sources of alkali for soap production. The peels were dried, calcined, and subjected to aqueous extraction to obtain alkali-rich solutions for soap production using the semi-boiled method. Eight soap formulations, comprising single and blended alkali sources, were produced with palm kernel oil, while a commercial laundry soap (Canoe Soap) served as the control. The elemental composition of the ashes was determined using Energy Dispersive X-ray Fluorescence (EDXRF), and the soaps were evaluated for pH, moisture content, Total Fatty Matter (TFM), Free Caustic Alkali (FCA), Total Alkali, foam height, and heavy metal content. Data were analyzed using one-way Analysis of Variance (ANOVA) and Tukey's HSD test at $p < 0.05$. Plantain peel ash contained the highest potassium oxide (K_2O) content (20.30%), followed by yam (17.17%) and cassava (5.16%). Significant differences ($p < 0.05$) were observed among the formulations for all physicochemical properties. The ternary blend (yam–cassava–plantain) produced the best-quality soap, recording the highest TFM (87.63%), greatest foam height (510 mL), acceptable pH (10.00), and safe FCA (0.19%), outperforming the commercial control. Heavy metal analysis showed that although the raw ashes contained detectable concentrations of some metals, the finished soaps contained non-detectable levels of Pb, Zn, and Cr, indicating that the aqueous extraction and saponification processes effectively minimized heavy metal transfer. The formulated soaps complied with WHO and Standards Organisation of Nigeria (SON) safety requirements. These findings demonstrate that agricultural waste-derived alkalis, particularly blended formulations, provide a safe, sustainable, and cost-effective alternative to synthetic alkalis for producing high-quality soap while promoting agricultural waste utilization and environmental sustainability.

INTRODUCTIONS

The increasing pressure on demand of raw materials for the production of various goods in recent time as a result of continual increase in population has resulted to quest for readily available, eco-friendly, cost effective and renewable resources for manufacturing purposes. One of such resources are usage of agricultural waste materials which are used in place of conventional materials for the production of goods for human daily needs. Agricultural wastes (Aws), as its name suggests, is a waste product from farming practices and can be generated from different sources such as residues from crops, agro-industries, aquaculture etc (Koul et al., 2022). These AWs can be turned to good use and when compared to conventional materials have little or no effect to the environment at production of the raw materials up to consumption and returning back to the product waste cycle again.

One of the products of which agricultural waste materials are being incorporated into is in the production of soaps. There have been researches aimed at looking for agricultural waste materials that minimizes environmental impact of soap production but also helps in waste disposal (Ramawat et al., 2020). The possibility of converting agricultural by-products which are now regarded as refuse, into useful products such as soap has been the main interest of researchers now (Tiwari, et al., 2021).

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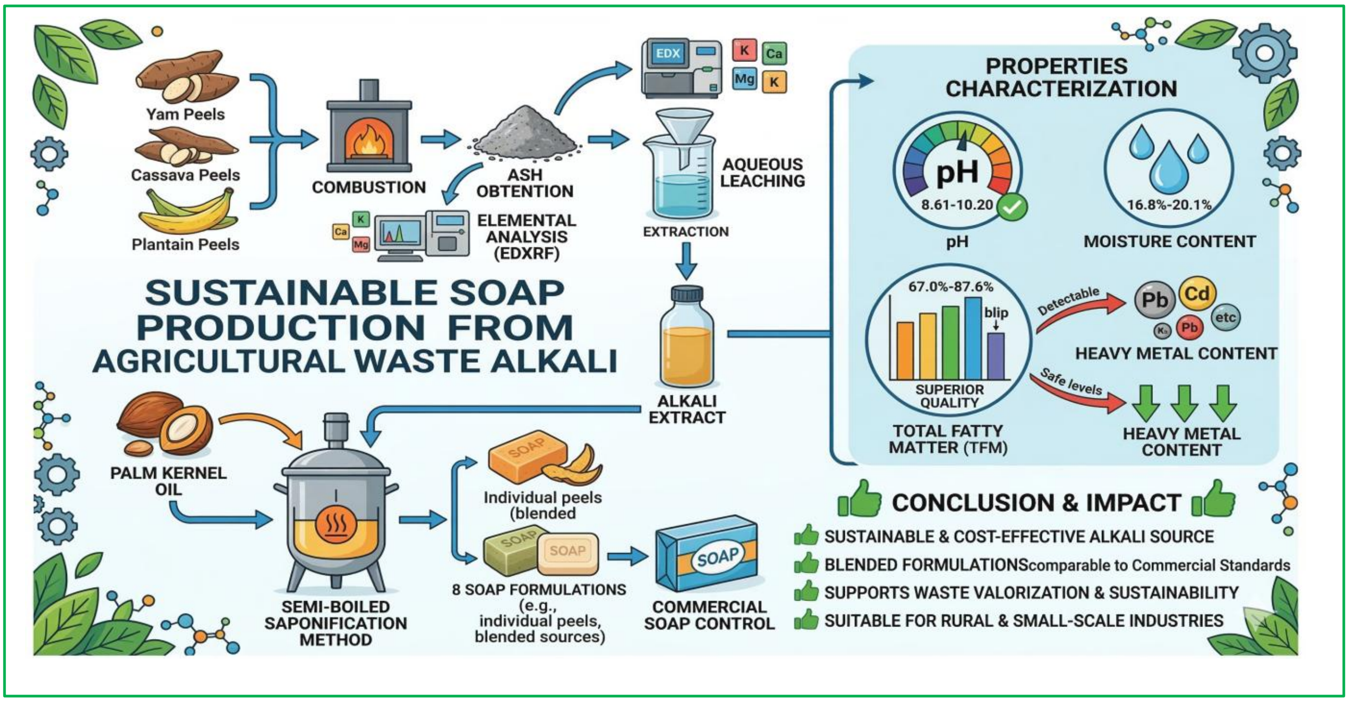

Such interest lies at the available choices of agricultural waste in which one can recover useful compounds, and how the saponification procedure will be optimized to generate proper soap. Hence, converting raw agricultural by-products to usable everyday consumables (Phiri, et al., 2023).

Agricultural waste comprises tissues remaining after the harvest of crops such as stalks; and organic waste such as food scraps, especially fruit skins. This type of waste poses environmental challenges because of the poor means of disposal. Nevertheless, a certain category of agricultural waste does include some alkali, which is a key component in soap manufacture. Wood ash, banana peels, cocoa pod husks, and a variety of plant scalps are particularly high in potassium, which is abundantly available as potash (Akindejoye, et al., 2017). Potash has always been used as an alkali for soap, by rural people, made from ashes; however, it can replace synthetic chemical alkalis like soda in soapy formulations.

One advantage with potash sourced from waste is that there is the possibility of a process that can result in some biodegradable product. The issue of soaps produced by utilizing natural alkalis is that they are energetically better toward the environment in water bodies and do not pose excessive risk for the nature. Soaps containing potash are also believed to be milder and softer on the skin which makes it possible for use in personal care products where consumers are on the rise demanding organic and chemical free products (Rahimpouret, et al., 2024).

Processing of the agricultural waste to recover alkali incorporates burning agricultural wastes to get ash, which is dissolved in water to obtain lye, a solution with potassium carbonate. The lye produced is the alkali required for the saponification process. Which agricultural waste ash was used: banana peel, cocoa pod and hardwood to produce lye, which is effective in soap production demonstrating similar properties with those made from commercial alkalis (Adeyemi, et al., 2020). Since the extraction process is straightforward, it can be performed by local and small-scale soap makers especially in rural and developing areas where chemical alkalis are either lacking or are quite costly. Production of soap with agricultural waste alkali also has positive social impacts, as soap production in developing areas can also be a means of earning income and empowering local people. Farmers would be able to produce good quality soap by recycling waste materials available in their locality rather than importing over-priced soap or chemical industrialist’s soap (Igbashio, et al., 2022).

In spite of the wide-ranging benefits, there are still some challenges in the use of agricultural waste as an alkali source in soap production. This challenge lies in the availability and the quality of the waste materials which might differ in different countries and the bush ash production process should be well managed to maintain the level of alkali. Further, research is focused on the methods of extraction for improvement of process and operation efficiency scalable to bigger production plants. Also, there has been a lack of adequate research into the safety and the long-term impacts of use of agricultural waste soaps on humans and the environment and this needs to be studied (Koul, B., et al., 2020).

To know if soap produce from this source meets the required standard for human usage and fit for environment when discharged after usage, some selected test needs to be carried on it. This test includes studying the physical and chemical characteristics like pH, lathering ability, moisturizing effect, and biodegradability. These features determine soap quality and efficacy of use, and assurance that the product is at least at par with the ones found in the market (Aminullah Zakariyyah Abdul et al., 2024).

This study therefore is proposing to investigate the feasibility of using agricultural residues such as (yam peels, plantain peels, and cassava peels) as a new alkali source in soap making, with emphasis on its environmental, economic, and social advantages. This study believes that since soaps manufactured from waste derived alkalis offer some beneficial properties, these benefits will even foster the development of sustainable product development and waste reclamation. Results from this research could support the green initiative in the soap industry and enhance the effective use of agricultural sources.

MATERIALS AND METHODS

Sample Collection

Plantain peels (Musa paradisiaca) were sourced from local roasted plantain vendors and domestic waste streams in Abraka, Asaba, Warri, and Effurun, Delta State. Preference was given to unripe and semi-ripe peels to minimize caramelization of sugars during combustion, which can occlude the ash matrix and reduce alkali permeability (Umeh-Idika & Maduakor, 2013; Onyegbado et al., 2002). Yam peels (Dioscorea rotundata) were collected from restaurants and household kitchens, while cassava peels (Manihot esculenta) were procured from small- to medium-scale garri processing facilities in Abraka and Ozoro. Palm Kernel Oil (PKO) was selected as the triglyceride source due to its unique fatty acid profile and was sourced from a local oil mill in Delta State to ensure the study simulated a fully indigenous production cycle. The oil was purified by heating to 100 °C to eliminate moisture and filtered to remove suspended mucilage before use.

Sample Preparation

The peels were manually sorted to remove non-biomass contaminants such as plastics and stones, then washed thoroughly with tap water, followed by a final rinse with distilled water to remove adhering soil particles. The wet peels were cut into pieces approximately 2–3 cm in diameter (to increase the surface area for drying) and then sun-dried for a period of 14 days on elevated racks covered with mesh to prevent dust contamination (Nnyia et al., 2023; Umeh-Idika & Maduakor, 2013). This was followed by oven drying at 105 °C for 24 hours to achieve a constant weight and a moisture content below 10%, ensuring efficient combustion and preventing the formation of incomplete combustion products (soot) (Zauro et al., 2016).

The dried peels were subjected to open-air combustion in a simplified perforated metal combustion pan and then combusted in a muffle furnace at 600 °C for 5 hours (Nnyia et al., 2023). The resulting white-grey ash was allowed to cool in a desiccator to prevent the absorption of atmospheric moisture and stored in airtight hermetically closed containers prior to alkali extraction (Nnyia et al., 2023; Kalembkiewicz et al., 2018).

Extraction of Alkali (Lye) from Ash

Alkali was extracted using the aqueous leaching method (Yu et al., 2010). 200 g of the ash was mixed with 1 L of boiling distilled water. The slurry was stirred vigorously for 30 minutes using a magnetic stirrer and allowed to stand for 24 hours. The mixture was filtered using Whatman No. 1 filter paper, and the filtrate (crude lye solution) was collected. The lye concentration was determined by acid – base titration using standard 0.1 M Hydrochloric acid (HCl) solution and methyl orange indicator. The concentration of alkali (M) was calculated using the equation:

$$C_aV_a = C_bV_b \qquad \text{eq. 1}$$

where C represents concentration and V volume (Babayemi et al., 2010; Jedidiah, 2019; Umeh-Idika & Maduakor, 2013).

Characterization of Ash and Alkali Extracts

The ash samples were subjected to non-destructive elemental analysis using an Energy Dispersive X-ray Fluorescence (EDXRF) Spectrometer (Model: Thermo Scientific ARL QUANT'X) at the Centre for Energy Research and Training, Umaru Musa Yar'adua University, Katsina. 5 g of each ash sample was mixed with a binder (wax) and pressed into a pellet using a hydraulic press at 15 tons pressure to ensure a uniform surface for X-ray irradiation. The samples were analyzed under four condition sets (Low Za, Low Ze, Mid Zb, and High Za) to cover the full spectral range of elements from Sodium (Na) to Uranium (U). Key parameters included a tube voltage of 40 kV, a tube current of 1.24 mA, and a live time of 60 seconds in an air atmosphere. Characteristic X-ray photons emitted by the elements were detected by a Silicon Drift Detector (SDD). The spectra were deconvoluted using the AXIL software to quantify the concentration of elemental oxides (K_2O, CaO, SiO_2, P_2O_5, etc.).

Soap Formulation and Synthesis

The soap synthesis was executed using the semi-boiled method as described in Zauro et al. 2016, with the exemption of sodium hydroxide. 500 ml of palm kernel oil was heated to 60 °C, after which the concentrated alkali extract (or blend) was added slowly to the warm oil under continuous mechanical stirring at 500 rpm. The mixture was maintained at a constant temperature until trace was achieved, indicating completion of saponification. The thickened soap paste was poured into pre-lubricated silicone molds and allowed to set at room temperature for 24 hours and subsequently air-dried for 21 days.

Experimental Design of Formulations

The experimental design (Table 3.1) involved eight soap formulations developed to evaluate the individual and combined effects of plant-derived alkali sources on soap quality. Alkali extracts from yam peel, cassava peel, and plantain peel ashes were used singly or in defined blends, with a commercial laundry soap serving as the control.

Physicochemical Analysis of Soap

The produced soaps were evaluated for key quality parameters to determine their quality parameters relative to Nigerian Industrial Standards (NIS 187:1984) and international benchmarks.

Determination of Total Fatty Matter (TFM)

The acid hydrolysis method was employed (Henderson & Henderson, 2025). 5 g of soap shavings were dissolved in 100 mL of hot water and treated with 40 mL of 0.5 M HNO_3 to decompose the soap into fatty acids. The mixture was heated until the fatty acids separated as a clear oily layer. The fatty acids were extracted using 50 mL of petroleum ether in a separating funnel. The organic layer was washed with water until neutral to methyl orange, dried over anhydrous sodium sulfate, and the solvent evaporated. The residue was weighed, and TFM expressed as a percentage of the original soap mass (Betsy, Jilu, Fathima, & Varkey, 2013).

$$\text{TFM (\%)} = \frac{\text{Mass of Extracted Fatty Acids}}{\text{Mass of Soap Sample}} \times 100 \qquad \text{eq. 2}$$

Determination of Moisture content

Five grams of pulverized soap was weighed into a pre-dried and weighed porcelain crucible. The sample was dried in an air oven at 105 °C for 4 hours until a constant weight was achieved. The weight loss was quantified as moisture content (American Oil Chemists' Society, 2024).

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Determination of pH

A 10% (w/v) soap solution was prepared by dissolving 10 g of soap in 100 mL of distilled water. The solution was allowed to equilibrate at 25 °C, and the pH was measured using a calibrated digital pH meter (Hanna Instruments) (NEMI Method Summary - 973.41, 1990).

Determination of Free Caustic Alkali

5 g of soap was dissolved in 30 ml of neutralized ethanol. The solution was titrated against 0.05M H_2SO_4 using phenolphthalein indicator. The disappearance of the pink color indicated the neutralization of free alkali. The result was calculated as % Na_2O or K_2O (American Oil Chemists' Society, 2024).

Determination of Foam Height

Foam height, indicating lathering capacity, was determined by dissolving 2 g of the soap sample in 100 mL of distilled water inside a 500 mL graduated measuring cylinder. The cylinder was stoppered and shaken vigorously for exactly 2 minutes. The solution was allowed to stand for 10 minutes, after which the volume of the generated foam was recorded in milliliters (mL).

Toxicological Assessment (Heavy Metal Analysis)

1.0 g of each soap sample was weighed into a digestion flask. A 15 mL mixture of concentrated nitric acid (HNO_3) and perchloric acid ($HClO_4$) in a 3:1 ratio was added. The mixture was heated on a hot plate in a fume cupboard until the emission of red nitrous fumes ceased and a clear solution was obtained. The digestate was cooled, filtered into a 50 ml volumetric flask, and made up to the mark with deionized water, and the concentrations of Lead (Pb), Zinc (Zn), Chromium (Cr), and Copper (Cu) were determined using Flame Atomic Absorption Spectrophotometry (AAS) (Model: Shimadzu AA-6800) (Ismail & Al-Swefee, 2023).

Statistical Analysis

Data obtained from the physicochemical, elemental, and toxicological analyses were expressed as Mean ± Standard Deviation (SD) of triplicate determinations. Statistical evaluations were performed using SPSS software (Version 25.0). One-Way Analysis of Variance (ANOVA) was utilized to test for significant differences among the various soap formulations and the commercial control. Where significant differences existed ($p < 0.05$), Tukey's Honestly Significant Difference (HSD) post hoc test was applied to separate the group means and assign significance superscripts.

RESULTS AND DISCUSSION

Physicochemical Properties of Produced Soaps

The physicochemical properties of the soaps produced from different agricultural waste alkalis (specifically Yam peel, Cassava peel and Plantain peel along with their blend), including pH, moisture content, and Total Fatty Matter (TFM), are summarized in Table 4.1 and a graphical representation of these physicochemical trends is provided in Figure 4.1 below. EDXRF spectrum of the Cassava, Yam and Plantain samples are presented in the Supplementary Material Figure S1, S2 and S3

pH Analysis

The pH values of the formulated soaps ranged from 8.61 to 10.20. Soap produced from plantain ash recorded the lowest pH (8.61), while the yam ash and cassava ash soaps exhibited pH values of 9.34 and 9.80, respectively. The binary blend of yam and plantain ash, as well as the commercial control, showed the highest pH (10.20). Soaps are naturally alkaline salts of weak fatty acids; a pH below 9 usually indicates the presence of excess free fatty acids resulting from super-fatting or incomplete saponification, while a pH above 11 suggests the presence of excess harsh alkali (Yahaya et al., 2012; Oyekunle et al., 2021). The standard pH range for laundry soaps is typically 9.0 to 11.0 (Idoko et al., 2018, Yahaya et al., 2012). The formulated soaps, particularly the blends (9.81 to 10.20), compared favorably with the commercial control (10.20) and fall strictly within the accepted range for laundry and toilet soaps (Yahaya et al., 2012). The plantain ash soap (pH 8.61) was notably mild, falling slightly below the typical alkaline range. This mildness makes it potentially suitable for sensitive skin or facial cleansing and corroborates the traditional use of indigenous black soap for skin conditions (Nangbes et al., 2014). This finding aligns with Nangbes et al. (2014), who reported a similarly mild pH of 8.19 ± 1.12 in toilet soaps from Plateau State, noting their safety for dermal application. Furthermore, Nnyia et al. (2023) produced a local black soap using plantain peel biochar with a pH of 9, noting that while alkaline, such soaps are generally not corrosive to the skin if the saponification is complete. The yam ash soap (pH 9.34) and cassava ash soap (pH 9.80) fell within the standard pH range for toilet soaps, indicating good saponification efficiency. The yam and plantain ash blend (pH 10.20) exhibited the highest alkalinity. This synergistic increase may be due to the interaction of the high phosphate content in yam ash buffering the solution at a higher pH, or to a more efficient extraction of alkali when the ashes are mixed. These results are consistent with the findings of Maduelosi et al. (2025), who reported that commercial laundry soaps in Port Harcourt markets had pH levels ranging from 9.5 to 11.0, and Beetseh and Anza (2013), who recorded a pH of 9.0 for cassava-based local soap "Chahul Mtse."

Moisture Content

Moisture content is an important property that affects the shelf life, hardness, and solubility of bar soaps. The moisture content of the soaps ranged from 16.8% (Plantain) to 20.07% (Yam and plantain blend). The plantain ash soap recorded the lowest moisture content (16.8%), followed by yam ash soap (17.8%) and cassava ash soap (18.02%). The blended soaps exhibited moisture contents between 18.0% and 20.07% (Yam and Plantain blend). Standard commercial soaps typically contain 10 – 15% moisture (Olayinka et al., 2016), whereas traditional potassium-based African black soaps are hygroscopic and commonly retain 15–25% moisture (Edah et al., 2017). The relatively lower moisture content of the plantain ash soap suggests a firmer texture. In contrast, the higher moisture levels observed in the blended soaps, particularly the yam–plantain blend (20.07%), indicate improved solubility but may result in softness and faster wear during use (Maduelosi et al., 2025). This aligns with the work of Ogunsuyi and Akinnawo (2012), who found that improved soaps made from purified palm bunch ash had significantly higher moisture (29.05%) compared to traditional black soaps (9.53%), attributing the higher moisture to the hygroscopic nature of the potassium salts. Umeh (2024) also noted that soaps produced with local alkali (13.0% moisture) were softer than those made with industrial potassium hydroxide (8.2% moisture). Similarly, Nnyia et al. (2023) reported a moisture content of 15.1% for plantain biochar soap, concluding that such levels allow for stability without rapid water-induced deterioration. Nevertheless the moisture levels of the experimental soaps matched closely with the commercial control (18.3%) and fall within the acceptable range (~20–25%) specified by the Standards Organization of Nigeria (SON) for laundry soaps, although further drying may be required for commercial bar production.

Total Fatty Matter (TFM)

Total Fatty Matter (TFM) is an important quality parameter that influences the cleansing ability, lathering properties, and overall quality of soap. The Total fatty matter (TFM) values obtained were 72.0% for yam soap, 76.0% for cassava soap, 67.0% for plantain soap, and 87.6% for the yam – cassava – plantain blended soap. According to the Bureau of Indian Standards (IS 2888:2004), toilet soaps are graded based on their Total Fatty Matter (TFM) content: Grade 1 (76% min), Grade 2 (70% min), and Grade 3 (60% min). This classification system has been widely adopted in Nigerian quality assessments, as seen in Idoko et al. (2018), to categorize locally produced soaps. In this study, the Cassava soap (76.0% TFM) meets the requirements for a Grade 1 soap as defined by IS 2888:2004. The Yam soap (72.0%) falls within the Grade 2 category (70–76%), while the Plantain soap (67.0%) is classified as a Grade 3 or Laundry soap, consistent with the standards for lower fatty matter content (<70%). These TFM results are high when compared to other studies on indigenous soaps. For instance, Beetseh and Anza (2013) reported a TFM of 62% for cassava peel soap, lower than the 76.0% achieved here, suggesting that the extraction and saponification protocols used in this study were highly efficient. Similarly, Umeh (2024) reported a TFM of 72% for local alkali soap, which aligns with the yam soap result. The ternary blend's TFM of 87.6% is comparable to the high-quality avocado pulp/seed soaps formulated by Oloruntele et al. (2025), which achieved TFM values between 68.4% and 89.1%. This confirms that blending agricultural waste alkalis can produce premium-grade soaps that rival or exceed commercial standards.

Free Caustic Alkali (FCA) and Total Alkali

Free Caustic Alkali determines the harshness of the soap on the skin and fabric. The free caustic alkali (FCA) content of all formulated soaps ranged from 0.08% to 0.19%, which was below the Standard Organisation of Nigeria (SON) maximum limit of 0.20% for laundry soaps (SON, 2018). The ternary blend (0.19%) and the commercial control (0.18%) had similar FCA values, indicating that the amount of alkali used was well balanced with the oils during the saponification process. Total alkali followed a similar trend, ranging from 0.80% (Plantain) to 1.40% (Yam & Plantain), reflecting safe, highly efficient alkali consumption (Johnson et al., 2024). Alkalinity in soap is conventionally expressed as percentage free alkali (as Na_2O), with recommended limits of less than 0.2% for toilet soaps and less than 2.0% for laundry soaps in order to minimize the risk of skin irritation (Nangbes et al., 2014). This is a significant improvement over many indigenous soaps; for example, Oyekunle et al. (2021) found free caustic alkali levels ranging from 2.49% to 11.41% in local black soaps, which far exceed safe limits. Similarly, Beetseh and Anza (2013) reported a free caustic alkali content of 2.95% in their cassava soap. The extremely low values in this study compare favorably with the 0.02% free caustic alkali reported by Johnson et al. (2024) for high-quality black soaps made from palm kernel oil and plantain peel potash. Among the samples, the Plantain soap exhibited the lowest alkalinity value (0.80%), supporting its classification as the mildest and most skin-friendly formulation, consistent with its lower pH.

Foam Height

Foam height is an important quality parameter that reflects the lathering ability and cleaning performance of soap. The foam height of the formulated soaps ranged from 350 mL (Plantain) to 510 mL (ternary blend). The binary blends produced foam heights between 430 and 460 mL, while the ternary blend recorded the highest value (510 mL), exceeding the commercial control (450 mL). The improved foam production of the blended soaps is likely due to their higher Total Fatty Matter (TFM) content and the presence of potassium, which enhance foam formation and stability (Abdul et al., 2024).

Statistical Analysis of Physicochemical Properties

A one-way ANOVA showed significant differences among the soap formulations for all the physicochemical properties evaluated, including pH ($F(7,16) = 96.397$, $p < 0.001$), moisture content ($F(7,16) = 24.088$, $p < 0.001$), Total Fatty Matter (TFM) ($F(7,16) = 158.467$, $p < 0.001$), Free Caustic Alkali (FCA) ($F(7,16) = 14.012$, $p < 0.001$), Total Alkali ($F(7,16) = 33.393$, $p < 0.001$), and foam height ($F(7,16) = 37.316$, $p < 0.001$).

These results indicate that the type and combination of agricultural waste alkalis significantly influenced the quality of the formulated soaps. Appendix Table 3 presents the one-way Analysis of Variance (ANOVA), while Tables 4.2 and 4.3 summarize the ANOVA results and Tukey's Honestly Significant Difference (HSD) post hoc comparisons for the physicochemical properties of the soap formulations.

Table 4.2: Summary of One-Way ANOVA for the

Physicochemical Property	**F**	**df**	**p-value**	**Decision**
pH	96.397	7, 16	<0.001	Significant
Moisture Content (%)	24.088	7, 16	<0.001	Significant
Total Fatty Matter (%)	158.467	7, 16	<0.001	Significant

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Free Caustic Alkali (%)	14.012	7, 16	<0.001	Significant
Total Alkali (%)	33.393	7, 16	<0.001	Significant
Foam Height (mL)	37.316	7, 16	<0.001	Significant

Physicochemical Properties of Soap Produced from Agricultural Waste Alkali

Table 4.3 Tukey's Honestly Significant Difference (HSD) Post Hoc Comparison of the physicochemical Properties Soap Formulations

Soap Formulations	pH	Moisture Content (%)	Total Fatty Matter (%)	Free Caustic Alkali (%)	Total Alkali (%)	Foam Height (mL)
Control	10.2 ± 0.10^{a}	18.3 ± 0.40^{b}	70.2 ± 1.10^{c}	0.18 ± 0.02^{ab}	1.15 ± 0.05^{bcd}	450 ± 15.0^{b}
Yam	9.34 ± 0.08^{c}	17.8 ± 0.35^{bc}	72.0 ± 0.85^{c}	0.12 ± 0.01^{cd}	1.00 ± 0.04^{d}	380 ± 10.0^{de}
Cassava	9.80 ± 0.09^{b}	18.0 ± 0.42^{b}	76.0 ± 0.90^{b}	0.15 ± 0.02^{abc}	1.20 ± 0.06^{bc}	410 ± 12.0^{cd}
Plantain	8.61 ± 0.05^{d}	16.8 ± 0.30^{c}	67.0 ± 0.70^{d}	0.08 ± 0.01^{d}	0.80 ± 0.03^{e}	350 ± 10.0^{e}
Yam & Cassava	9.84 ± 0.11^{b}	18.0 ± 0.38^{b}	78.1 ± 1.05^{b}	0.14 ± 0.01^{bc}	1.10 ± 0.05^{cd}	430 ± 14.0^{bc}
Yam & Plantain	10.2 ± 0.12^{a}	20.1 ± 0.50^{a}	86.0 ± 1.20^{a}	0.18 ± 0.02^{ab}	1.40 ± 0.08^{a}	460 ± 16.0^{b}
Cassava & Plantain	9.81 ± 0.07^{b}	17.9 ± 0.32^{bc}	78.6 ± 0.95^{b}	0.16 ± 0.02^{abc}	1.15 ± 0.04^{bcd}	440 ± 15.0^{bc}
Yam, Cassava & Plantain	10.0 ± 0.09^{ab}	19.9 ± 0.45^{a}	87.6 ± 1.15^{a}	0.19 ± 0.02^{a}	1.30 ± 0.07^{ab}	510 ± 18.0^{a}

Values are presented as mean ± standard deviation (n = 3). Mean within the same column bearing different superscript letter differ significantly ($p < 0.05$) according to the tukey's HSD post hoc test

Tukey's HSD post hoc test was further used to identified the formulations responsible for these differences. For pH, the Plantain (8.61^{d}) and Yam (9.34^{c}) soaps had significantly lower values, whereas the ternary blend (10.00^{ab}) showed no significant difference from the commercial control (10.20^{a}) and the Yam–Plantain blend (10.20^{a}). For moisture content, the ternary blend (19.93^{a}) and the Yam–Plantain blend (20.10^{a}) recorded the highest values, while the Cassava soap and the Yam–Cassava blend were statistically similar to the commercial control (18.30^{b}). Total Fatty Matter was highest in the ternary blend ($87.63 \pm 1.15\%^{a}$) and the Yam–Plantain blend ($86.00 \pm 1.20\%^{a}$), both of which were significantly higher than the commercial control ($70.20 \pm 1.10\%^{c}$). For FCA, although significant differences existed, all formulations remained below the SON permissible limit, indicating acceptable soap quality. Similarly, the Yam–Plantain ($1.40 \pm 0.10\%^{a}$) and ternary ($1.30 \pm 0.10\%^{ab}$) blends recorded the highest Total Alkali values while remaining within acceptable limits.

Foam height was also significantly improved by blending, with the ternary blend (510 ± 10 mL[a]) producing significantly higher foam than the commercial control (450 ± 10 mL[b]), whereas the Yam–Plantain, Cassava–Plantain, and Yam–Cassava blends showed foaming performance comparable to the commercial soap. Overall, the results demonstrate that blending agricultural waste alkalis, particularly in the ternary and Yam-Plantain formulations, significantly enhanced the physicochemical properties of the soaps and produced products that were comparable to or better than the commercial control

Chemical composition of Ashes of Plantain, Cassava and Yam Peel

Elemental Oxide Profiling (EDXRF)

The effectiveness of ash in soap production is primarily influenced by its chemical composition, particularly the relative proportions of alkali metals, alkaline earth metals, and silica. The oxide composition of plantain, yam, and cassava peel ashes was determined using Energy Dispersive X-ray Fluorescence (EDXRF) analysis and shown in Table 4.4. The comparative elemental oxide composition of agricultural waste ash are displayed in Figure 4.2.

Plantain peel ash exhibited the highest potassium potential, with a potassium oxide (K_2O) content of 20.3%, confirming its suitability as a superior source of soluble alkali and validating its traditional use in indigenous soap production. This finding is consistent with Yahaya et al. (2012), who established the order of potassium content in agricultural residues as CPH > KPH > PP > OP, affirming plantain peel (PP) as a high-yielding source relative to others like orange peel. The high K_2O content indicates a tendency toward the production of soft, highly soluble potassium soaps, a characteristic noted by Olabanji et al. (2012), who found potassium to be the dominant metal (32.54%) in plantain ash

Yam peel ash also demonstrated strong suitability, with a K_2O content of 17.2%, closely comparable to plantain. This highlights yam peels as a viable alternative or supplementary feedstock. A distinguishing feature was its relatively high phosphorus pentoxide (P_2O_5, 6.56%), which may impart water-softening properties through sequestration of hardness ions. Yam ash contained the lowest CaO content (4.09%), advantageous for minimizing scum formation, while its SiO_2 level (15.2%) was similar to that of plantain

In contrast, cassava peel ash showed a markedly different profile, characterized by low K_2O (5.16%) and high CaO (22.7%), resulting in an inverted potassium-to-calcium ratio. This composition generally favors the formation of insoluble calcium soaps, which do not lather, potentially explaining the limited traditional use of cassava ash for soap making compared to plantain. However, the high quality of the cassava soap produced in this study (76% TFM) suggests that the extraction method successfully isolated the soluble potassium while leaving the insoluble calcium carbonate in the residue. This selective leaching process is critical, as highlighted by Shoppert et al. (2017) in the context of alkali recovery. Furthermore, Uduma et al. (2025) noted that a balanced K:Na ratio (typically 2:1 in agro-wastes) is vital for optimal saponification, and while this study focused on oxides, the extraction efficiency clearly overcame the raw material's limitations.

Table 4.4: Summary of the Comparative Elemental Oxide composition of Agricultural Waste Ashes

Oxide	**Chemical Formula**	**Plantain Peel (%)**	**Yam Peel (%)**	**Cassava Peel (%)**
Potassium Oxide	K_2O	20.3±0.11	17.2±0.25	5.16±0.11
Calcium Oxide	CaO	5.17±0.12	4.09±0/08	22.7±0.45
Silicon Dioxide	SiO_2	14.6±0.22	15.2±0.30	20.6±0.38

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Phosphorus Pentoxide	P_2O_5	3.32±0.05	6.56±0.14	4.09±0.07
Aluminium Oxide	Al_2O_3	6.21±0.10	6.65±0.09	6.90±0.15
Iron (II) Oxide	Fe_2O_3	0.97±0.02	1.41±0.03	2.76±0.06
Magnesium Oxide	MgO	0.93±0.03	0.93±0.02	0.92±0.01

All samples contained substantial SiO_2 (14 – 20%). While silica does not participate directly in saponification, soluble silicates can function as builders, enhancing soap hardness and stability. Azeez and Abegunde (2016) similarly found high silica content in plantain peel ash (16.7%) and noted its utility in aiding the drying of detergents. However, excess silica may cause grittiness, underscoring the importance of the effective filtration methods employed in this study (Umeh-Idika & Maduakor, 2013).

Statistical Analysis of Elemental Oxide

A one-way ANOVA showed significant differences in the elemental oxide composition of the three agricultural ashes. Significant differences were observed for K_2O ($F(2,6) = 6728$, $p < 0.001$), CaO ($F(2,6) = 4366$, $p < 0.001$), SiO_2 ($F(2,6) = 315.8$, $p < 0.001$), P_2O_5 ($F(2,6) = 955.1$, $p < 0.001$), and Fe_2O_3 ($F(2,6) = 1598$, $p < 0.001$), indicating that the botanical source significantly influenced the mineral composition of the ashes. In contrast, MgO showed no significant difference among the three ashes ($F(2,6) = 0.214$, $p = 0.813$).

Tukey's HSD post hoc test showed that all three ashes differed significantly in K_2O content, following the order: Plantain (20.30 ± 0.11^{a}) > Yam (17.17 ± 0.25^{b}) > Cassava (5.16 ± 0.11^{c}). For CaO and SiO_2, Cassava ash recorded the highest concentrations and formed a separate statistical group (^{a}), indicating significantly higher levels than Yam and Plantain ashes. However, all three ashes belonged to the same statistical group for MgO (^{a}), confirming that there was no significant difference in MgO content among them. These findings show that while the major mineral composition of the ashes varied with the agricultural waste source, MgO remained relatively constant across all three ashes

Table 4.5: Summary of One-Way ANOVA for the elemental oxide composition from Agricultural Waste Ash

Physicochemical Property	**F**	**df**	**p-value**	**Decision**
K_2O (%)	6728	2,6	<0.001	Significant
CaO (%)	4366	2,6	<0.001	Significant
SiO_2 (%)	315.8	2,6	<0.001	Significant
P_2O_5 (%)	955.1	2,6	<0.001	Significant
Al_2O_3 (%)	27.05	2,6	0.001	Significant
Fe_2O_3 (%)	1598	2,6	<0.001	Significant
MgO (%)	0.214	2,6	0.813	Not Significant

Table 4.6 Tukey's Honestly Significant Difference (HSD) Post Hoc Comparison of elemental oxide composition of Soap Formulations

Chemical Formula	**K_2O**	**CaO**	**SiO_2**	**P_2O_5**	**Al_2O_3**	**Fe_2O_3**	**MgO**
Plantain Peel (%)	20.3 ± 0.11^a	5.17 ± 0.12^b	14.6 ± 0.22^b	3.32 ± 0.05^c	6.21 ± 0.10^b	0.97 ± 0.02^c	0.93 ± 0.03^a
Yam Peel (%)	17.2 ± 0.25^b	4.09 ± 0.08^c	15.2 ± 0.30^b	6.56 ± 0.14^a	6.65 ± 0.09^a	1.41 ± 0.03^b	0.93 ± 0.02^a
Cassava Peel (%)	5.16 ± 0.11^c	22.7 ± 0.45^a	20.6 ± 0.38^a	4.09 ± 0.07^b	6.90 ± 0.15^a	2.76 ± 0.06^a	0.92 ± 0.01^a

Values are presented as mean ± standard deviation (n = 3). Mean within the same column bearing different superscript letter differ significantly ($p < 0.05$) according to the tukey's HSD post hoc test.

Heavy Metal Analysis and Safety Assessment

Heavy metal in the raw ash

The concentrations of selected heavy metals in the raw agricultural waste ashes were quantified to evaluate potential safety risks associated with the soaps produced from the extracted alkali. Lead (Pb), Zinc (Zn), Chromium (Cr), and Copper (Cu) were analyzed, and the results are presented in Table 4.7.

Table 4.7 Heavy Metal Concentrations in Agricultural Waste Ash (mg/kg)

SAMPLE CODE	**Pb(mg/kg)**	**Zn(mg/kg)**	**Cr(mg/kg)**	**Cu(mg/kg)**
CASSAVA PEEL ASH	24.4±0.17	26.5±4.16	0.10±0.10	27.3±2.01
PLANTAIN PEEL ASH	34.4±0.98	149±1.01	0.10±0.11	35.8±1.41
YAM PEEL ASH	31.4±0.17	131±1.56	1.80±0.04	67.3±1.35

Lead concentrations in the raw ash samples ranged from 24.4±0.17 mg/kg in cassava ash to 34.4±0.98 mg/kg in plantain ash. These values exceed the permissible limit of 10 mg/kg specified by the Standards Organisation of Nigeria (SON) and Health Canada for finished cosmetic products (Health Canada, 2012; SON, 2006). The elevated lead levels may be attributed to bioaccumulation from contaminated soils during plant growth or contamination introduced during the open-air combustion process (Sobukola et al., 2010). However, it is critical to note that these concentrations refer to the raw ash and not the final soap product. During alkali extraction, water selectively leaches soluble alkalis such as potassium carbonate, while heavy metals tend to form insoluble hydroxides or carbonates that remain in the solid residue. Olabanji et al. (2012) conducted a similar metal analysis on banana and plantain ash-derived alkalis and found that while the raw materials contained metals, the final alkali extracts had negligible lead levels (0.02~mg/kg), which were well below the hazardous limit. Research by Shoppert et al. (2017) further supports that alkali leaching processes effectively partition heavy metal impurities into the sludge, thereby purifying the liquid extract. In addition, the incorporation of oils during saponification further dilutes any residual metal content. Therefore, the partitioning of metals into the sludge and the dilution effect likely reduce the final lead content in the soap to within safe limits (<10 mg/kg). This stands in contrast to the findings of Oyekunle et al. (2021), who reported high lead levels (22.57~µg/g) in some indigenous black soaps, likely due to the use of unpurified raw materials or contaminated water sources.

Zinc concentrations were notably high in plantain and yam ashes, with values of 149±1.01 mg/kg and 131±1.56 mg/kg, respectively. Unlike lead, zinc is not considered toxic at these levels. Zinc compounds are widely used in dermatological formulations due to their anti-inflammatory and antimicrobial properties (Gupta et al., 2014). The elevated zinc content observed in plantain ash suggests that soaps derived from this source may possess added protective benefits against skin irritation and acne, potentially enhancing their functional and medicinal value. This aligns with the findings of Nnyia et al. (2023), who characterized plantain peel biochar and noted the presence of beneficial minerals that can aid in skin health.

Copper was detected at moderate concentrations ranging from 27.3±2.01 (Cassava ash) to 67.3±1.35 (Yam ash) across the ash samples. Although these levels are not considered toxic for rinse-off products, copper is known to act as a pro-oxidant. Its presence in soap formulations can catalyze the oxidation of unsaturated fatty acids, leading to rancidity and the development of unpleasant odors over time (Warra, 2011). This indicates that soaps produced from yam ash, in particular, may require antioxidant incorporation to improve shelf stability. However, comparative studies by Oyekunle et al. (2021) found copper levels in local soaps to be lower (20.71~µg/g for cocoa pod soap), suggesting that the leaching process may also naturally reduce copper transfer to the final product.

Chromium concentrations were negligible in all samples, with the highest value being 1.80±0.04 in yam ash. This is well below the safety threshold of 50 mg/kg typically cited for cosmetics (US FDA, 2016). Given that hexavalent chromium is a known skin sensitizer, the very low chromium content confirms that the soaps pose no risk of chromium-induced allergic contact dermatitis (Basketter et al., 2001). Furthermore, unlike the study by Oyekunle et al. (2021) which found high levels of Mercury (Hg) in indigenous soaps (106.50 – 273.58~mg/kg), the ashes in this study were free of such contamination, indicating a safer raw material source

Heavy metal in the finished soap

Post-saponification analysis showed a substantial reduction in heavy metal concentrations in the finished soaps, indicating that the soap-making process effectively limited the transfer of heavy metals from the agricultural ashes. Table 4.8 gives the heavy metal concentration in the finished soaps and different blends, wihle figure 4.4 shows the visual heavy metal concentration in the finished soaps and blends. Lead (Pb), zinc (Zn), and chromium (Cr) were not detected in any of the formulated soaps, with concentrations below the method detection limit (ND, <0.001 mg/kg). Copper (Cu) was detected at relatively low concentrations in selected formulations, including Yam soap (2.60 mg/kg), Cassava soap (5.01 mg/kg), the binary blends, and the recommended ternary blend (3.85 mg/kg). These concentrations are below recommended safety limits, suggesting that the finished soaps are safe for use. The reduction in heavy metals is likely due to their precipitation as insoluble compounds during alkali extraction and saponification, leaving most of the metals in the waste residue rather than the final soap (Olabanji et al., 2012; Shoppert et al., 2017). These findings indicate that the developed soaps comply with WHO and SON safety guidelines for cosmetic products.

Table 4.8 Heavy Metal Concentrations in formulated Agricultural Soap (mg/kg)

SAMPLE	Pb(mg/kg)	Zn(mg/kg)	Cr(mg/kg)	Cu(mg/kg)
Cassava soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	5.01 ± 0.09
Plantain soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	BD (<0.001)
Yam soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	2.60 ± 0.06
Yam & Cassava soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	6.46 ± 0.12

Yam & Plantain soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	2.20 ± 0.07
Cassava & Plantain soap	BD (<0.001)	BD (<0.001)	BD (<0.001)	5.01 ± 0.08

BD = Below the detection limit of the instrument

CONCLUSION

This study shows that agricultural wastes such as Plantain, Yam, and Cassava peels can serve as effective and sustainable sources of alkali for soap production. The results demonstrated that blending these agricultural wastes, particularly in the ternary formulation, produced high-quality Grade 1 soaps with significantly higher Total Fatty Matter (TFM) and better foaming properties than the commercial control. The study also confirmed that the soap production process is safe. Most heavy metals present in the raw ashes were removed during alkali extraction and saponification, resulting in finished soaps with either non-detectable or very low heavy metal concentrations that comply with WHO and Standards Organisation of Nigeria (SON) safety guidelines. Overall, converting agricultural wastes into soap provides an environmentally friendly and cost-effective approach to waste management while creating high-quality products that can support local industries and rural livelihoods.

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Table 3.1 Experimental Design of soap Formulation

Sample Code	Formulation Description	Composition
A	Control	Commercial Standard
B	Yam Peel Alkali Soap	100% Yam Peel alkali
C	Cassava Peel Alkali Soap	100% Cassava Peel Alkali
D	Plantain Peel Alkali Soap	100% Plantain Peel Alkali
E	Yam + Cassava Alkali Blend	50% Yam: 50% Cassava (v/v)
F	Yam + Plantain Alkali Blend	50% Yam: 50% Plantain (v/v)
G	Cassava + Plantain Blend	50% Cassava: 50% Plantain (v/v)
H	Yam + Cassava + Plantain Alkali Blend	33% Yam: 33% Cassava: 33% Plantain (v/v)

Table 4.1 Physicochemical Properties of Soap produced from Agricultural Waste Alkali

Soap Formulations	pH	Moisture Content (%)	Total Fatty Matter (%)	Free Caustic Alkali (%)	Total Alkali (%)	Foam Height (mL)
Control	10.2±0.10	18.3±0.40	70.2±1.10	0.18±0.02	1.15±0.05	450±15.0
Yam	9.34±0.08	17.8±0.35	72.0±0.85	0.12±0.01	1.00±0.04	380±10.0
Cassava	9.80±0.09	18.0±0.42	76.0±0.90	0.15±0.02	1.20±0.06	410±12.0
Plantain	8.61±0.05	16.8±0.30	67.0±0.70	0.08±0.01	0.80±0.03	350±10.0
Yam & Cassava	9.84±0.11	18.0±0.38	78.1±1.05	0.14±0.01	1.10±0.05	430±14.0
Yam & Plantain	10.2±0.12	20.1±0.50	86.0±1.20	0.18±0.02	1.40±0.08	460±16.0
Cassava & Plantain	9.81±0.07	17.9±0.32	78.6±0.95	0.16±0.02	1.15±0.04	440±15.0
Yam, Cassava & Plantain	10.0±0.09	19.9±0.45	87.6±1.15	0.19±0.02	1.30±0.07	510±18.0

Note: Values reflect Mean ± Standard Deviation of three replicates

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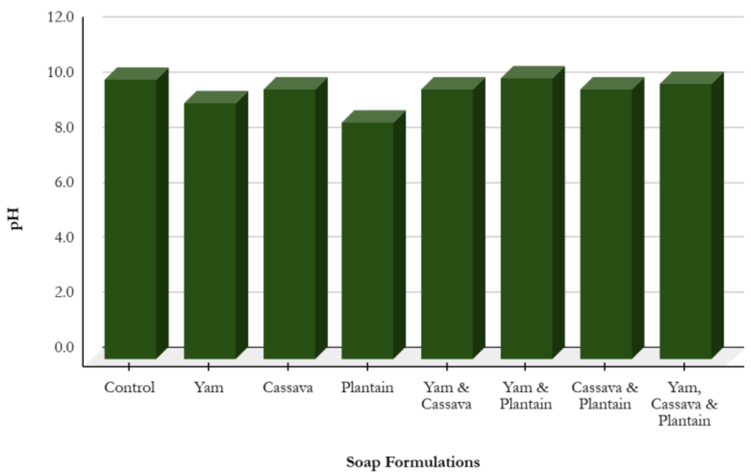
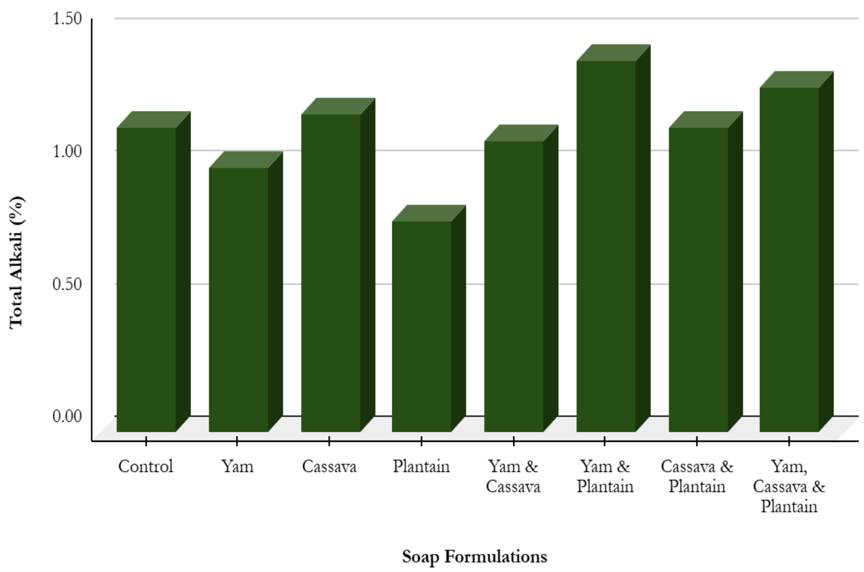
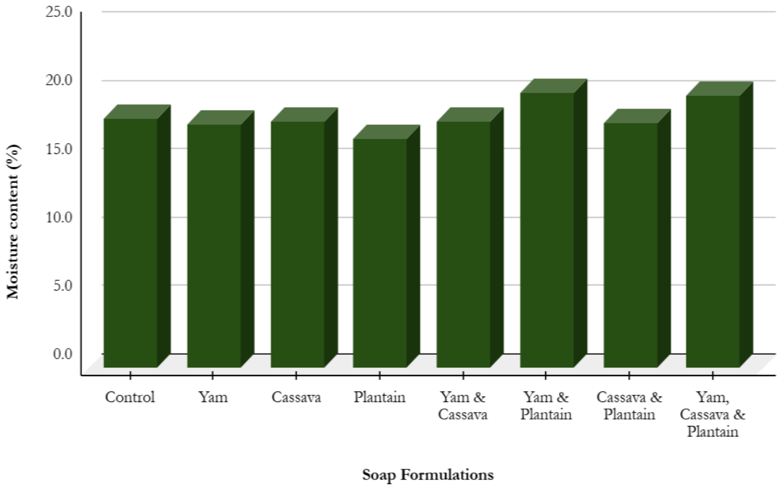
(A)



(B)



(C)



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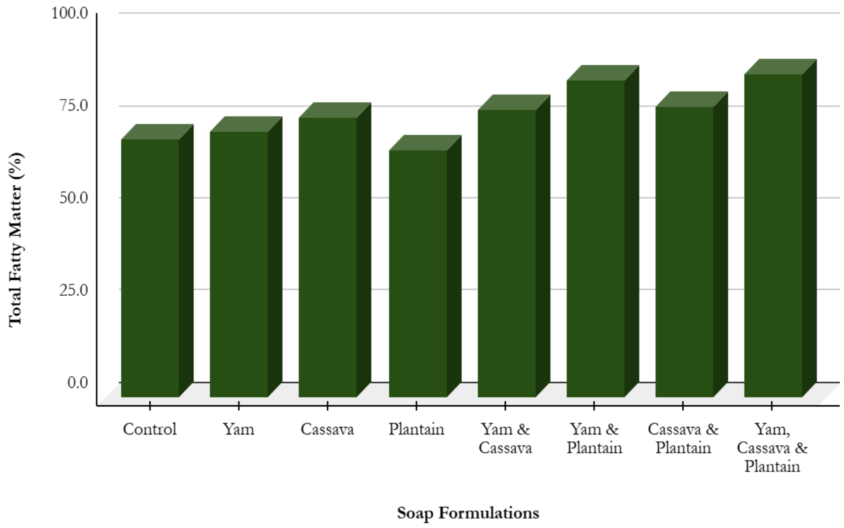
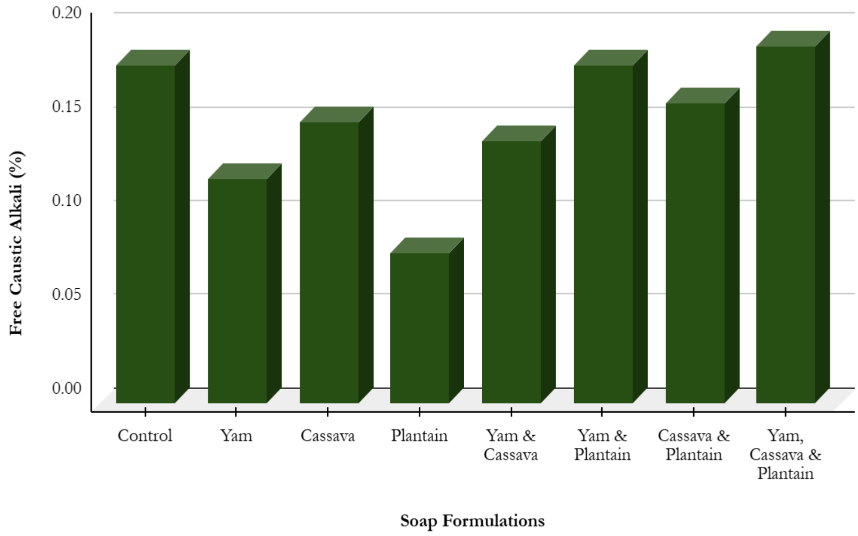
(D)



(e)



(f)

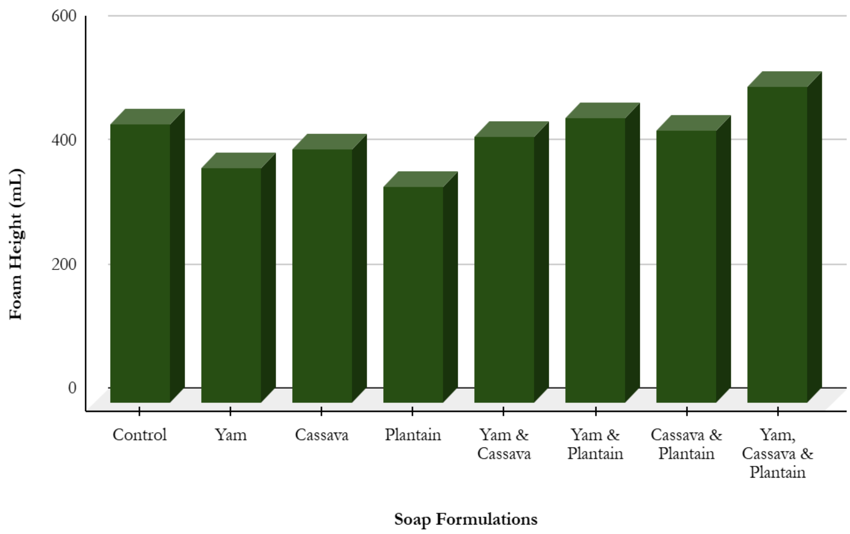


Figure 4.1 Physicochemical Properties of Soaps Produced from Agricultural Waste (a) pH **(b)** Total Alkali (%) **(c)** Moisture content (%) **(d)** Total fatty matter (%) **(e)** Free caustic alkali (FCA) **(f)** Foam height (mL)

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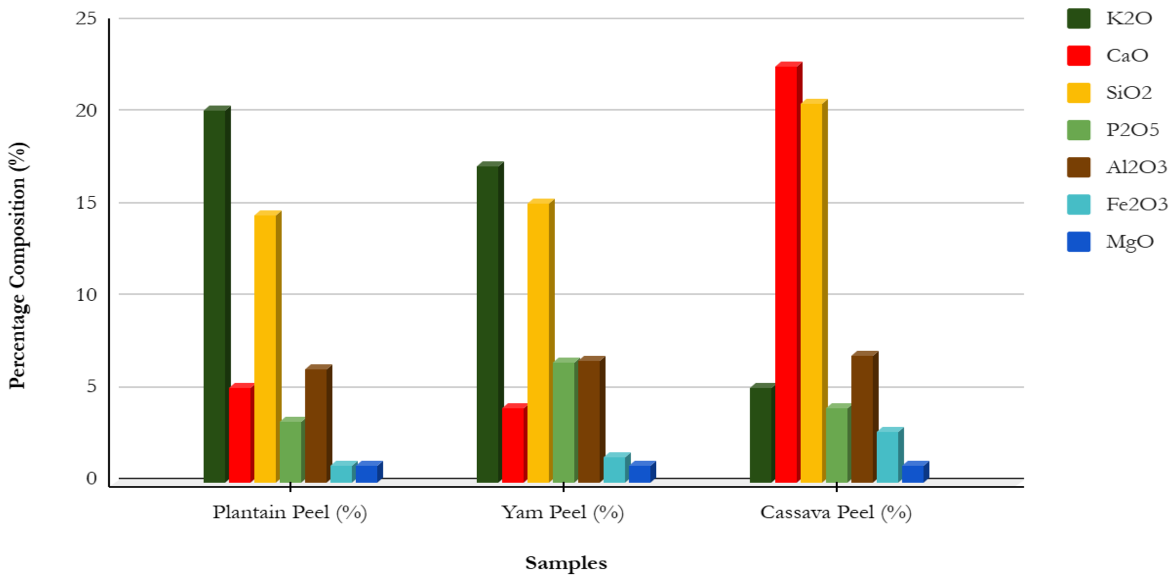

Figure 4.2 Comparative Elemental Oxide Composition of Agricultural Waste Ash

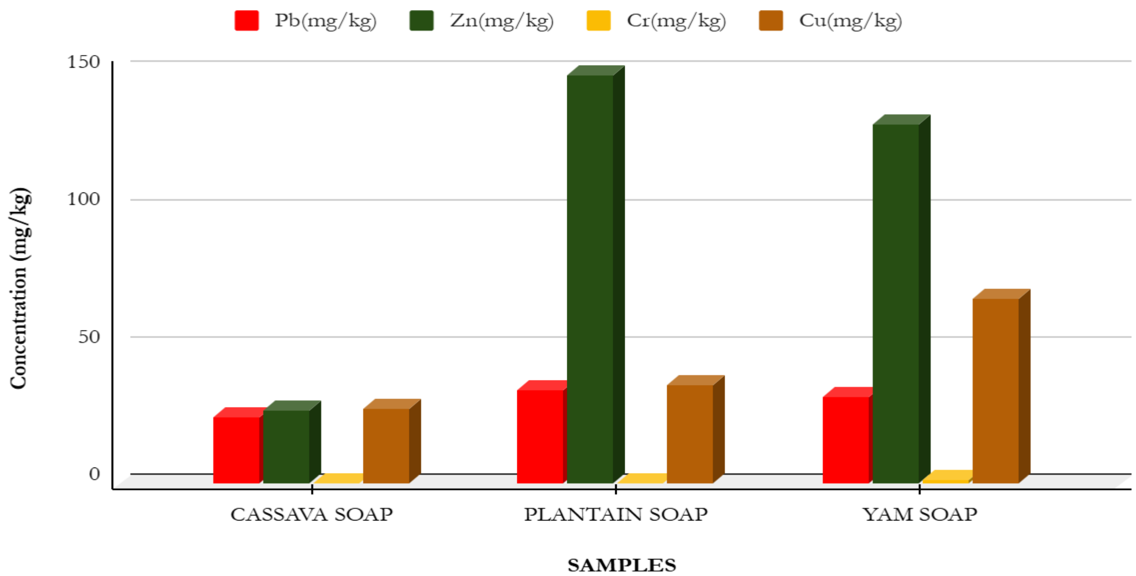

Figure 4.3: Concentration of Heavy Metals in Agricultural Waste Ash

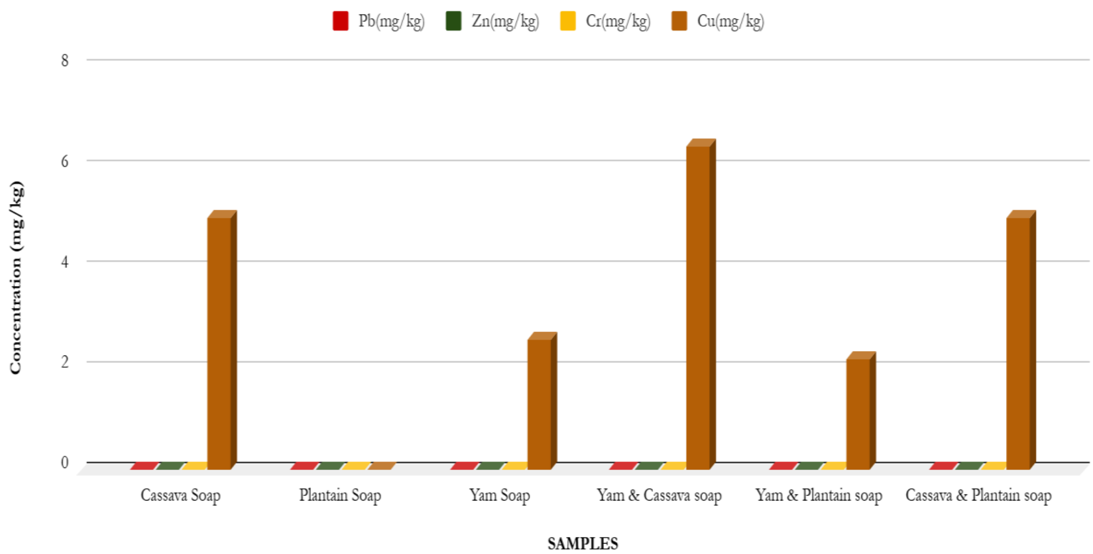

Figure 4.4 Heavy Metal Profile of formulated agricultural soap