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Production and Analysis of Eco-Friendly Soap from Millet, Maize, and Guinea Corn Stalks Using Blended Oils

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ABSTRACT
This study investigates the production of eco-friendly soap using potassium hydroxide (KOH) extracted from agricultural waste materials—specifically millet, maize, and guinea corn stalks ashes—combined with blended oils from palm kernel, hump, and beef tallow. The alkali extraction process was optimized to yield potassium-rich ash, and elemental analysis using X-ray fluorescence (XRF) revealed K_2O contents between 63.25%–68.52%. Flame photometry confirmed potassium concentrations suitable for lye production. Gas Chromatography-Mass Spectrometry (GC-MS) analysis of oil blends identified oleic acid as the dominant fatty acid. The formulated soaps exhibited Total Fatty Matter (TFM) between 70.8–75.3%, moisture content below 5%, and strong foaming capacity (>25 mm). Antimicrobial tests demonstrated significant inhibition zones against Staphylococcus aureus (15 mm), Staphylococcus epidermidis (19 mm), Trichophyton rubrum (29 mm), and Candida albicans (24 mm). The study establishes agricultural residues as viable alkali sources and highlights their role in producing high-quality, sustainable soaps with promising antimicrobial properties.The combination of 150 cm^3 60cm^3 of palm kernel oil, 50 cm^3 of hump oil, and 40cm^3 of beef tallow oil was shown to be the best. This oil blend was discovered to have an iodine number of 77.96±0.72 and a saponification number of 249.57±0.78, both of which are greater than the individual values. The study highlights the viability of agricultural waste as a sustainable source of alkali and demonstrates the potential of eco-friendly soap formulation with notable antimicrobial activity.

Keywords:
Agricultural waste, Anti-microbial activity, -blended oils, Lye and Physicochemical Analysis

INTRODUCTION

Soap has apparently been invented by accident 4,500 years ago. Animals were sacrificed to the ancient gods by burning them in burnfire (Taiwo *et al.*, 2008). When animal fats were mixed with the ash, they reacted with potassium carbonate contained in the ash, and soap was produced. Soap is a mixture of many different fatty acid's sodium salts. Soap can be prepared by heating oil with sodium hydroxide (Onyegbado *et al.*, 2002). Soaps are divided into two main types: toilet and industrial. There are a few definitions of soap. One of definitions, soap is an alkali metal salt of a long-chain fatty acid and is manufactured using vegetable and animal fats. Besides, soap is a chemical compound or mixture of chemical compounds forming from the interaction of fatty acids and alkaline solution. The alkaline solution, often called lye usually used in soap making are sodium hydroxide (NaOH) also known as caustic soda and potassium hydroxide (KOH). Usually, sodium hydroxide is used to make solid soap while potassium hydroxide is used to make liquid soap. NaOH and KOH are water soluble soaps which are different from those made from divalent metals such as calcium and magnesium, which are water insoluble (Berger *et al.*, 2003).

Soap production traditionally relies on industrial alkalis such as sodium and potassium hydroxides derived from mineral sources. Below is a representation of the soap molecule's structure:

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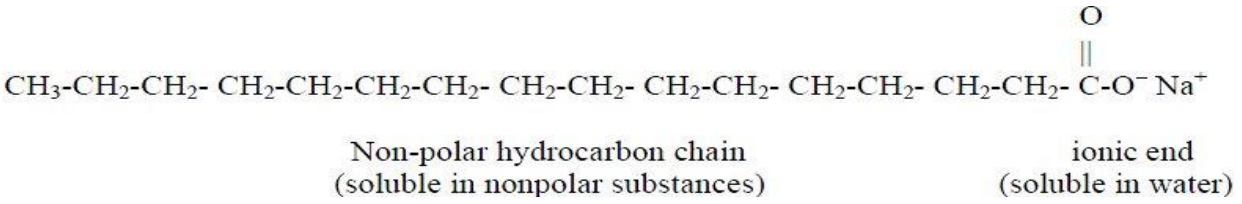

In developing regions, agricultural residues like millet, maize, and guinea corn stalks represent abundant but underutilized materials with potential as alkali precursors. This research explores a sustainable approach to soap formulation using lye derived from these residues and blended oils to reduce cost, promote environmental sustainability, and enhance antimicrobial quality.

The Chemistry of Soap

Soap making involves the hydrolysis of a triglyceride (fat or oil) using an alkaline solution usually **lye,** chemical name sodium hydroxide (Opeke *et al.*, 2003). Triglycerides are typically triesters consisting of 3 long-chain aliphatic carboxylic acid chains appended to a single glycerol molecule. This process of making soap is known as saponification (Prieto *et al.*, 2018). The common procedure involves heating animal fat or vegetable oil in lye (sodium hydroxide), therefore hydrolyzing it into carboxylate salts (from the combination of carboxylic acid chains with the cations of the hydroxide compound) and glycerol (Opeke *et al.*, 2003).

Fats/oils can also be saponified with calcium hydroxide, but this will usually result in liquid soaps. Soap is a chemical compound resulting from the saponification of fatty acid or glyceride with lye, for bathing, and cleaning purpose (kuntom *et al.*, 1996). A soap molecule has a long hydrocarbon chain with a carboxylic acid group end, which has ionic bond with metal ion, usually sodium or potassium. The hydrocarbon end is non polar which is highly soluble in non-polar substances and the ionic end is soluble in water (Opeke *et al.*, 2003).

Soaps have a cleaning action because they contain negative ions composed of a long hydrocarbon chains attached to a carboxyl group. The hydrocarbon chain has an affinity for grease and oil and the carboxyl group has an affinity for water. That is why soaps are mostly used with water for bathing, washing and cleaning. They are also used in textile industries for textile spinning (Opeke *et al.*, 2003).

There are many agricultural waste materials generated in homes and littered all over the environment. These materials include palm tree bunch, cocoa pod, plantain peels, banana peels, maize cobs, cassava peels and others. Some of these agricultural wastes like cocoa pod adversely affects soil fertility and so constitute environmental nuisance to man. However, they are potential viable sources which need to be harnessed for other uses to create wealth and to protect the environment. According to Adewuji *et al., 200*, several agricultural waste of vegetable origin yield a high potash when combusted (Opeke *et al.*, 2003).

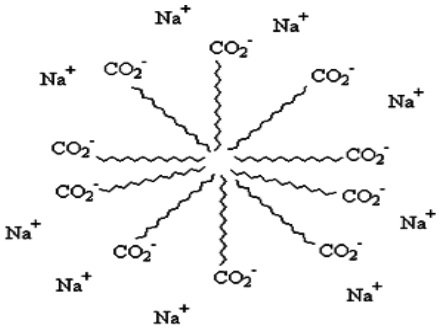

Diagram of Soap Micelle

Saponification

Saponification is an organic chemical reaction which soap is formed from the reaction between alkali and fat in order to cleave ester into carboxylic acid. Basically, triglycerides are reacted with sodium hydroxide to produce glycerol and a fatty acid salt. The triglycerides are most often animal fats or vegetable oils (Opeke *et al.*, 2003). During saponification process, sodium hydroxide is dissolved in water, and the oils are made into a warm liquid state, either by heating a liquid or melting a solid. Both chemical mixed until two stages are fully emulsified. When using sodium hydroxide, a hard soap will be produced. While when using the potassium hydroxide will result in a soft soap. Lipids that contain fatty acid ester linkages can undergo hydrolysis. This reaction is catalyzed by a strong acid or base. Saponification is the alkaline hydrolysis of the fatty acid esters (Opeke *et al.*, 2003).

MATERIALS AND METHODS

Sample collection and treatment

Sample collection and treatment of millet, maize and guinea corn stalk collected from Musawa Town in Katsina State. The sample dried using (DHG-9101-15A, laboratory dry oven, Changzhou, China) at 40 °C to a constants weight after which they were burned into ashes

using muffle furnace at 550°C for 5hr official method (Opeke *et al.*, 2003).

Extraction of Alkali from Maize, Millet and Guinea Corn Stalks

The sample was dried using (DHG-9101-15A, laboratory dry oven, Changzhou, China) at 40 °C to a constants weight after which they were burned into ashes using muffle furnace at 550 °C for 5hr official method (Opeke *et al.*, 2003). The ignited sample was ashed crushed, homogenized and sieved to remove large particles. The sieved samples (150g) was placed in distilled water, agitated for 5 minutes and allowed to stand for 12 hours. The slurry was then decanted and the filtrate was heated for 10 hours at 60°C and the bleached alkali was filtered using Whatman No 44 filter paper to obtain the extract. The molarity of the extracted alkali was determined by titrating against 0.1M *hydrochloric* acid using phenolphthalein indicator (Asiagwu, A. K. et al., 2013)

Blended Oil Ratio

The blended oil was transferred into a beaker and placed in water bath at 70 °C for 30 min to ensure *n*-hexane solvent was completely evaporated and the volume of the residual Blended oil was recorded and expressed as percentage oil yield according to equation.

$$\text{Oil yield}(\%) = \frac{\text{weight of oil}}{\text{weight of sample}} \times 100 \quad (1)$$

Determination of Specific Density

Blended oil (10 ml) was added into a pre-weighed measuring cylinder and the weight was measured. The weight of Blended oil was subtracted from weight of the oil and cylinder (Jimoh & Jimoh, 2021). The specific gravity of Blended oil was calculated from equation.

$$\text{Specific density (g/mL)} = \frac{W_1 - W_O}{V_O} \quad (2)$$

Determination of Saponification Value

The Blended oil, 2.0 g was transferred into a round bottomed flask containing 30.0 mL of 0.5 M ethanolic KOH and the flask was mounted on a condenser for 30 min to ensure the oil was completely dissolved. A blank was also set up using the same reagent but without the Blended oil. Both samples were refluxed for 1hr and allowed to cool. Next, 1 mL of phenolphthalein was added and the samples were titrated with 0.2 M HCl until the pink color of phenolphthalein disappeared indicating an end point (Paulin & Irene, 2019). The saponification value (SV) was calculated from equation.

$$\text{Saponification value (mgKOH/g)} = \frac{(B - S)\text{x M x } 56.1}{W} \quad (3)$$

Determination of Acid Value

A method reported by Adane (2021) was modified in this study. Blended oil, 10. 0 g was added to 100 mL of ethanol in a 250 mL beaker and the mixture was brought to boiling. After removing the heat, the mixture was titrated with 0.1 M KOH using phenolphthalein indicator until a permanent pink color was noted at the end point. The acid value (AV) was calculated from equation.

$$\text{Acid value (mgKOH/g)} = \frac{M \times C \times T}{W} \quad (4)$$

Determination of Iodine Value

For this study, a method of Mechqoq et al. (2021) was modified. Blended oil, 0.50 g was dissolved in 15 mL of carbon tetrachloride in conical flask (100 mL) and 5.0 mL of Wij's iodine solution was added. The solution was allowed to stand in a dark at 25 °C for 2 h and a solution of KI (5.0 mL) was added. The mixture was titrated with 0.1 M $Na_2S_2O_3$ using starch indicator. A blank titration without Blended oil was also carried out and the iodine Value (IV) was calculated from equation.

$$\text{Iodine value (g/100 g)} = \frac{12.69 \times M(V_1 - V_2)}{W} \quad (5)$$

Determination of pH Value

The soap, 10.0 g was dissolved in 100 mL of distilled water to form a 10% of soap solution. The electrode of a pH meter was immersed in the soap solution and the pH value of the soap was measured using a pH meter (JENWAY 3505, UK) (Zauro *et al.*, 2016).

Antimicrobial Susceptibility Test

The well diffusion method was used to test for antibiotic susceptibility in accordance with clinical laboratory standards or national committee guidelines. Using the serial dilution method, soap stock solutions were made at concentrations of 50 mg/ml, 25 mg/ml, 12.5 mg/ml, and 6.25 mg/ml. Mueller Hinton Agar plates were used to test the soap samples for the presence of antimicrobial properties. Using a sterile borer, a 6 mm diameter hole was punched into the media prior to the plates being infected with bacteria.

Characterization of Blended Oil by Gas Chromatography –Mass Spectrometry (GC-MS)

The GC–MS analysis of the palm kernel oil was carried out using a gas chromatography instrument (GC–7890B model) coupled to a mass spectrometer detector (MSD–5977A series) from Agilent Technologies (Santa Clara, CA, USA). The injector and detector temperatures of the ultra-inert capillary column: 190915–433UI HP–5MS (30 m x 0.25 mm x 0.25 µm) were set to 250 °C and 260 °C, respectively. A 0.5 µL sample (split ratio of 1/20) aliquot was injected and analyzed at 60 °C, held for 2 min and

then elevated to 260 °C at a rate of 3 °C/min. The helium carrier gas was set at 1.6 mL/min. Each component was expressed as a proportion of the total peak area. An electron ionization mode with a 70 eV energy was used for the mass-spectral detection. The resultant fragmentation pattern was compared to the National Institute of Standards and Technology (NIST) spectral library (Jacob *et al.*, 2022). Figure 3.3 shows the GC–MS Instrument used to characterize the fatty acids composition of Blended oil.

RESULTS AND DISCUSSION

Table 1. Physicochemical Properties of Blended Oils

Physicochemical parameter	Value obtained
Oil Ratio (%)	45.51 ± 1.10
Relative density (g/mL)	0.87 ± 0,03
Saponification value (mgKOH/g)	224.40 ± 3.13
Acid value (mgKOH/g)	3.25 ± 0.11
Iodine value (g I_2/100g)	14.72 ± 0.25

The chemical properties of the blended oil in table 1 revealed an oil yield of 45.51 ± 1.10%, a relative density of 0.87 ± 0.03 g/ml, a saponification value of 224.40 ± 3.13 mgKOH/g, an acid value of 3.25 ± 0.11 mgKOH/g, and an iodine value of 14.72 ± 0.25 g I_2/100g.

Table 2. : GC Data of fatty acids compositions of blended oil ratio PKO_{60}:HPO_{50}:BTO_{40}

S/No	Common Name	IUPAC Nomenclature	RT (min)	Peak Area	SI% to T.C.
1	Oleic acid	Cis-9-octadecenioc acid	31.00	3000	4.90
2	13-octadecenoic acid	Cis-13-octadecenioc acid	27.41	800	1.32
3	Elaidic acid	Trans-9-octadecenioc acid	29.36	2000	3.30
4	Petroselinic acid	Cis-6-octadecenioc acid	35.66	2000	3.300
5	Vaccenic acid	Trans-11-octadecenioc acid	36.00	8000	1.32
6	Caprylic acid	octanoic acid	18.00	500	0.82
7	Capric acid	Decanoic acid	19.50	1000	1.65
8	Lauric acid	Dodecanoic acid	37.05	7000	11.55
9	Myristic acid	Tetra decanoic acid	23.50	1500	2.47
10	Pentadecanoic acid	Pentadecanoic acid	24.50	800	1.32
11	Palmitoleic acid	Hexadec-9-enoic acid	29.36	2000	3.30
12	Palmitic acid	Hexadecenoic acid	32.00	5500	9.07
13	linoleic acid	°Ctadeca-9,12-dienoic acid	33.00	6000	9.90
14	Arachidic acid	Eicosanoid acid	36.50	8000	30.20
15	Gadoleic acid	Eicos-9-enoic acid	20.00	1500	2.47
16	Behenic acid	Docosanoic acid	32.02	5000	30.30

As shown in Table 2. respectively, the blended oils in this study contained different chemical compounds of which 16 fatty acids were detected using their retention times (RT). The primary fatty acid present in the oil was octadecenoic acid with various isomers comprising of oleic acid (31.00 min), 13-octadecenoic acid (27.44min), elaidic acid (29.36 min), petroselinic acid (35.66 min) and vaccenic acid (36.0 min). The fatty acid with the highest percentage similarity to target compound was oleic acid (78%), and all the five fatty acids excited as isomers.

Table 3: Optimization of ashing process

S/N	Sample	Temperature (°C)	Time (hr)	Mass (g)	KOH (g/dm3	Pkb	PH
1	MiS(Ao°C)	350	5	300	92.96	-0.22	9.9
2	MiS(50°C)	450	4	300	106.96	-0.28	9.9
3	MiS(100°C)	550	3	300	115.08	-0.31	10.1
4	MaS(A°C)	350	5	300	107.08	-0.28	9.7
5	MaS(50°C)	450	4	300	115.44	-0.35	9.7
6	MaS(100°C)	550	3	300	131.16	-0.37	9.9
7	GUS(A°C)	350	5	300	99.4	-0.25	10.2
8	GuS(50°C)	450	4	300	126.56	-0.36	9.8
9	GuS(100°C)	550	3	300	131.60	-0.38	10.4

Identification Key: MiS=Millet Stalk, MaS=Maize Stalk, GuS=Guinea Corn stalk, pH = potential hydrogen.

For the same quantity of ashes for maize stalk, millet stalk and guinea corn stalk increase in temperature decreases marginally KOH yield. This could be as a result of intermolecular forces between the ash particles.

Table 4. Chemical compositions of ashes millet stalk, maize stalk and guinea corn stalk(wt%**)**

S/N	**Oxide**	**Chemical Formular**	**Millet**	**Maize**	**Guinea Corn**
1	Potassium oxide	K_2O	17.12	21.95	14.17
2	Magnesium oxide	MgO	1.33	1.73	3.98
4	Calcium oxide	CaO	1.13	1.81	6.47
5	Silicon oxide	SiO_2	16.11	10.98	20.31
6	Ferric oxide	Fe_2O_3	0.67	0.32	0.99
7	Aluminum oxide	Al_2O_3	1.28	0.83	1.10
8	Phosphorus (v) oxide	P_2O_5	1.52	1.07	1.59
9	Cerium (v) oxide	CeO_2	1	1.12	4.87
10	Strontium oxide	CrO	4.77	4.84	4.84
11	Bromine	Br	0.94	1.00	1
12	Chlorine	Cl	4.05	3.13	1.17
13	Others		60.37	48.78	49.94

Table 5. Result on Flame Photometer Analysis of Ashes for Lye Production

S/N	STD	MEAN	READING	SAMPLE ID	K(ppm)
1	0.13	5.2	0.58	MiS	5.2
2.	0.16	9.3	0.54	MaS	9.3
3	0.08	2.4	0.52	GuS	2.4

Identification Key: MiS=Millet Stalk ,MaS=Maize Stalk, GuS=Guinea Corn Stalk

Flame Photometer Analysis of Ashes for Lye Production

The result of flame photometer analysis of ashes for K is consistent with XRFs result of similar ashes. The actual lye used in the saponification of the blended oil is KOH . The formulation of the soaps with a KOH, accounted for the moderate stability of the soaps (Wiyantoko *et al.*, 2021).

Table 6. Result on Ratio of the Blended Oils

S/N	SAMPLE	PKO	HPO	BTO	Total Volume (Cm^3)
1	A $^{\circ}C$	75	40	30	150
2	50 $^{\circ}C$	60	50	40	150
3	100 $^{\circ}C$	55	50	45	150

Identification Key: PKO= palm kernel oil, HPO=hump oil and BTO= beeftalow oil.

Ratio of the Blended Oils

The quality of the formulated soap was in the order of PKO_{75}:HPO_{40}:BTO_{30} > PKO_{60}:HPO_{50}:BTO_{40}> PKO55:HPO_{50}:BTO_{45}. Researches have shown that increase in saponification number increases soap quality and because PKO has the highest saponification value, increase in the volume of PKO will directly increase the soap quality and vice versa (Zauro *et al.*, 2016).

Table 7: Showing the Antibacterial activity of Different brands of soap against *Staphylococcus aureus*

Soaps	50 mg/ml	25 mg/ml	12.5 mg/ml	6.2 mg/ml
MiS (A°C)	14	8	7	4
MiS (50°C)	15	10	9	6
MiS (100°C)	15	11	10	6
MaS (A°C)	13	9	9	4
MaS (50°C)	14	10	9	5
MaS (100°C)	14	11	10	**6**
GuS (A°C)	12	8	8	6
GuS (50°C)	12	9	8	7
GuS (100°C)	13	10	9	8

Key: Mean ±Standard deviation

0 = Not detected, Control = Ciprofloxacin (*S.aureus*= 38mm) Dettol Soap = 35mm

Table 8: Showing the Antibacterial activity of Different brands of soap against *Epidermis aureus*

Soaps	**50 mg/ml**	**25 mg/ml**	**12.5 mg/ml**	**6.2 mg/ml**
MiS (A°C)	17	10	9	6
MiS (50°C)	18	10	10	5
MiS (100°C)	19	10	9	5
MaS (A°C)	15	8	6	6
MaS (50°C)	16	8	8	5
MaS (100°C)	16	9	8	6
GuS (A°C)	13	7	7	4
GuS (50°C)	15	8	7	4
GuS (100°C)	15	8	8	5

Key: Mean ±Standard deviation
0 = Not detected

Control = Ciprofloxacin (*S.epidermidis* = 50mm) Dettol Soap = 35mm
The results showed the antimicrobial activity of the different soaps which were labelled as (MiS,MiS50,MiS100,MaS, MaS50, MaS100, GuS, GuS50 and GuS100) with varying degrees of activities which were shown in (Table .7 and 8) .The antibacterial activity of soaps against S.aureus ranged from 4±0.47 mm to15 .33±0.mm and 4±0.47 to 19±0.05respectively presented in (Table 7); (Gajic *et al.*, 2022).

CONCLUSION

This research demonstrates the successful utilization of millet, maize, and guinea corn stalks ashes as alkali sources for soap production. The produced soaps met acceptable physicochemical standards and showed strong antimicrobial properties. The findings support the potential of agricultural waste valorization for sustainable, cost-effective, and environmentally friendly soap production, promoting circular economy principles in chemical manufacturing. The combination of, 60cm^3 of palm kernel oil, 50cm^3 of hump oil, and 40cm^3 of beef tallow oil was shown to be the best. This oil blend was discovered to have an iodine number of 77.96±0.72 and a saponification number of 249.57±0.78, both of which are greater than the individual values.

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