




EVALUATION OF SODIUM AND POTASSIUM SOAPS PREPARED FROM BEESWAX: ADDING VALUE TO MATERIAL

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ABSTRACT

Article History

Received: 28 April 2022

Revised: 24 June 2022

Accepted: 8 July 2022

Published: 26 July 2022

Keywords

Antimicrobial activity

Beeswax

Cleaning effectiveness

Foam stability

Lubrication

Valoration.

This study investigated the potential of producing quality soaps from beeswax with a view to widening its applications and providing alternative to the high demanding fats and oils. Its potassium and sodium soaps were prepared by hot method and their properties examined. The FTIR spectroscopy indicated carboxylate band at 1560.46 cm^{-1} for both soaps, which is, however, absent in the original beeswax, showing that metal ions associated with the COO^- ions in monodentate structures. The soaps have good qualities with respect to pH, foam ability, longevity, hardness, free alkali and total fatty matter, with the potassium soap having higher qualities. The foam ability and stability of the potassium soap were comparable to those of some commercial toilet and laundry soaps. The sodium soap, however, has better lubricating properties due to its unstable foam and low water solubility. Both prepared soaps showed lower antibacterial activity than tested antiseptic soaps but displayed almost the same activity with those of tested toilet soaps. The results showed that beeswax has the potential to serve as a good alternative to the conventional fats and oils in soap industry.

Contribution/Originality: Beeswax is rich in basic soap production ingredients: fatty esters and acids. There are no reports on exclusively beeswax-produced soap. This study showed that beeswax is a good potential alternative material to the conventional and high demanding fats and oils in soap industry.

1. INTRODUCTION

Soaps are salts of fatty acids, usually with minimum 12 carbon atoms and are mainly used as surfactants for washing, bathing, and cleaning. They are also used in textile spinning and are important components of lubricants. Metallic soaps are used as thickeners for oils and other organic media, as lubricants and release agents, as well as drying agents [1, 2]. Soaps are usually obtained by treating vegetable or animal oils and fats, which are composed of triglycerides, with a strongly alkaline solution. A useful by-product of this reaction is glycerin, which can be left in the soap product as a softening agent, or isolated for other uses [3]. Soaps are key components of most lubricating greases, which are usually emulsions of calcium or lithium soaps and mineral oil. Many other types of metallic soaps are also useful, including those of aluminum, sodium, and their mixtures. Such soaps are also used as thickeners to increase the viscosity of oils. In ancient times, lubricating greases were made by the addition of lime to olive oil [1, 2, 4]. The kind of alkali metal used determines the kind of soap product. Sodium soaps are firm, whereas potassium soaps are softer or often liquid. Lithium soaps tend to be hard, and they are usually exclusively in greases [1, 2]. Waxes are esters of fatty acids and fatty alcohols. They are usually mono- and di-esters containing one or two moles of long chain fatty acid esterified with one or two moles of high molecular weight

monohydroxy alcohol. Thus, when saponified, the soap together with fatty alcohols rather than glycerol is formed. Waxes usually consist of more complex mixture of different fatty acid portion of esters than oils or fats. This implies that more complex mixture of alkali salt of fatty acid would be obtained with wax. According to Mishra [5] mixture of oils produced better qualities of soaps than a single oil. This could be attributed to the enhanced variation in the carbon chains in the soap from a combination of oils or fats over that from an individual oil or fat. Consequently, rather than using a mixture of oil or fat, substances that have complex variable components hydrocarbon chain could serve as alternatives to these oils and fats which have high competitive utilities advantage. Beeswax is the natural substance that makes up the hexagonal structures of honeycombs made by honey bees in the hive. It has an acid, saponification and an ester values of 17-24, 89-103 and 72-79, respectively [6-8]. It is a complex mixture consisting of 27 to 40% monoesters, 9 to 23% hydroxymonoesters, 7 to 16% diesters, 3.9% hydroxydiesters, 11 to 16% long chain hydrocarbons, 12 to 18% free fatty acids, 4 to 8% of other substances and < 1% free fatty alcohols. The wax monoesters and hydroxymonoesters are derived fundamentally from palmitic, 15-hydroxypalmitic and oleic acids [9-13]. The complex wax esters contain 15-hydroxypalmitic acid or diols, which through their hydroxyl group, are linked to another fatty acid molecule [14]. However, the composition of beeswax may vary between and among the different families and different breeds of bees, because it is probable that wax production is closely related to bee genetics and diet [10, 15]. Nevertheless, beeswax is predominantly composed of about 80% or more fatty esters and fatty acids which are precursors for soap production. Aside its use for foundation comb in beekeeping, which is probably the main use, beeswax is used in cosmetics industries as an emulsifier and moisturizer, in pharmaceutical products, in candles production, et cetera [7, 8, 10]. Apart from its use as a moisturizer in palm oil's soap, there is no report on application beeswax in soap making. Majority of this natural material is, however, disposed as waste after extraction of its honey content. Beeswax is biodegradable and does no harm to the environment; it has a pleasant smell, and a pleasant yellow color. It has also been reported to be active against growth of microorganisms such as *E. coli*, *Aspergillus fumigatus*, *S. aureus* and *C. albicans*, etc [6, 16]. These characteristics are expected to make beeswax a good candidate in soap making. Furthermore, beeswax is expected to give harder bar soaps with good conditioning quality, due to its high degree of saturation, than those from conventional oils and fats. This attribute could be an advantage in producing sodium or potassium soap that could serve lubricating purpose and therefore, an alternative to metallic soaps obtained from lithium, magnesium, calcium and transitional metals. Moreover, it would be an added value to beeswax which is usually discarded as waste, and as alternative to the fats and oils which have high competitive advantage in many areas of applications. Based on the foregoing, this study was designed to prepare sodium and potassium soaps from beeswax, investigate their physiochemical properties as well as their antimicrobial activity with a view to expand the scope of applications of this underutilized material.

2. MATERIALS AND METHODS

2.1. Materials

The honeycombs were obtained from a beekeeper in Ondo, Nigeria. The chemicals and their assays and manufactures include: NaOH (96%, Burgoyne BurBridges & co, India); KOH (85%, Nice Chemicals Pvt Ltd, India); Methanol (99%, Qualikems); Ethanol (99%, Qualikems); Glacial acetic acid (99.5%, Qualikems); HCl (36%, Qualikems); CHCl₃ (99.7%, Qualikems); Na₂S₂O₃ (99%, Qualikems); H₂SO₄ (98%, Qualikems); HNO₃ (70.5%, Qualikems); BaCl₂ (Guangdong Guandhua Chemical Factory Co Ltd, China); Starch (Burgoyne BurBridges & co, India); ICl (99%, Omkar Speciality Chemicals PVT LTD, India); KI (99%, Burgoyne BurBridges & co, India); Phenolphthalein (Loba Chemie Pvt Ltd).

2.2. Extraction of Beeswax from Honeycomb

The honeycombs were packed in a clean cotton cloth (felt) and dipped in hot water in a stainless bowl regulated at 65–70°C on a thermostatic hot plate. The beeswax melted out through the felt onto the hot water leaving the impurities behind. The floated hot beeswax was allowed to cool to room temperature and the solid was then removed from the water. The process was repeated until there was no trace of impurity in the obtained beeswax.

2.3. Saponification Procedure

A 100 g of beeswax was melted. The melted sample was cooked with gradual addition of 4 M NaOH regulated at about 65–70°C on thermostatic hotplate and with steady stirring. The cooking continued until the liquid system formed a solid material upon further addition of the NaOH solution, leaving out unreacted alkali solution. Heating source was withdrawn and the whole system was allowed to stand for 30 minutes. Thereafter, the soapy solid material was rinsed with 120 ml distilled water in three batches to remove excess adhering NaOH. The soap was then filtered using a clean felt to remove most of the liquid still present. The process was repeated with 4 M KOH solution. The products obtained were dried to constant weight in an oven at 80°C.

2.4. Fourier Transformed Infrared Spectrophotometry Analysis

The starting beeswax and the prepared soap samples were analysed for the variation in their compositions using Fourier infrared spectrophotometer (Shimadzu FT-IR-8400S).

2.5. Determination of Chemical and Physical Parameters of the Beeswax and Soap Samples

The chemical and physical parameters determined for the beeswax and/or the soap samples were in triplicate. These parameters include:

Saponification value: The Saponification value (SV) of the beeswax was determined by adding 50.0 mL of 1 M ethanolic KOH to 3.0 g beeswax and the mixture was refluxed for 120 minutes at 70°C. The solution was cooled to room temperature and titrated against 0.5 M HCl to phenolphthalein end point. Blank determination was also carried out. Equation 1 describes the calculation for the SV.

$$SV = \frac{(B - S) \times M \times 56.1}{\text{Weight of sample}} \quad (1)$$

B and S are the volume of titrant (mL) for the blank and sample titrations, respectively. M is the molarity of HCl and 56.1 is molecular weight of KOH.

Acid value: To determine the acid value (AV) of the beeswax, 2.0 g of the beeswax sample was dissolved in 50 mL neutralized 99% methanol and warm until melted. The resulting solution was titrated against 0.5 M methanolic KOH using phenolphthalein indicator until a permanent, faint pink color was obtained. Equation 2 was used to obtain the free fatty acid value.

$$AV = \frac{\text{titre value (mL)} \times M \times 56.1}{\text{Weight of sample}} \quad (2)$$

M is the molarity of KOH solution. The difference between the SV and the AV gave the ester value.

Iodine value: The iodine values (IV) of the beeswax and the soap samples were determined using the Association of Official Analytical Chemist (AOAC) [17] description of Wijs' method for fats and oil with slight modification. One gram of beeswax sample was dissolved in 30 mL CHCl_3 followed by addition of 25 mL of 25% ICl solution (in glacial acetic acid). The mixture was left in the dark for 45 minutes, followed by addition of 25 mL of 10% KI solution and 100 mL distilled water. The resulting solution was titrated against 0.1 M $\text{Na}_2\text{S}_2\text{O}_3$ solution till the color became pale yellow after which 2 mL of starch solution was added (as indicator) and the titration was continued until the blue color just disappeared. The procedure was repeated for the prepared soaps. Blank determination was also carried out alongside the samples. Equation 3 shows how the IV was calculated.

$$IV = \frac{(B-S) \times M \times 126.9 \times 10^{-1}}{\text{Weight of sample}} \quad (3)$$

B and S are as defined in Equation 1. M is the molarity of $\text{Na}_2\text{S}_2\text{O}_3$ and 126.9 is the molecular weight of iodine.

Peroxide Value: The peroxide value (PV) of the beeswax sample was determined by dissolving 1.0 g of beeswax sample in 30 mL chloroform/glacial acetic acid mixture (2:3 v/v). About 1 mL of freshly prepared saturated KI solution was added. Distilled water (30 mL) was added, shaken and titrated slowly with 0.01 N $\text{Na}_2\text{S}_2\text{O}_3$ till the color changed to pale yellow. Then 0.5 mL of 1% starch indicator was added and the titration continued till the disappearance of the blue color. The procedure was repeated for the blank solution. Equation 4 works out the PV.

$$PV = \frac{(S - B) \times M \times 1000}{\text{Weight of sample}} \quad (4)$$

B and S are as defined in Equation 1. M is the molarity of $\text{Na}_2\text{S}_2\text{O}_3$ solution.

pH value: The pH values of the soaps were determined by dissolving 2 g of each of the soaps in separate 20 ml distilled water (10% soap solution) and shaken well [18]. The resulting soap suspensions were allowed to stay for 15 hours before the pH meter (Hanna Instrument) was inserted into small plastic tubes containing the various soap suspensions, and the readings were recorded. The pH meter was calibrated using a buffer solution of pH 6.86 and 10.01 before use on the soap solutions. **Moisture content:** The soaps' moisture contents were determined by heating 5 g of each soap sample in crucible at 103°C in an oven to a constant mass. Equation 5 was employed in calculating the percentage moisture content.

$$\text{Moisture content (\%)} = \frac{WS_b - WS_a}{WS_b} \quad (5)$$

Where, WS_a and WS_b are weights of soap sample after and before drying respectively.

Test for cleaning effectiveness: The cleaning effectiveness of the prepared soaps was examined and compared with those of commercial laundry (Canoe) and toilet (Lux) soaps. Four strips of filter paper were stained with 2 drops of groundnut oil, and they were immersed in the different tubes, each contained 2% of a soap solution in tap water. The four tubes were shaken vigorously for 3 minutes in a centrifuge. The filter papers were removed and rinsed with enough tap water and their levels of cleanliness were observed. **Foaming power and foam stability:** A 50 ml of each of different concentrations (0.5, 1, 1.5, 2, 2.5, 3 and 4%) of the prepared soaps solutions were shaken vigorously in a 250 ml measuring cylinder for 1 minute and the foam volumes were noted after 1 minute of settlement. For the foam stability, 50 ml each of 4% soap solutions (2 g of each of the soaps dissolved in 50 ml distilled water) was transferred into a 250 ml measuring cylinder. The mixture was shaken vigorously for about 1 minute. The foams generated were allowed to settle and their levels were noted after 1, 6, 20, 30, 40, 50 and 60 minutes. The foam stability was expressed as a ratio of the foam volume V_t at time t in minute to that at 1 minute V_0 , (V_t/V_0). For comparison, the process was repeated for two commercial toilet and one commercial laundry soaps.

Free caustic alkali: The method used by Legesse [19]; Boadu, et al. [20]; Ashrafy, et al. [21] was modified and adopted. A 2.5 g of each of the soaps was dissolved in warmed 40 mL ethanol. Few drops of phenolphthalein indicator and 10 mL of 20% BaCl_2 were added to each soap solution. The resulting solutions were titrated against 0.05 M H_2SO_4 to colorless solutions. Equation 6 shows how the free caustic alkali (FCA) value was obtained.

$$\text{FCA} = \frac{0.31}{W} \times V_A \quad (6)$$

Where, V_A = Volume of acid added; W = Weight of soap.

Total alkali content: Total alkalinity is the total alkaline material present in soap. It is expressed as a percentage of sodium hydroxide or potassium hydroxide according to whether sodium or potassium soaps are concerned. It was determined by titrating excess acid contained in aqueous phase with standard volumetric NaOH solution. A 5 g of each soap sample was mixed with 100 mL of neutralized ethanol and 5 mL of 1 N H_2SO_4 (aq) solution was added and then heated till the soap sample dissolved. The flask was gradually cooled to room

temperature, and the remaining sulfuric acid (after hydrolysis and neutralization of all alkaline components in soap) was estimated by back titrating test mixture with standard 1N NaOH using phenolphthalein indicator. The total alkali value was obtained using Equation 7 [19]:

$$\% \text{ Total Alkali} = \frac{V_A - V_B}{W} \times 3.1 \quad (7)$$

Where, V_A = volume of acid added in the experiment, V_B = volume of base at the end point, w = weight of soap used in the experiment. Total fatty matter: Slightly modified method of Betsy, et al. [22] was used. A 2.5 g of each soap sample was dissolved in 60 mL hot water. About 30 mL of 0.5 N HNO_3 was added to make it acidic. The mixture was heated until fatty acids floated as a layer above the solution. It was cooled in ice water to solidify the fatty acids. The fatty acids were separated and the aqueous solution was treated with 40 mL CHCl_3 to remove the remaining fatty acids. The separated fatty matter was mixed together, solvent was evaporated and the yield noted. Equation 8 presents the calculation for the total fatty matter content [22]

$$\% \text{ total fatty mater} = \frac{(y - x)}{\text{Weight of soap sample}} \times 100 \quad (8)$$

Where x and y are the weight of the dish, and the combined weight of the dish and soap after drying, respectively. The soaps were also observed for color, hardness, longevity, odor and solubility.

3. RESULTS AND DISCUSSION

The cooking period of the beeswax with each of the alkaline solutions took about 50 minutes.

3.1. Fourier Transformed Infrared Spectroscopy Characterization

The Fourier transform infrared (FTIR) spectroscopy spectra for the beeswax and prepared soaps are shown in Figures 1-3. The spectra indicated the presence of asymmetric carboxylate (COO^-) ions band at 1560.46 cm^{-1} in both soaps and symmetric carboxylate (COO^-) ions band at 1423.51 cm^{-1} and 1421.58 cm^{-1} for sodium and potassium soaps, respectively (Figure 2 and 3). These bands are however, absent in the original beeswax (Figure 1). This shows that the potassium and sodium ions associated with the COO^- ions in the monodendate structures and confirmed successful saponification. In addition, a band at about 1720 cm^{-1} (carbonyl stretch of fatty acid) appearing as a shoulder to the peak at around 1735.99 cm^{-1} (carbonyl stretch of ester) in beeswax spectrum (Figure 1); this band together with the ester stretch are absent in spectra of the two soap samples. These further show that the fatty acids and fatty esters in the starting material had been converted to metal carboxylates, and an evidence of complete saponification. However, the similar bands at around $3462\text{-}3452$, $1190\text{-}1016$ and 719 cm^{-1} could be attributed to OH stretch (of alcohol), C-O stretch, and long-chain (CH_2) group, respectively.

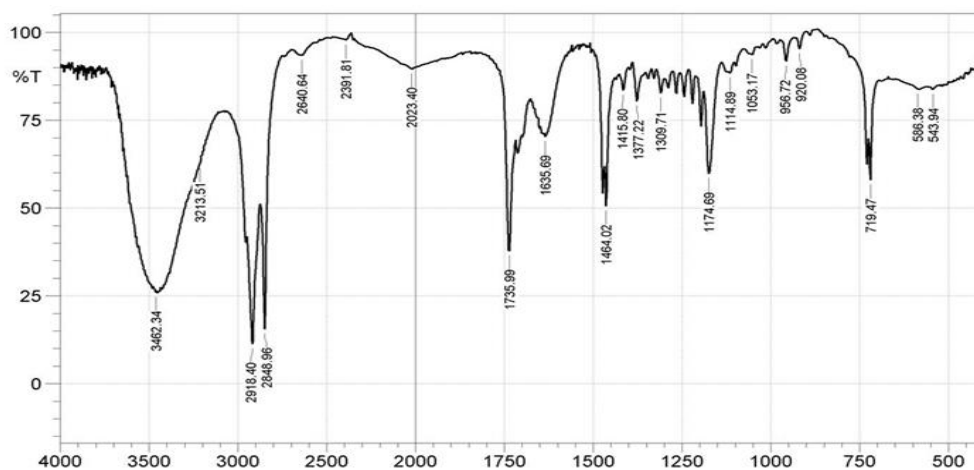


Figure 1. FTIR spectrum of the beeswax sample.

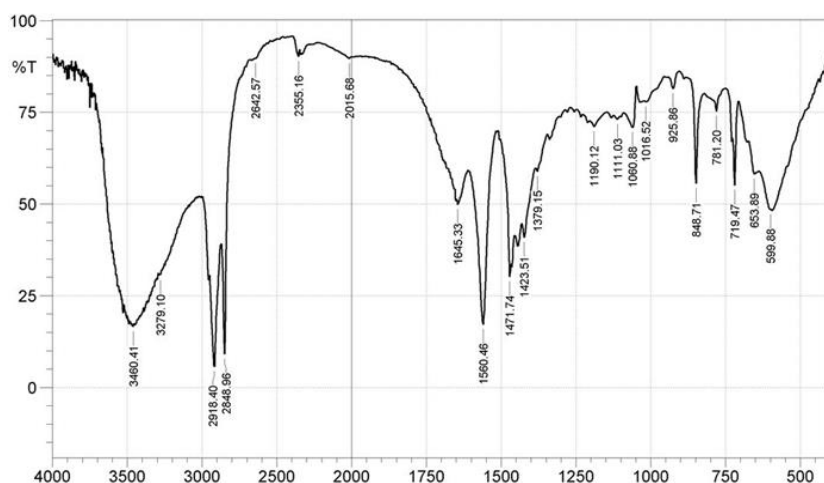


Figure 2. FTIR spectrum of the sodium soap sample.

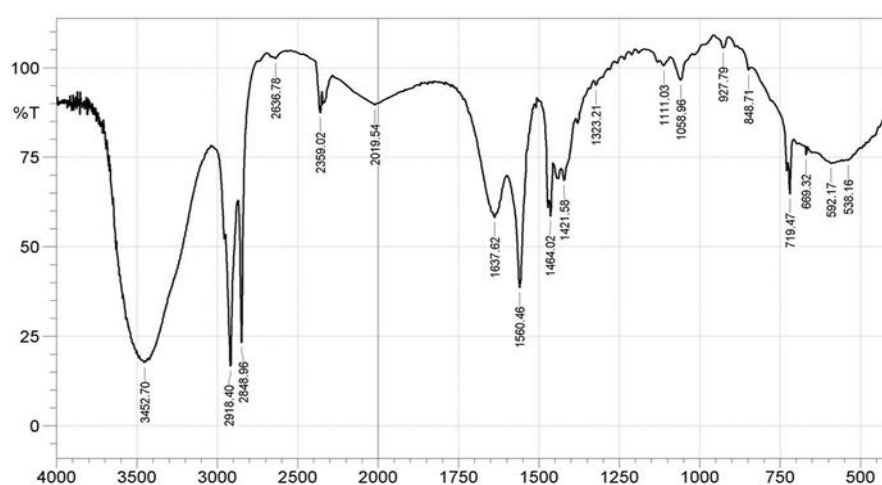


Figure 3. FTIR spectrum of the potassium soap sample.

3.2. Physicochemical Properties of the Beeswax and Soap Samples

Table 1 presents the physicochemical properties of the beeswax and the soap samples. The soaps produced good suds and gave pleasant smell. The potassium soap has better solubility and foaming qualities than the sodium soap. Moreover, the soaps have good cleansing property.

Table 1. Physico-chemical properties of beeswax and prepared soap samples.

Parameter	Beeswax	Soap type	
		Na	K
Color	Yellow	Cream	Cream
Saponification value (mg KOH/g sample)	95.29 ± 0.33	-	-
Acid value (mg KOH/g sample)	18.58 ± 0.31	-	-
Ester value (mg KOH/g sample)	76.71 ± 0.26	-	-
Peroxide value (millimoles/kg sample)	0.00 ± 0.00	-	-
Moisture content (%)	0.00 ± 0.00	9.60 ± 0.25	10.25 ± 0.55
pH (2 g soap/20 ml deionized water)	-	9.12 ± 0.01	9.01 ± 0.01
Solubility (2 g soap/20 ml deionized water)	Insoluble	Fair	Moderate
Foaming ability (2 g soap/20 ml tap water)	-	Good	Good
Foam stability	-	Poor	Good
Iodine value (grams of I ₂ /100 grams of soap)	9.45 ± 0.14	8.25 ± 0.04	8.62 ± 0.02
Hardness (to touch)	Hard	Hard	Hard
Cleansing power	-	Good	Good
Longevity	-	High	High
Total fatty matter (%)	-	81.75 ± 1.50	84.34 ± 0.88
Free caustic alkali (%)	-	0.09 ± 0.01	0.06 ± 0.01
Total alkali content (%)	-	0.31 ± 0.03	0.28 ± 0.01

Note: Values are mean ± Standard deviation, n = 3.

pH value: The mean pH value of NaOH and KOH soaps are 9.12 and 9.01 respectively, for 2 g of the soap in 10 ml of deionized water. Alkaline pH value for soap in aqueous solutions is not unexpected because it is the salt of a weak acid (fatty acid) and a strong base. The values fall in the lower limit of pH range (9.0 – 10) of most commercial soaps [23]. High pH value of soap is an indication of incomplete hydrolysis from the saponification reaction, and the harshness of soap resulting from high pH could be reduced or overcome by the addition of excess fat or oil or any other super fatting agent [19, 21, 24]. Thus, the moderate pH of the soaps in this study could be attributed to complete hydrolysis and the presence of fatty alcohols, which are not separated from the soaps, acting as fatting agent. The values obtained in this study are lower than those reported for used coconut oils [19] many commercial soaps [21] cotton seed oil [24] and toilet soap prepared from neem seed oil [25] comparable to that reported for Shea butter soap [20] but higher than those reported by Warra, et al. [26] for some triacylglycerol sources.

Iodine value (IV): The IV (or iodine number) is the mass (in g) of iodine absorbed for every 100 g of a material, and it gives a measure of the degree of unsaturation of a substance. The two soap samples have low iodine numbers Table 1 which imply low degree of unsaturation. This observation is reflected in their high hardness and longevity. Also, the soaps would be stable to oxidation and have long shelf lives since the low iodine values connote low number of C=C double bonds susceptible to oxidation [19, 27].

Total Fatty Matter (TFM): This is a very important property as it measures the tenderness of soaps. Its value reduces with high moisture content and presence of unreacted alkali in the mixture [21, 28]. The kinds and quantities of the employed fatty materials, and the saponification method also influence the TFM values [21]. The mean total fatty matters (TFM) of the prepared sodium and potassium soaps were found to be 81.74% and 84.34%. These high values could be attributed to low moisture content and free alkali as well as the presence of fatty alcohols that were not separated from the metal carboxylates. These soaps would be suitable for dry skin as TFM above 80% re-hydrates the skin, making it smooth [21, 25]. In addition, the fatty alcohol content within the soap would act as a lubricant on the skin.

Free Caustic Alkali: Free caustic alkali specifies the abrasiveness of any soap [29]. It is the amount of alkali free to counter and avert the soap from becoming oily. In this study, the mean free alkali obtained were 0.09% (sodium soap) and 0.06% (potassium soap). Excess free caustic alkali causes skin itching [21]. The observed values showed that the prepared soaps would be saved to use.

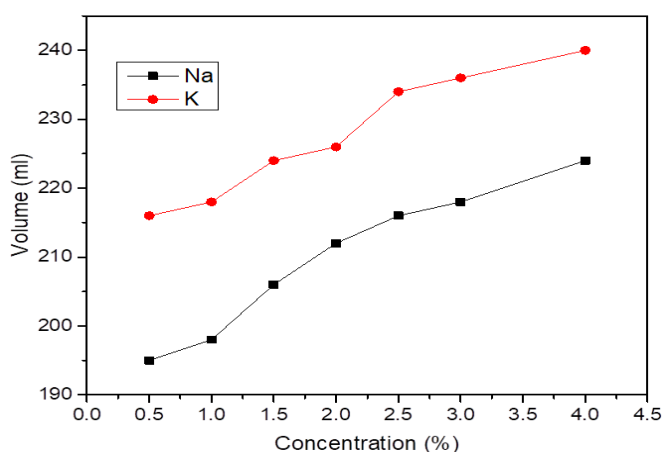


Figure 4. Foam volume by sodium and potassium derived surfactant concentrations.

Foaming power and foam stability: The two soap samples produced good foam with different concentrations (Figure 4). The foam was up to 224 mL (for sodium soap) and 240 mL (for potassium soap) level of 250 mL measuring cylinder for a 2 g soap/50 mL distilled water. These levels are comparable to those obtained for two commercial toilet soaps (Lux: 205 mL; Premier: 240 mL) and much higher than that for a commercial laundry soap (Canoe: 176 mL). However, Figure 5 shows that the stability of foams produced by the prepared soaps was poorer

than those for commercial toilet soaps, with sodium soap giving the poorest stability with many air spaces. The potassium soap foam's stability is similar to that laundry soap (Canoe). It is reported that in all metal working sectors as well as in the metal cutting field, the formation of stable foam on the processing fluids is an undesirable phenomenon [3]. Thus, the sodium soap could find application as a low foaming emulsion. This characteristic could be linked to its low solubility water and low surface tension [3].

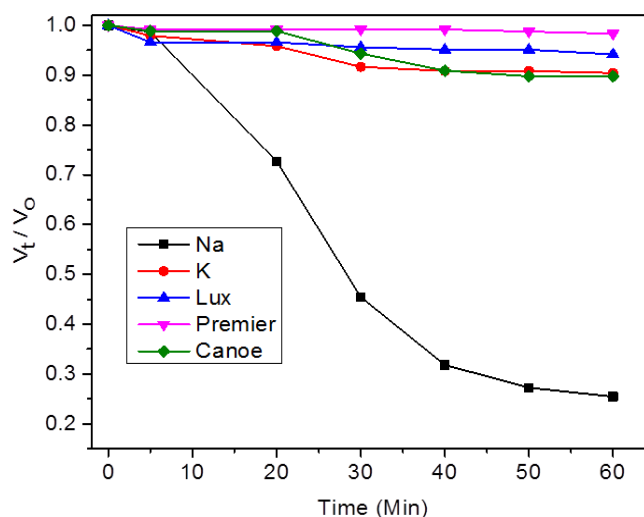


Figure 5. Comparison of foams' stability of the prepared and some commercial soaps.

3.3. Antibacterial Screening

Table 2 shows the inhibition of various kinds of soap samples against bacteria strains. Potassium soap showed same zone of inhibition against all the tested bacteria strains with a diameter of 0.2 mm each. Sodium soap showed lesser inhibition than potassium soap against *klebsiella species* and *Proteus microbilis* with a diameter of 0.1 mm each. Thus, in efficacy comparison, potassium soap was found to be more effective against *klebsiella species* and *Proteus microbilis* in agar diffusion method. Dettol soap showed highest zone of inhibition against all the bacteria strains, which was due to its antiseptic nature. Premier soap (medicated) showed same zone of inhibition against *klebsiella species* and *Bacillus amyloliquefaciens* with a diameter of 0.3 mm each. Imperial also showed the same zone of inhibition against *klebsiella species* and *Bacillus amyloliquefaciens* with a diameter of 0.2 mm each. Lux showed same zone of inhibition against *klebsiella species* and *Bacillus amyloliquefaciens* with a diameter of 0.2 mm each.

Table 2. Inhibition zone diameter in mm of various kinds of soap samples against bacteria strains.

Soap sample		Bacteria strain			
		Bacillus plimilus	klebsiella species	Proteus microbilis	Bacillus amyloliquefaciens
Prepared	Na soap	0.2	0.1	0.1	0.2
	K soap	0.2	0.2	0.2	0.2
Dettol (Medicated)		0.7	0.4	0.4	0.4
Premier (Medicated)		0.4	0.3	0.2	0.3
Imperial (Toilet)		-	0.2	0.3	0.2
Lux (Toilet)		0.5	0.2	0.3	0.2

4. CONCLUSION

An evident of successful saponification was shown in the FTIR spectra of the Na and K soaps with a quite distinct band at 1560.46 cm⁻¹ for carboxylate (COO⁻) ions which is absent in the starting beeswax spectrum. The beeswax soaps have good qualities with respect to pH, foaming ability, longevity and hardness. The sodium soap displayed low solubility in water and poor foam stability which are desirable characteristics of a good lubricating

surfactant. Sodium soap has lower antibacterial activity than KOH soap. Both beeswax soap samples showed lower antibacterial activity than tested antiseptic soaps (Dettol and Premier) but showed almost the same antibacterial activity with the tested toilet soaps (Imperial and Lux). The results showed that beeswax is a good material for soap making, and with more scientific findings, it would be a good candidate for the production of high quality soaps for both industrial and domestic uses. Consequently, it would serve as good alternative to the high demanding fats and oils from animals and plants in soap industry and would significantly reduce disposal of honeycombs as waste.

Funding: This study received no specific financial support.

Competing Interests: The author declares that there are no conflicts of interests regarding the publication of this paper.

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