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Laundry Soap Production from the Respective Tallows of Goat, Sheep and Cow: Evaluation of **Physicochemical Properties for the Best**

Abdullahi Nwaha Isah^{1*}, Umi Aisah Asli², Nasiru Audu¹, Sadiku Itopa Bello¹ Jibrin Waziri³ and Siyaka Omonowo Salihu⁴

¹Department of Chemical Engineering Technology, School of Engineering, Federal Polytechnic Nasarawa, Nigeria.

²Department of Chemical Engineering, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, Malaysia.

³Department of Chemical Engineering Technology, Faculty of Engineering, Kaduna Polytechnic, Kaduna, Nigeria.

⁴Department of Mechanical Engineering Technology, School of Engineering, Federal Polytechnic Nasarawa, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. Author ANI conceived the research work and designed the experiment. Authors ANI, NA and SIB coordinated the experimental work. Authors ANI, UAA, NA, SIB, JW and SOS contributed to the review and editing and authors ANI, UAA, NA and SIB managed the analyses of the study and supervision. All authors read and approved the final manuscript.

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ABSTRACT

Tallow mainly consists of triglycerides, whose major constituents are derived from stearic, palmitic and oleic acids, and its usage reduces production cost of soap, adds lather stability and hardness to soap. Laundry soaps were produced with variation on amount of tallow (sourced from cow, sheep and goat) and labelled as A, B, C, D and E formulations. The respective tallows were

*Corresponding author: E-mail: isnab2006@yahoo.com;

characterized in terms of saponification value and acid value and determined to be 192.14 and 2.24mg KOH/g (cow tallow); 200.56 and 2.38mgKOH/g (sheep tallow) and 197.75 and 1.96 mgKOH/g (goat tallow). The physicochemical properties of soap which determine its area of usage and cleansing properties were determined. The properties considered in this work were hardness, moisture content, foam capacity, pH, free acidity content, and total fatty matter. The hardness, moisture content, foam capacity, pH, free acidity content and total fatty matter of the produced soaps were determined and ranged between mild-deep penetration level; 11-21%; 1-9cm; 8-10.5; 0.16-0.82% and 40-86% respectively. From the comparative analysis, soap made from sheep tallow has the lowest penetration level (with formulations B and E), lowest free acidity content of 0.16% (with formulation A), highest total fatty matter of 86% (using formulation E), highest foam height of 9cm (with formulation A), lowest moisture content of 11% (with formulation A) and mild alkalinity of 8 (with formulations A, B and E). These results showed that the soaps produced from sheep tallow are the best in terms of hardness, lather and skin friendliness, due to its high degree of longer carbon chain lengths of fatty acids. These values satisfy the standard limit set for good quality laundry soap by National Agency for Food and Drug Administration and Control and Encyclopaedia of Industrial Chemical Analysis, respectively.

Keywords: Tallow; formulation; physicochemical properties; laundry soap; lather.

NOMENCLATURES

A : Weight of petri dish and soap after drying, g

B : Weight of petri dish, g

 C_h : Weight of crucible and sample after heating, g

 C_s : Weight of crucible and sample, g

 C_w : Weight of crucible, g

M: Molarity of H_2SO_4 solution, mol,/L

V : Volume of H_2SO_4 solution used in titration, MI

W : Weight of the soap, g

ABBREVIATIONS

EICA : Encyclopaedia of Industrial Chemical Analysis

ISO : International Standard Organisation

NAFDAC : National Agency for Food and Drug Administration and Control

1. INTRODUCTION

Soaps are water-soluble salt of fatty acids containing twelve or more carbon atoms, as derivatives from the reaction between fats/oil and alkali at appropriate conditions. Soaps produced from the saponification of sodium hydroxide and glycerides are referred to as solid or bar soaps, and with potassium hydroxide results to liquid or toilet soaps. Properties of the soap such as hardness are function of the metallic element present in the salt [1-3]. A soap molecule has a long hydrocarbon chain with a carboxylic acid group on one end (anions), which has ionic bond with metal ion (univalent cation), usually sodium or potassium for soluble soap for cleaning and bathing, while that of calcium and magnesium are used as component of lubricants, being water insoluble [4]. Soaps are produced for varieties of purpose ranging from washing. bathing. medication, on selection of etc, based

ingredients and method of preparation [1]. Fats and oils are mainly composed of fatty acids, which differ in chain length and degree of saturation. Fats are solid esters of fatty acids and glycerine while oils are the liquid glycerol esters of fatty acids at room temperature [5]. The number of carbons in the chain could be used to classify the fatty acid into short chain (C1-5), medium chain (C6-12) and long chain (C13-21), while the bond between carbons determines the degree of saturation of fatty acid as either saturated (single bonds) or unsaturated (double bonds). Best soap properties are achieved with fatty acids having C12 to C18 carbon chains and the distribution of unsaturated and saturated fatty acid determines the hardness, aroma, cleansing, lather, and moisturizing abilities of soaps [6-10]. Tallow is a rendered form of beef or mutton fat, and is primarily made up of triglycerides. A triglyceride consists of a three carbon glycerol head group to which are added

Fig. 1. The hydrolysis of triglycerides with sodium hydroxide

three fatty acid chains [11]. Tallow is considered as an alternative to vegetable oils in soap production, because it contains high content of saturated fatty acids and some traces of unsaturated fatty acids, which adds lather stability, hardness to soap and increases its solubility [12]. In addition, it is readily available, cheap and reduces soap production cost. Among the animals, tallow from beef and sheep or goat contain high content of palmitic, stearic and oleic acids (as shown in Table 2), which make soaps produced from these fats to be soluble and milder with good detergency [8]. Tallow soap is produced from exothermic reaction between tallow and alkali (either sodium hydroxide or potassium hydroxide), with glycerine as a byproduct, as shown in Fig. 1 [13].

The attachment of long hydrocarbon chains to the carboxyl group (carboxylate anion) induces the cleaning effect to soap made from tallow. When the soap is dispersed in water, the hydrocarbon chain (nonpolar tail of the soap) is attracted to grease (dirt), and the carboxyl group to water. The dirt is dissolved in the alkyl groups of the soap molecules while the ionic end (usually sodium or potassium) allows it to be dissolved in water and can be washed away. This is the phenomenon at which dirt is removed from clothes and skins of human body using soaps [14-16]. The chemical characteristics of soap depend on some factors: the strength and purity of alkali, the kind of oil used (in terms of its saponification and acid values), method of soap making process(cold process, semi-boiled or full boiled process), completeness of saponification, and age of the soap, while the characteristics that denote the quality of soaps include fragrance, colour, lather ability, moisturizing

capabilities, hardness, skin compatibility and chemical stability during storage and usage [17,6]. Physicochemical properties which quality of soap depends on include hardness, form forming capacity, pH, moisture content, free acidity content and total fatty matter [17,15]. In this comparative study, soaps were produced from cow, sheep and goat respective tallows using semi-boiled method and their quality ratings were investigated in terms of physicochemical properties such as pH, moisture content, foam height, hardness, free acidity content and total fatty matter.

2. MATERIALS AND METHODS

2.1 Reagents

Sodium hydroxide (purity:98%). sodium carbonate (purity;98%), sodium laureth sulphate(purity;98%), sodium sulphate (purity;99%), calcium carbonate (purity;98%), sodium silicate (purity; 97%), distilled water (purity;99%), ethanol (purity;98%), potassium hydroxide (purity;98%), tetraoxosulphate (VI) acid (purity;98%), and phenolphthalein indicator. They were all obtained at analytical grade from the chemical vendors.

2.2 Sample Collection and Preparation

Respective tallows of goat, sheep and cow were collected from slaughterhouse (abattoir) in Nasarawa, Nasarawa state, Nigeria. The fat was rendered according to the description made by Wara et al. [18]. After removing the meat tissues from the fat, it was placed in 4L stainless pot containing 1L of distilled water. The mixture was

boiled until the fat has completely melted. The solution was allowed to cool overnight, and the next day a layer of clean fat (tallow) was formed on the top of water. The tallow was then scooped for soap production, as the dirt was underneath the water.

2.3 Formulation

In all the formulations, the ratio of sodium hydroxide to the tallow used was 1: 7 (14.28%) by weight, and in terms of representative tallow weight to cumulative weight including additives, soap recipes were classified into : A (63wt%); B (67wt%); C (61wt%); D (60wt%) and E (65wt%), based on calculated lye and for complete saponification. The essence is to determine the best soap from the recipes and respective tallows. Semi-boiled method was adopted due to its suitability for laboratory preparation of soap and ease of separation of by-product and excess starting materials.

2.4 Soap production

30g of sodium hydroxide was dissolved in 60mL of distilled water and stirred vigorously. Thereafter, caustic soda solution was poured slowly into 160mL of melted tallow and stirred until there is proper dispersion. 15g of each of sodium carbonate, sodium sulphate, and calcium carbonate were added in a 100mL beaker and stirred properly until they blend, and then added to 5g of sodium laureth sulphate (SLS) that was dissolved in 15mL of distilled water in a 500mL beaker. Then the solution containing soda ash, sodium sulphate, SLS and calcium carbonate was added to the soap recipe and stirred vigorously. 30mL of sodium silicate was added to the recipe and stirred until a homogeneous mixture and trace was attained. Thereafter, the mixture was heated to a gel state at 80°C and on cooling; lavender (for fragrance) and dyes (for colour inducement) were added with thorough mixing before pouring into mould, where it cured for 48h. The same procedure was adopted for respective soap recipes containing 200mL, 240mL, 280mL and 300mL of tallow.

2.5 Analysis on Chemical Characterization of Tallow

2.5.1 Determination of saponification value

2g of the oil was introduced into a 300mL conical flask. 0.5M KOH was added to the solution and heated to 55°C over water bath with continuous

stirring. Thereafter, the temperature was raised to 100°C and allowed to boil for an hour in order to complete the saponification process. The excess KOH was titrated against the mixture using phenolphthalein indicator until pink colour was observed. Saponification value was calculated using Eq. 1[1].

SAP value =

56.1× normality of KOH×net volume of HCl used between real titration and blank (1)

Weight of the oil sample(q)

2.5.2 Determination of acid value

Acid values were determined using the modified method of Wara et al. [18]. 5g of fat was added to 50mL of alcohol in 250mL conical flask and the mixture was boiled in a water bath. Two drops of phenolphthalein indicator was added and titrated against 0.1KOH until the end point was marked by the appearance of pink colour. Acid value was calculated using Eq. 2.

Acid value =
$$\frac{56.1 \times normality \ of \ KOH \times vol.of \ standard \ KOH \ solution}{Weight \ of \ the \ fat \ sample}$$
 (2)

2.6 Physicochemical Analysis on the Prepared Soaps

2.6.1 Determination of free acidity content in soap

6 g of the soap sample was dissolve in 70mL hot neutral alcohol and titrated against 2M H₂SO₄ using phenolphthalein as indicator. The free acidity was calculated using Eq.3 [19]:

Free acidity(%) =
$$3.1 \times \frac{M \times V}{W} \times 100$$
 (3)

2.6.2 Determination of total fatty matter

A modified method as described by Mak-Mensah and Firempong [17] was used to carry out total fatty matter test. 10g of produced soap was weighed and 150mL of distilled water was added and heated for 25 min. The soap was dissolved in 20mL of 15% $\rm H_2SO_4$ and heated until a fatty acid layer was formed. The resulting solution was filtered by using filter paper and transferred into a pre-weighed petri dish. The content was evaporated using electric oven (Thermostat oven-DHG9023A) and the residue was weighed. The total fatty matter was calculated using Eq. 4.

$$%TFM = \frac{A-B}{W}$$
 (4)

2.6.3 Moisture content determination

5g of prepared soap samples was accurately weighed using analytical balance (Ohaus SP-401) into dried, tarred dish and dried in an oven (Thermostat oven-DHG9023A) for 2hr at 101°C and repeated until constant weight was reached. The moisture content (%) was calculated using Eq. 5 [20].

Moisture content (%) =
$$(C_s-C_h)/(C_s-C_w) \times 100$$
 (5)

2.6.4 Determination of faomability

2g of shaved soap was added to a 500mL measuring cylinder containing 100mL of distilled water. The mixture was shaken vigorously for 2 minutes and the cylinder was allowed to stand for 10 minutes. The height of the generated foam in the cylinder was measured and recorded [11].

2.6.5 Determination of the hardness of soap

Hardness test on the soap was done by piercing a needle through the surface of the soap. The distance at which the needle pierced through the bar of soap determined its hardness.

2.6.6 pH determination

The pH was determined using pH meter (Jenway 3505). 10g of the soap shavings was weighed and dissolved in distilled water in a 100mL volumetric flask (10% soap solution). The pH reading was recorded for every soap [17].

3. RESULTS AND DISCUSSION

Saponification value and acid value provide information on the quantity, type of glycerides and mean weights of the acids in a given sample [21].

3.1 Saponification Value

Saponification value is an indicator of the average molecular weight and hence chain length of fatty acids present in the oil/fat. The saponification values of respective tallows of sheep, cow and goat employed in this research work are shown in Table 1. These values are higher than 140.3mgKOH/g for tallow reported by Warra et al. [18]. Chizoo et al. [22] determined the SAP value of tallow to be 199.5mgKOH/g, which is within the range of saponification values

of tallows used in this research work. The high saponification value of sheep tallow could be attributed to the predominantly high proportion of longer carbon chain lengths of fatty acids and in turn more glyceride molecules [23]. The larger the saponification value, the better the soap making ability of the oil/fat [21]. The saponification values are within the limit (190-202mg KOH/g) set by NAFDAC in its Fats and Oils Regulation [24].

3.2 Acid Value

Acid value indicates the proportion of free fatty acid present in oil or fat and may be defined as the number of milligrams of caustic potash required to neutralize the acid in 1g of the sample. The acid values of respective tallows of sheep, cow and goat employed in this research work are shown in Table 1. These values are less than maximum value of 2.5mgKOH/g set by [24]. The low acid values indicate that the triacylglycerols have not been hydrolysed, which could indicate a good stability and safe for making skin related products [25-26]addition, a low acid value of oils is responsible for its resistance to rancidity, gum formation and corrosion and they can be stored for a longer time [27].

Table 1. Chemical characteristic of tallow used

Parameter	Type of tallow	Amount
Saponification	Sheep	200.56
value (mg KOH/g	Cow	192.14
of oil used)	Goat	197.75
Acid value	Sheep	2.38
(mg/KOH/g)	Cow	2.24
	Goat	1.96

Physicochemical characteristics of the soaps prepared were evaluated in terms of lowest and highest value of the parameters considered by comparing with standard values and other literature, in order to assess the quality of soap samples.

3.2.1 Free acidity content

Free acidity content is a prominent parameter used to determine the abrasiveness of soap. As shown in Fig. 2, soap made from sheep tallow has the lowest free acidity of 0.16% (using formulation A) compared with that of cow and

goat, and it implies higher reaction conversion. However, the values of free acidity content in soaps made from respective tallows of cow and goat are within the range of free acidity content (0.06-0.88%) reported by Popescu et al. [19]. Since the lesser the free acidity content, the better the soap, soaps made from sheep tallow were overly the best among other soaps.

3.2.2 Total fatty matter (TFM)

TFM is the ratio of mass of fatty matter to the total mass of the soap. Fig. 3 shows the variation in TFM, as an important characteristic describing the quality of soap. Soap made from tallow of sheep has the highest percentage of total fatty mater of 86% (using formulation E), while soap made from goat tallow has the lowest content of total fatty matter of 40% (using formulation E). High content of TFM in soap (at least 76%) rehydrates the skin, making it smooth, and in turn the high oil content within the soap acts as a lubricant throughout the day [20,9]. The lower TFM value is due to the presence of unreacted

sodium hydroxide in the mixture and degrades the quality of the soap. According to ISO, good quality soaps must have TFM above 76% [32]. However, the lowest acceptable percentage of TFM according to [33] is 60%, which also guarantee the safe usage of soaps made from cow (with formulations D and E) and goat (using formulation D). From the overall assessment, soaps made from sheep tallow were of best grade according to the aforesaid standards.

3.2.3 Moisture content

Moisture content is attributed to the existence of liquid especially water, usually in trace extent. Soap made from sheep tallow has the lowest moisture content of 11% (against formulation A), while soap made from goat tallow has the highest moisture content of 21% (with formulations B and D), as shown in Fig. 4. In addition, moisture contents of soap samples from sheep tallow with formulations D and E were determined to be 14 and 13.6, respectively, while others ranged from 16.2 to 21.

Table 2. The fatty acids composition distribution of cow, sheep and goat tallows

Fatty acids (%)	Myristic	Palmitic	Stearic	Linoleic	Oleic	References
Cow	2.79-3.7	21.5-26.29	33.54-34.85	1.19-3.51	22.34-41.4	[28,29]
Sheep	3-6	24-32	19-25	2-3	37-45	[11]
Goat	28-50	15-31	6-17	4-15	23-35	[30, 31]

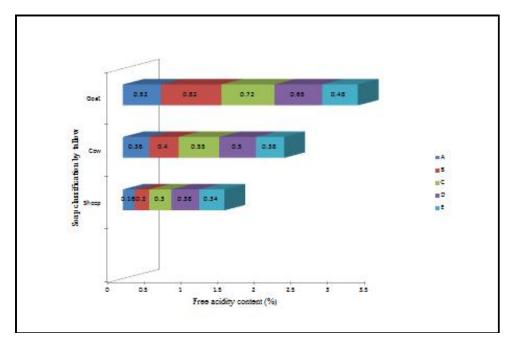


Fig. 2. Variation in values of free acidity content of the prepared soaps

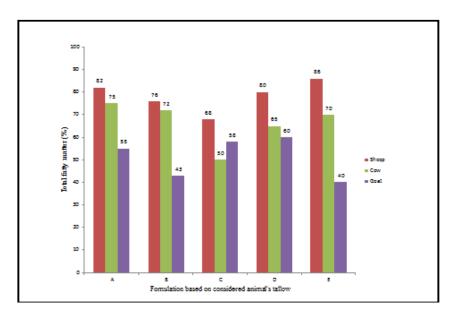


Fig. 3. Variation in values of total fatty matter of the prepared soaps

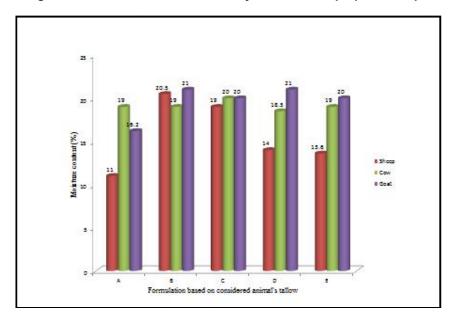


Fig. 4. Variation in values of moisture content of the prepared soaps

Habib et al. [3] reported that soap which contain minimum amount of moisture content aids in elongating its shelf life. Mahesar et al. [15] reported that high moisture content in soap would lead to reaction of excess water with unsaponified fat to give free fatty acid and glycerol in a process called hydrolysis of soap on storage. According to the standard set by EICA [34] and [33], the acceptable range for moisture content of soap is between 10-15%. As such,

soaps made from sheep tallow which meet this set standard are the best in terms of lathering and cleansing property [35].

3.2.4 Foaming power (Foamability)

Foam test is an important parameter for acceptability of soap. Fig. 5 shows that foamability of the soap samples based on the type of tallow and formulation. Soap made from

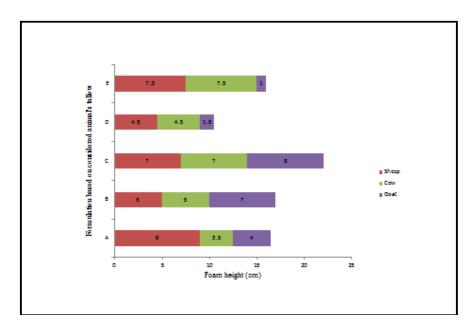


Fig. 5. Variation in values of foam height of the prepared soaps

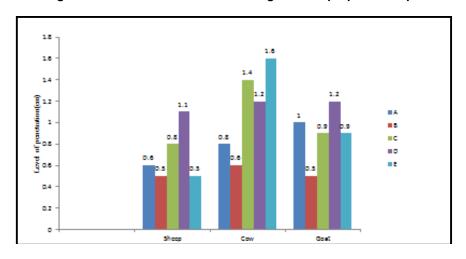


Fig. 6. Variation in levels of penetration of needle through the prepared soaps

sheep tallow produced the highest amount of foam (9 cm) (with formulation A), while soap made from of goat tallow produced the lowest amount of foam (1 cm) (with formulation E). The highest amount of foam produced via sheep tallow made soap exceed the value of 4.6cm reported by Hassan and Wawata [36], and the range of foam height (2.3-8.5cm) reported by Mahesar et al. [15]. The variation in the amount of foam produced could be attributed to the differences in formulation and fatty acids present in the tallow. The higher the foam height, the better the lather of the soap [37].

3.2.5 Hardness

The physical hardness of a bar of soap is largely relative to the amount of water used in the formula, as well as its curing time [35]. Fig. 6 shows the level of penetration of needle during the hardness test of the bar soaps. Soaps made from tallow of sheep and goat have the lowest level of penetration (with formulations B (sheep and goat) and E (sheep only)), while soap made from cow tallow has the highest penetration level (with formulation E). The harder the bar of soap the less distance the needle will penetrate. Hard soaps have lower solubility, which in turn prolong their shelf lives [38, 3].

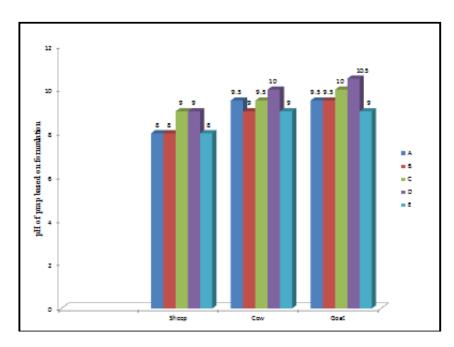


Fig. 7. Variation in pH of the prepared soaps

3.2.6 pH

pH is a vital parameter in determining the quality of soap. Fig. 7 shows the variation in pH of soaps produced from respective tallows of goat, sheep and cow. Soaps made from sheep tallow have the lowest pH of 8 (with formulations A, B and E) while soap made from goat tallow has the highest pH of 10.5 (with formulation D). In addition, pH values of other soap samples ranged from 9 to 10. The alkaline nature of soap is to serve as barrier against abnormal bacterial flora and virus by neutralizing the body's protective acid nature (ranging from 5.4 to 5.9 for healthy body) and makes it lather easily[17, 36]. Soap with pH below 5 and above 10 causes harshness on the hands and skin. Mild alkaline nature of soap is preferable (marginally above 7), since alkali contained in the soap is released when it comes in contact with water and increases the skin pH to 10-11[39,40]. Tarun et al. [41] reported that the usage of soap with high pH causes an increase in skin pH which in turn causes an increase in dehydrative effect, irritability and alteration in normal bacteria flora on the skin.

4. CONCLUSIONS

Soaps were made from respective tallows of cow, sheep and goat through semi-boiled process method and their qualities were

established with respect to physicochemical properties. Tallow majorly contains saturated fatty acids with some trace of unsaturated fatty acids, which is responsible for its lather and good conditioning properties to the soap. The physicochemical properties of soaps tested were hardness, foam capacity, moisture content, free acidity content and total fatty matter and were compared to identify the best among the tallow made soaps. From the comparative analysis, soap made from sheep tallow has the lowest penetration level (with formulations B and E), lowest free acidity content of 0.16% (with formulation A), highest total fatty matter of 86% (with formulation E), highest foam height of 9cm (using formulation A), lowest moisture content of 11% (with formulation A) and mild alkalinity of 8 (using formulations A, B and E). It is evident that soaps made from sheep tallow are excellent in terms of friendliness to the skin, hardness and lather, which could be attributed to its high proportion of longer carbon chain lengths of fatty acids. These values are within the specification limit for good laundry soap set by Encyclopaedia of Industrial Chemical Analysis and Nigerian Industrial Standard. It could be concluded that the quality of the soaps depends on the formulation and the nature of tallow used.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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