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Cold- Process Synthesis and Properties of Soaps Prepared from Different Triacylglycerol Sources

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ABSTRACT: This study was conducted to find out how different fats and oils produce soaps of different characteristics. It describes cold-process saponification using different fats and oils. Shea nut oil (SAP value:183.9mgKOH/g), groundnut oil (SAP value:187.7mgKOH/g) and Tallow (SAP value: 140.3mgKOH/g) were used. Colour, texture, lathering and cleansing power of the prepared soaps were analyzed. shea butter soap had the best lathering capacity. The groundnut oil soap had the most effective cleaning power .The soaps were also recommended for household use .This activity was also provided to share a delight in chemistry with senior school students and to actively engage them in hands-on-active learning.
Key words: cold-process, saponification, triacylglycerols and properties

INTRODUCTION

Soap is a mixture of sodium or potassium salts of various naturally occurring fatty acids. Soap is a substance of ancient origin, the manufacture of which according to Gunstone *et al*,(1986)has evolved from primitive beginnings into a sophisticated chemical process. Evidence of soap making dates back to the Egyptian and Babylonians. Several millennia ago crude mixture of animal fats and the alkaline plant ash were found to generate crude soaps which lathered and cleaned effectively (Phansteil *et al*, 1998). Through the centuries, there have been other times when people were able to make soap using sodium or potassium salts as is done recently. When the Germans tribesmen of Caesar's time boiled goat tallow with potash leached from the ashes of wood fires, they were carrying out the same chemical reaction as one carried out on a tremendous scales by modern soap manufacture by hydrolysis of glycerides (acylglycerols) (Morrison and Boyd, 2004). The commonest oils used in soap making are lard and tallow from animal sources, coconut, palm and olive oil from vegetable source (Pavila *et al,*1982).The Commercial manufacturers use 1-7% excess fat to reduce the harshness of soap, to produce a dense creamy lather, and to leave the skin feeling smooth and soft (Mabrouk, 2005).The formulation in this paper uses 10% excess fat and oils.

This paper describes the cold-process saponification and use hands-on-active learning approach. This strategy is vital in teaching students laboratory techniques and inquiry skills. Laboratory exercises should be used frequently throughout a science course to promote science learning (Chiappetta and Koballa, 2006). Therefore activities of this kind should be encouraged in schools. This project was carried out in the Chemistry Laboratory of Haliru Abdu senior Arabic Secondary School, Jega, Kebbi State, Nigeria.The fats and oils used were obtained from Jega central market. While the sodium hydroxide (analytical grade) used as the alkali in the hydrolysis of the fats and oils were obtained from laboratory store in the department of chemistry, Haliru Abdu Senior Arabic secondary School, Jega.

MATERIALS AND METHODS

Preparation of Tallow from Animal Fat: The preparation of fats for soap making consist of cleaning the fats to be free from impurities

contained in it. This is called **rendering**. To render the fat, all meat tissues were removed. The fat was then placed in a stainless steel pot containing 500cm^3 of distilled water. The mixtures were boiled until the fat melted. The solution was allowed to cool down and left overnight. The next day the fat had solidified and floated to the top forming a layer of clean fat. All the impurities being denser as the fat remained in water underneath the fat.

Saponification Procedure: For each soap formulation 200g/dm^3 NaOH solution was poured directly into the stainless steel container (pot) containing the fat and oil in the ratio 1:1 (v/v). When the mixture of tallow and groundnut oil was used the fat/oil mixture contains 3:2 (v/v). The fats/oil was warmed gently and was poured into the stainless steel soap making pot followed by the alkali solution to form an intimate mix and then stirred frequently for 10-15 minutes using wooden stirrer. Perfume and other ingredients were added as additives at the last time before pouring the saponification mixture into moulds. After pouring, the soap was allowed to harden by air-drying for 24hours to obtain the soap bars, according to the method reported by Warra,(2009) and the soap bars were then observed for colour, texture, lathering and cleaning power.

Note: To reduce the harshness of soap which may be cause by excess sodium hydroxide, 10% excess fat and oil was used in the formulations (super fatting).

Analytical method: Three different fats and oils were used for this research; shea nut oil, Tallow, and Ground nut oil. The chemical analysis of the oils were carried out using the methods adopted by Diamond, *et al* (1973), Bassir,(1978), Plummer,1987, Association of Official Analytical Chemists(AOAC) ,(1990) Akpan *et al*,(2006), and Bahl, *et al*, (2007), with modifications.

Saponification value: About 2g of the oil sample was added to a flask with 30ml of ethanolic KOH and was then attached to a condenser for 30 minutes to ensure that the sample is fully dissolved. After sample has cooled 1ml of phenolphthalein was added and titrated with 0.5 M HCl until a pink colour indicated the end point.

The expression for saponification value (S.V.) is given by: S.V = 56.1N (V_0-V_1)/M, where V_0 = the volume of the solution used for blank test; V_I = the volume of the solution used for determination; N = actual normality of the HCl used; M = Mass of the sample.

Iodine value: 0.4g of the sample was weighed into a conical flask and 20ml of carbon tetra chloride was added to dissolve the oil. Then 25ml of Dam's reagent was added to the flask using a safety pipette in fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours 30 minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 125ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium-thiosulphate solutions until the yellow colour almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples. The iodine value (I.V) is given by the expression: I.V = 12.69C (V_1-V_2)/M, where C = Concentration of sodium thiosulphate used; V_1 = Volume of sodium thiosulphate used for blank; V_2 = Volume of sodium thiosulphate used for determination, M = Mass of the sample.

Acid value: 100 ml of neutral ethyl alcohol was heated with 10g of oil or fat sample in a 250ml beaker until the mixture began to boil. The heating was stopped and the solution was titrated with N/10 KOH solution, using two drops of phenolphthalein as indicator with consistent shaking for which a permanent pink colour was obtained at the end point.

The Acid value was calculated using the expression

A.V = 0.56 x No. of ml. N/10 KOH used.

pH Determination: The pH was determined using a pH meter (827 pH lab Model). 10g of the soap shavings was weighed and dissolved in distilled water in a 100ml volumetric flask. This was made up to prepare 10% soap solution in line with literature report (Dalen and

Mamza,2009) The electrode of the pH meter was inserted into the solution. The pH reading was recorded. The steps were repeated using soaps produced from each fat or oil.

Foam ability Tests: About 2.0g each of soap (shavings) was added to a 500ml measuring cylinder containing 100ml of distilled water as reported by Isah, (2006) for synthetic detergent. The mixture was shaken vigorously so as to generate foams. After shaking for about 2 minutes, the cylinder was allowed to stand for about 10 minutes. The height of the foam in the solution was measured and recorded. The steps were repeated using soaps produced from each fat or oil.

Test for effectiveness in cleaning: To determine the cleaning property of the prepared soaps, a drop of oil was placed on four separate strips of filter paper. The filter papers with the oil spot were immersed in a separate test tubes containing soap solution (2g soap shavings/100ml distilled water) each was shaken vigorously for 1 minute The filter papers were removed and rinsed with distilled water and the degree of cleanliness in each filter paper was observed.

RESULTS AND DISCUSSION

The results of the chemical analysis carried out on the oils are presented in the Table 1.

Table 1: Chemical analysis of oils used

	SAP value(mgKOH/g)	Iodine value (I_2/100g)	Acid value (mgKOH/g)
Tallow(Animal fat)	140.3	44.3	16.1
Shea-nut oil	183.9	54 .1	10.5
Ground nut oil	187.7	84.8	3.83

The values are mean of three replicates.

Chemical Analysis: The physicochemical values are relatively similar to the literature values. For the indigenous crude shea fat : saponification value 183.7, was obtained ,the value is lower than that of olive oil(192.0) and sunflower oil(188.7) but higher than that of beeswax(93.0) which are commonly used in soap making (Mabrouk,2005).This indicates that the oil could be used in soap making since its saponification value fall within the range of these oils. It could also substantially be used in the preparation of cosmetic products (Warra, 2009). Oils with high saponificaton values such as coconut oil (257.0) and palm oil (199.1) are better used in soap making (Abayeh *et al* , 1998). Some soap manufacturers blend their oils with coconut oil (with high saponification value) to get yield of better quality soaps. Iodine value, 54.1(less than 100) was obtained which shows that the oil belongs to class of Non-drying oils which are useful in the manufacture of soaps (Kochhar, 1998) . The Acid value (10.5) authenticates the raw material and conforms relatively to 10.49 3mgKOH/g reported by Oyedele (2002), which signifies a minimum purity. The values obtained for Tallow includes; saponification value(140.3mgKOH/g) which is lower than 185.00mgKOH/g in Cyperus esculentus oil (Hassan *et al*,2007) and 213mgKOH/g in neem seed oil (Akpan,1999) and 253.2mgKOH/g in coconut oil (Oshinowo,1987) which are also oils used in soap making. This is higher than 56.10mgKOH/g value obtained for cassia siamea oil also recommended for soap making (Hassan *et al*, 2005). 44.3I_2/100g was obtained as the iodine value of Tallow, this value is relatively comparable to that of Avocado seed oil (42.6) which is used in soap making (Sani and Hassan ,2007). Tallow is one of the principal fatty acid material used in soap making. It contains mainly the palmitic, stearic and oleic acids (Pavila *et al*, 1982). The acid value (which indicates the amount of free fatty acids present in the Tallow) was 16.1mgKOH/g relatively similar to the value reported for Albizia lebbeck,L seed oil (Hassan *et al*,2005) recommended for soap making. This value also shows that the oil is advantageous for soap making (Owoyale,1987) For groundnut oil the saponification value was 187.7 comparable to the value obtained for corn oil (187.0)

(Zhang,*et al*,1986) .Even though ground nut oil is a known edible oil its high saponification value is an indication of its suitability for soap making. The value is lower than that of olive oil (192.1) (Mabrouk, 2005) but higher than that of bees wax (93.0)which are all used in soap making. The ground nut oil has an iodine value of 84.8. This value is comparable to (81.8) the value obtained for olive oil (Almustapha *et al*, 1995) and 85.00I_2 / 100g value of Cyprus esculents oil (Hassan *et al*, 2007). High iodine value justifies utilization of the oil in soap and shampoo productions (Hassan *et al*, 2007) groundnut oil is an example of non drying oils whose iodine numbers are less than 100 (Kochhar, 1998) they have the advantage of not undergoing oxidation to form a film, hence are useful in the manufacture of soaps (Kochhar 1998) .3.83mgKOH / g was obtained as the acid value which is comparable with 4mg KOH /g recommended by a Cordex Alimenterius commission for ground nuts (Abayeh *et al*, 1998) The value is lower than 16.83mgKOH / g for cassia siamea(Hassan at et al, 2005) but higher than (1.84) reported for sesame seed oil (Almustapha *et al*, 1995) which are used in soap making.

Table 2: Foam height of soaps prepared.

Prepared Soaps	Height of foam over water(cm)
Animal fat soap	2.5
Shea nut oil soap	2.7
Ground nut oil soap	2.3

The values are mean of three replicates

Table 3: Soap Types, Lathering and Cleaning properties.

Soap Formulation	Fat/oil	Ingredient	Type of Soap	Colour of soap	Texture	Lathering	Cleaning	Possible Uses
1	Mixture of tallow and g/oil	Perfume	Designer	Cream	Soft	---	Effective	Washing
2	Shea nut oil	Perfume	Multi-purpose	White	Very hard	Excellent	More effective	Antiseptic for bathing, washing
3	Mixture of tallow and g/nut oil	Honey, bees wax and perfume	Toilet	Brown	Hard	---	Effective	Antimicrobial soap for bathing
4	Tallow (Animal fat)	Sour lemon and Perfume	Laundry	Milk	Hard	Very Good	Effective	Washing
5	Groundnut oil	Perfume	Laundry	Milk	Soft	Good	Most effective	Washing

Physical Analysis: Table 2 shows the results of physical analysis carried out on the soap samples produced. The result indicates that colours of the soap formulation 1-5 were found to be cream, white, brown and milk respectively. The variation in colours could be due to the degree of unsaturation of the fatty acids. Increase in double bonds causes increase in intensity of colour (Mabrouk, 2005). Another possibility is that in all the formulations glycerol was retained, salting-out was not employed since it was cold-process method used; therefore all the soap samples maintained the color of the parent oil, similar to the findings reported by Warra, *et al* (2009b) with exception of formulation 3 which maintained brown colour due to the addition of brown colour ingredients (Honey and beeswax). For the texture of the soap, the variations could be or depends on the nature of their fatty acids composition length of hydrocarbon chain and nature of double bonds in the carboxylic acid portion of the fat or oil. Soap made from shea butter oil is the hardest followed by soap from tallow. Iodine number is another probable explanation for this variation. Oils with very

low iodine number yields soaps that are hard (Pavila *et al*,1982).Shea butter has the lowest iodine number compared to groundnut oil that produce soft soap. As a result of their molecular structures soaps are capable of dispersing oils and similar water –insoluble substances (Katz, 2000). The cleansing power and lather produced by different soap can be explained based on the fatty acids composition of oil used in soap formulation, it was reported that, lauric acid and myristic acid, which are all saturated fatty acids produces soap with fluffy lather and high cleansing power (phansteil et al, 1998). However, the observed difference in the cleansing power and nature of lather formed in the soap formulation may be due to the method use in the soap preparation in addition the nature of fatty acid composition of the fat or oil used.

Table 4. pH of prepared soaps.

Soap formulations	pH
1	8.3
2	8.0
3	7.8
4	8.5
5	8.8

The values are mean of three replicates

The students prepared soaps were all made by the same method .The majority of students preferred the soap made from shea butter oil for some reasons; it can serve asa multipurpose soap, it can be used for washing because of its excellent lathering and more effective when tested for its cleaning capability. Its use for bathing is related to its exceptional healing properties for the skin, shea butter contains vitamin A and E which are important for treatment of number of skin conditions (American Shea butter Institute, 2008). Shea butter has been used for centuries in Africa for its unsurpassed ability to maintain and protect the skin from environmental damage (Warra, *et al*, 2009). Shea butter oil is abundantly obtainable in most part of Kebbi state making it easier for students to obtain and prepare their home made soaps. The choice of honey soap (formula 3) as antimicrobial soap (or bathing) lies in its unique property of being antimicrobial agent as well as powerful antiseptic. The sugar concentration plus other factors including low pH, hydrogen peroxide and the flavonoids, phenolics and terpenes make honey an antimicrobial agent (Mizrahi, 2008).One student asked why we included sour lemon in the ingredient for soap formulation 4? Sour lemon is used to get rid of the odour in animal fat. Most fats (greases used in making soaps are smelly and they produce smelly soaps. In order to get rids of smells in soap animal fats are treated by adding lemon juice, vinegar or sour milk as described by Norman (2007). We measured the pH of our prepared soaps. Soap being salt of strong base and weak acids should be weakly alkaline in aqueous solution. But soap with free alkaline (PH 11 - 14) can cause damage to skin. Hence pH of 9-11 and 3-5 are considered as high and low levels respectively by NAFDAC a regulatory Agency in Nigeria (Umar, 2002). All the students formulated soaps were below the pH of 9. Our prepared soaps are closer to the pH of between 7 and 9 which is better.

Conclusion: From this experiment the students have increased their knowledge of the organic chemistry of soaps. Students were engaged in conversations and have expressed their feelings such as “I learn how to combine, many fats and oils to get soaps”. The students also appreciated how the modern day chemistry when blended with old tradition, accuracy and knowledge of the right ingredients and suitable methods yield the desired results. Another student remarked that he enjoyed the soaps; they wash more effectively than most commercial soaps he used to purchase from the market.

Fig. 1 Cold-processed soap samples

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