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Comparison of Various Fatty Acid Sources for Making Soft Soap (Part 1): Qualitative Analysis

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Abstract: This study compared the various fatty acid sources for making toilet soap via conventional method. These included palm oil, Palm Kernel Oil (PKO), groundnut oil and tallow fat. The toilet soap samples were developed using hot batch process by reacting 16.8 g caustic potash with 50 g oils/fats. The mixture was continuously stirred via electrically operated mechanical stirrer for 25-30 min. Later, 20 g of water was added to the system to break-up the foams, as well as aid the saponification process. Stirring was continued for 40-60 min, after which the salting process was carried out by adding the mixture to a solution of saturated sodium chloride. Also, stirring continued until the paste became thicker thereafter the reaction was stopped and was allowed to stand for several hours to cool and solidify. Analysis of the various soap samples was carried out to test for formability, corrosiveness, hardness, pH, rate of wear, acid value, saponification value and iodine value. Formability and corrosiveness results obtained are as follows: 9.90 cm for palm oil, 10.70 cm for (PKO), 10.74 cm for groundnut oil and 11.58 cm for tallow fat and 2.76% free alkaline for palm oil, 2.62% free alkaline for PKO, 2.60% free alkaline for groundnut and 2.52% free alkaline for tallow fat soap, respectively. The pH values are also given as 11.63 for palm oil-based soap, 11.0 for PKO-based, 10.30 for groundnut oil-based and 10.46 tallow oil-based soap.

Key words: Conventional method, fatty acid sources, hot-batch-process, toilet soap, foamability, corrosiveness

INTRODUCTION

The term soap derived its name from the people of the celts, who named it soap because they prepared soap from animal fats and plant ashes. Soap, whether ancient or present, is a cleaning substance that is used with water in the homes and industries for various cleaning purposes. In other words, it is a substance that when dissolved in water removes dirt from dirty materials. Scientifically, soap is one of the higher fatty acids or a mixture of such compound. The true soaps are salts of alkali metals and are soluble in water, but the term has been extended to include salts of other metals, some which are insoluble in water combination of fatty acids and certain organic bases such as ethanolamine, mixtures of the foregoing substances with alkaline silicates, glycerol and other additives. A typical soap contains 80% mixed oils and 20% coconut oil with not >0.2% free alkali. If an excess of alkali is used, the soap will contain free alkalis and the soap will have coarse action (Sharma *et al*., 2002). It is prepared in various forms as solid (in hard pieces) or as liquid or powder. Chemically, soap is a mixture of the sodium salts of stearic acid, $C_{17}H_{35}$ COOH, palmitic acid, $C_{15}H_{31}$ COOH and oleic acid, $C_{17}H_{33}$ COOH of the potassium salts of those acids (soft soap). Soaps are made by the action of sodium or potassium hydroxide on fats, a process of hydrolysis or saponification to form soap, with glycerol as a by-product. The term soap is also applied to fatty acid salts of metals other than sodium or potassium although, such compounds are unlike the ordinary soaps (Uvarov and Chapman, 1979). Soap itself is a detergent, but in common usage, the term detergent can be referred only to synthetic detergent for washing hence, all detergents found in stores or markets are synthetic. For soap or detergent to function effectively, it must be soluble in water. Thus, when the dirty materials are immersed in the soapy water, several reactions take place. The dirt is lifted and held in suspension until it is being rinsed off.

Initially, soap was produced from animal fats and wood ashes, simple wood or plant ashes containing potassium carbonate, which were dispersed in water and the mixture was subsequently boiled. This process is known as hot method. As time went by, the use of alkali and oil or fat was introduced. In this process, hot caustic potash acts on natural fats or oils, such as tallow or vegetable oil to form potassium fatty acid salt (soap) and glycerin. This reaction is called saponification and is the basis of modern soap making. The basic principle for making soap has not changed since about 2000 years ago. Soap was and still the earliest detergent known to man and was first produced nearly 5000 years ago by the

Romans fro animal fat and wood ashes (Kirk, 1983). Even later, a cold process of making soap was also developed, that is where, the mixture of the potassium/sodium carbonate dispersed in water was not boiled.

Today, technology has made a lot of impact in the life of soap manufacturers in the developed countries of the world, such that manufacturers can produce so many as 200 tons day^{-1} of soap depending on the capacity of the machines or plants. In Africa, generally and Nigeria in particular, not much has been done apart from the transfer of soap technology from the Western World by the way of employing an already established and existing formula in soap making.

In the present study therefore, an attempt was made to develop toilet soap in the laboratory conventionally using various fatty acid sources.

MATERIALS AND METHODS

Chemicals: The list of materials/chemicals used for the present study is given in Table 1. Conventional or the research code names of the materials/chemicals are used (Table 1). The various experiments were done in Chemical Engineering Laboratory, Chemical Engineering Department, Federal University of Technology (FUT), Minna-Nigeria.

Development of soft soap: The process of soap making employed in the present research entailed two steps namely, preparation of oil and caustic potash and saponification process using hot-batch process.

Hot-batch process: Fifty gram of palm oil was first of bleached at a temperature range of 50-60°C, thereafter, 50 g of the bleached oil was reacted with 16.5 g KOH in a double jacketed glass reactor. The mixture was stirred vigorously for 3 min and was allowed to heat for about 25-30 min with continuous stirring. The essence of stirring was to avoid sedimentation of the caustic potash, as well as to increase the rate of reaction. Twenty gram of water was subsequently added to break-up the foam, as well as aid the saponification process, which lasted for 40-60 min so as for a complete reaction to take place. The mixture was then added to a solution of saturated sodium chloride (NacI) (brine) for salting out. The neat soap floated on the surface of the solution, while the glycerol settled at the bottom. This was then separated using separating funnel and thereafter, the glycerol was reheated to obtain more soap sample.

Finally, 1.0 g dye and 1.0 g lemon grass perfume additives were added to the soap paste (Table 2). Ahmed (2002) and the soap material was subsequently poured into the moulder for moulding and shaping.

Extraction of lemon grass perfume: Lemon grass was collected, washed with water and dried under the sum for 3-5 days. The dried lemon grass was ground into powder and subsequently charged into a round bottom flask containing 20 g n-hexane. The mixture was vigorously shaken for many times until the n-hexane had turned deep green. At this stage, the extract was drained from the flask. The perfume was heated to removed the entrained n-hexane. After evaporation (removal) of n-hexane, the greenish liquid having the adour of lemon was decolourized.

Extraction of caustic potash: Palm kernel bunches (Elias genius plant) were collected and dried under the sun for a period of 14 days until a constant weight was attained. Subsequently, the bunches were gathered and placed in the furnace for proper ashing at a temperature of 600°C for a period of 7 h, until the ashes turned pure white.

Later 200 g of the ash was then mixed with 200 mL of distilled water and the resulting solution was filtered for caustic potash.

Qualitative analysis of the soap samples: The analysis of the various soap samples was carried out as follows:

Foamability: An amount of 2.95 g of each soap sample was weighed, ground to powdery form and subsequently dissolved in 800 mL of distilled water to form uniform soap solution. About 500 mL of the solution was poured into a

Table 1: List of materials/chemicals used for the experiment

Type	Materials/chemicals	Code	Source	Comment
Palm kernel bunch	Caustic potash	PKB	Alloma, Kogi State, Nigeria	Sourced locally by leaching
Lemon grass	Lemon grass perfume	LGP	G.R.A. Minna, Niger State, Nigeria	Extraction by soxlet means using n-hexane
Tallow fat	Fat	TAF	Itobe market Kogi State, Nigeria	-
Palm oil	Oil	Poo	Aku, Nssuka, Enugu State, Nigeria	Fat for saponification
Groundnut oil	Oil	GNO	Kano, Nigeria	Fat for saponification
n-hexane	Solvent	n-Hex	BDH chemical poole, England	Used as solvent for the extraction
Soap additives	dye	SOA	Lagos, Nigeria	Used to coloration

Table 2: The compositions of various soap samples of present research

Soap sample	Tallow fat (g)	Caustic potash (g)	Water (g)	Perfume (g)	Dye (g)
Palm oil	100.0	51.00	20.0	1.0	1.0
PKO	100.0	51.00	20.0	1.0	1.0
Groundnut oil	100.0	51.00	20.0	1.0	1.0
Tallow fat	100.0	51.00	20.0	1.0	1.0

conical flask and was stirred vigorously for 2 min (using an electrically operated mechanical stirrer) before allowing the foamy solution to settle for 5 min. Later, the weight of the foams was measured and recorded. The same procedure was repeated for other soap samples for comparison.

Hardness: This analysis was performed by cutting out some sizes of the soap samples and subsequently each of the samples was subjected to finger press for 10 sec. Their response on the fingers were measured and recorded.

Corrosiveness: The corrosiveness of the soap samples was determined by dissolving 2.95 g of the soap in 150 mL of hot distilled water. After dissolving it properly, 3-4 drops of methyl-orange were added to produce a yellow colour. A standard solution of 0.05 m H_2SO_4 was titrated against the soap solution until purple colour was observed, which signified the end point. The Volume of acid (V_1) was read from the burette and recorded. The percentage of free alkaline in the soap was calculated based on the given formula as follows:

$$KOH\ (\%) = \frac{V_1 \times M_w \times M_c}{W_g \times 20} \times 100$$

Where:

V_1 = Volume of acid used for titration
M_w = Molecular weight of KOH
M_c = Molarity of acid (concentration)
W_g = Weight of dissolved soap sample

Rate of wear: Twenty gram of the soap samples that have equal surface area and volume were cut with a knife and dropped into different conical flasks. Subsequently, equal volume of water (150 mL) was added to each of the conical flask containing the samples. The flask was shaken for 15 sec and allowed to settle for 1 min interval. This procedure was repeated once again after which the remaining soap samples were removed from the flask using forks and were reweighed and recorded.

Acid, saponification, iodine and titre analysis: The acid value, saponification value, iodine value and titres were also determined following some standard methods in the open study.

RESULTS AND DISCUSSION

The results obtained from the present study are presented as follows:

Foamability: The results of foamability are shown in Table 3, where the net heights of foams were recorded for the different soap samples prepared using different sources namely palm oil, groundnut oil and tallow fat.

From the above results, it can be shown that tallow fat-sourced-soap yielded the highest foam and was closely followed by that of groundnut oil-sourced-soap and the least was palm oil-sourced-soap. In descending order of the heights of the foams of the various soap sample, tallow fat-sourced>groundnut oil-sourced>palm oil sourced-soap. This order suggests that tallow fat is the best source for soft soap production especially where enhanced foamability is required.

Corrosiveness: The corrosiveness of the soap sample was given in Table 4, as the percentage of the unconverted potassium hydroxide (KOH) in the soap. The result reads as follows: 2.62% free alkaline for palm kernel oil sample, 2.58% for groundnut oil and 2.52% for tallow fat. These results imply that the more free alkaline the soap is, the less the foamability hence, the order of free alkalinity is thus: palm oil-based>palm kernel oil-based>groundnu-based>tallow-based soap. This observation is in order since increase in the percentage of concentration of KOH ought to give corresponding increase in the alkalinity and hence, corresponding increase in the pH values (Table 5). The less the free alkaline percentage the better the soap suggesting that tallow would be the best source, when compared to other sources since, it has the least prevent age of free alkaline.

Rate of wear: Table 6 gives, the rate of wear of the various soap samples. It shows that the palm kernel oil sourced soap whose initial weight was 20 g was reduced to 5.89 g whereas, those of groundnut oil and tallow fat sourced soaps were initially reduced to 5.65 and 5.54, respectively. This implies that the rate of wear of tallow fat sourced soap sample has the highest rat of wear, while that of palm oil sourced soap sample has the lowest rate. This also suggests that any tallow fat sourced soap may require filler typical of calcium carbonate as a filler to reduce drastically the rate of wear, as well as maintain its foamability at reduced alkalinity.

pH values: The results of the pH of the various samples of the soap are given in Table 5. From the Table 5, it can be shown that the soap sample prepared with palm oil showed the greatest increase in the alkalinity than those of groundnut oil and tallow fat. The order is given as

Table 3: Foamability as a function of foam height

	Net height (cm)			
Soap sample	Foam for groundnut based soap	Palm oil based soap	Foam for PKO oil based soap	Foam for tallow fat based soap
Hot-batch process	9.90	10.70	10.74	11.58

Table 4: The corrosiveness of the soap sample of present research

Soap sample	Palm oil	PKO	Groundnut oil	Tallow
Free alkaline KOH (%)	2.76	2.62	2.60	2.52

Table 5: pH of the various samples of present research

Soap sample	Palm oil	Palm kernel oil	Groundnut oil	Tallow fat
pH values	11.63	11.00	10.30	10.46

Table 6: Rate of wear of various soap samples of present research

Soap sample	Initial weight of palm oil (g)	PKO (g)	Groundnut oil (g)	Tallow fat (g)
20	5.64	5.89	5.99	5.54

Table 7: Hardness of the soap samples with time of present research

Soap samples	Hardness after 3 days	Hardness after 7 days
Palm oil	Still soft	Hard
PKO	"	"
Groundnut oil	"	"
Tallow fat	"	-

follows: palm oil>palm kernel oil>groundnut oil>tallow fat, as given in Table 5. This ranking contradicts the foamability result. This is plausibly explained by the variation in the chemical compositions of the oils/fats sources particularly the stearic acid constant. The tallow fat has the highest constant of stearic acid followed by that of groundnut oil and lastly the palm oil. The stearic acid reacts with the potassium hydroxide to form potassium stearate and glycerin as a by-product. However, it is a common practice to include about 20% sodium stearate in soap products partly to improve the foaming characteristics.

Hardness: The results of the hardness test are given in Table 7 and they revealed that there was no remarkable difference in the hardness of the samples 3 days after production. From Table 7, therefore, it can be said that the type of oils/fats used in developing any soft soaps on their hardness.

The results of the acid, saponification and iodine values are given in Table 8 and they are consistent with the standard specification for toilet (soft) soap.

Overall, the results of the present study of developing soft soap locally in the laboratory are suggesting that for good quality soft soaps, tallow fat is preferred (amongst other oils/fats), but must be statistically designed/ formulated with fillers and possibly foaming agents. Hopefully, this (designing with foaming agent and fillers) would be handled in the part II of the study.

Table 8: The acid, saponification, iodine titre values of various soap samples

Soap samples	Acid value (mg KOH g^{-1})	Saponification value (mg KOH g^{-1})	Iodine value (g/100 g)	Titre (°C)
Palm oil	207	208	46	42
Palm kernel oil-sourced	206	207	45	42
Groundnut oil-sourced	204	205	48	37
Tallow fat sourced	202	203	50	36

The characteristics of the present soap compared favourably with the study standard Encyclopedia American (1980). The characteristics of the present soap compared favourably with the study standard (Table 3-8).

CONCLUSION

The overall conclusions emerging from the development of soft soap via conventional method using hot-batch process by reacting 16.8 g KOH in 300 g H_2O with 50 g PKO, 20 g H_2O, 1.0 g dye and 1.0 g lemon grass perfume for $1^1/_2$ h are that:

- Appreciable quantity of caustic potash was extracted from palm kernel bunch that produced soft soap of comparable quality with the literature standard in terms of foamability, hardness, corrosiveness and rate of wear
- Hot-batch process has proofed to yield 'soft soap' of higher foamability and less corrosiveness (2.52% free alkaline)
- The present method of making soap has yielded soap samples that are not only qualitative and suitable, but reproducible and above all are in conformity with standard specifications (Table 3-8).

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