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Original Article

Comparative studies of the curing and hardening process of soaps produced from locally processed saturated and unsaturated fatty acids

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ABSTRACT

This paper presents a comparative study of the curing and hardening processes of five soaps produced from Nigerian local oils (Palm oil, Palm-kernel Oil, Groundnut oil, Shea-butter oil and Tallow oil). The curing process was investigated by drying the soap samples under natural conditions and obtaining their daily moisture loss for duration of 21 days. Hardness tests based on the Brinell Hardness Methodology was conducted using a locally improvised manual indenter with a test load of 6.585kgf or 64.553N. Graphical plots of the moisture loss versus a one day time interval showed that all the soaps exhibited similar behaviour in the curing process, characterized by exponential decay in the rate of moisture loss. Superimposition of the curves into one graph showed very close fitting between the curves, indicating that the rates of moisture loss are very close. Graphs of the hardening process of the soaps showed that there are similarities in the hardening behaviour of the soaps with the curves exhibiting two hardening regions, a region of non-linear hardening and a region of constant hardening. Results showed that the hardness of the soaps varies in this order: tallow (1.88 HN) >shea butter (0.3 HN) >palm kernel (0.25 HN >palm Oil (0.15 HN) > groundnut oil (0.13 HN).

1. Introduction

Soap may be defined as a chemical compound or mixture of chemical compounds resulting from the interaction of fatty acids or fatty glycerides with a metal radical (or organic base. Soap may also be described as any water-soluble salt of those fatty acids which contains eight or more carbon atoms. The metals commonly used in soap making are sodium and potassium, which produce water soluble soaps that are used for laundry and cleansing purposes [1]. The qualities of soap are determined by the amount and composition of the component of fatty acids in the starting oil. Soap is a mixture of sodium or potassium salts of various fatty acids produced by saponification reaction. It is a substance of ancient origin the manufacture of which has evolved from primitive beginning into a sophisticated chemical process [2]. Soap making is a well-established technology that has progressed tremendously through the years with production of high quality soaps. In the process of this development new raw materials were evaluated and technology modified to accommodate these new materials. In the Soap making industry, raw material choice depend on three important factors, such as properties of the oils or fats, availability and cost competitiveness of raw materials. Soap can be said to be any water-soluble salt of fatty acids containing eight or more carbon atoms.

Soaps are produce for varieties of purposes ranging from washing, bathing, medication and so on. The cleaning action of soaps is due to the negative ions on the hydrocarbon chain attached to the carboxylic group of the fatty acids [5]. Although, the preparation of soaps is same worldwide, it is produced in different varieties for various purposes using either vegetable oils or animal fats. The

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process of making soaps involves saponification/hydrolysis reaction between a fatty acid and an alkaline solution, resulting in the production of long hydrocarbon chain end and a carboxylic acid group that is bonded to a metallic, usually sodium or potassium ion

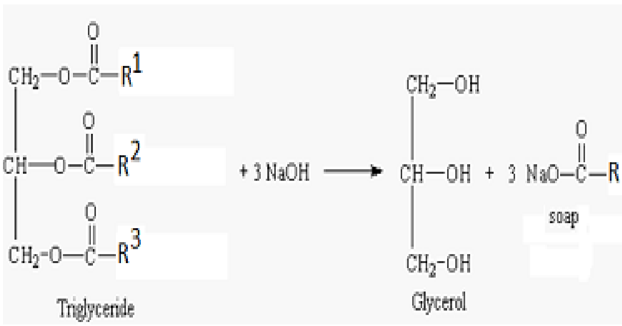

Fig 1. Saponification of Triglyceride (Source: [4])

It is this long chain molecule that is usually called 'soap'. In most cases sodium alkali is generally more widely used as the former alkali produces hard soaps compared to the latter. During the saponification process a by-product known as glycerol is also produce. This by-product is found widely used as a raw material in the cosmetic industries. The hydrocarbon tail end of the soap molecule (hydrophobic) is non-polar and is highly soluble in non-polar solvents such as grease dirt, while the ionic metallic head end of the soap molecule (hydrophilic) is polar and is soluble in polar solvents such as water.

The major characteristic property of soaps that makes them ideal for use in cleaning un-wanted materials is its ability to attract the dirt and removes them from materials surfaces. Most dirt are oil based [7] like the grease of frying fan. Water cannot cleanse oil base dirt because water is a polar solvent and grease is a non-polar solvent and the "like dissolves like'' cannot apply in this situation. When applied to clean the grease,
Water will just rollover the surface of the dirt material.

However, when soap is applied to clean the grease or dirt the non-polar tail of the soap molecules attaches itself to the dirt forming a micellar bi-layer. When water is added to the mixture the polar head of the soap molecules attaches itself to the polar end of the water. The soap acts like a bridge between the water and the dirt and the resulting mixtures called an emulsion. The emulsion being a liquid-liquid phase can now be easily washed away with water, thereby, cleansing the grease from the surface.

Soap has many properties. One of its properties is that it can exist both as a liquid and as a solid and still performs the same function. Liquid soap when allowed to cure under free air or under higher oven temperature can be transformed into a solid soap. The liquid paste losses its moisture content through evaporation and become a solid mass with characteristics hardness. The process by which this takes place is called curing process. Soaps can undergo curing process by losing their moisture content through evaporation to produce a solid mass of hardened particles [3]. Hardness is one of the physical properties of soaps and can be used to determine the quality of soaps. Soap that possesses large hardness can be adjudged as good quality soap and it would be expected to be characterized by a rapid moisture loss.

As such, the major aim and objective of this study is to carry out an in-depth investigation into the curing and hardening process of five different locally manufactured soaps; so as to ascertain the qualities of the soaps.

2. Materials and Methods

2.1 Materials

In this study, materials used for the production of soaps were purchased from the central market in Birnin Kebbi, Nigeria. The unsaturated fatty acids purchased include palm oil, palm kernel oil, groundnut tallow oil (beef oil) and shea butter oils. The production of the soap samples and the hardness tests were conducted at the chemistry laboratory of Federal University Birnin Kebbi, Kebbi State, Nigeria in September, 2019.

2.2 Procedure for Soap Production

200g each of the fatty acid oil was weighed into a beaker and heated to about 80^{o}C on a hot plate with continuous stirring. The content was then poured into a plastic container and allowed to cool down to almost zero degrees centigrade.

Fig 2. Some of the Soap Samples produced using (A) groundnut oil, (B) tallow oil, (C) palm kernel Oil, (D) shea butter oil and (E). palm oil.

The quantity of sodium hydroxide used for saponification of the oils was obtained by multiplying 200g of the oil by its corresponding saponification value. Using the calculated quantity a lye solution of 25% concentration was then prepared and added gradually to the cool oil and stirred thoroughly until a trace level was observed. When the soap has started to solidify the thick viscous paste was quickly transferred into the mould cavity, which was covered with blanket to prevent the soap from absorbing moisture and becoming rancid after solidification. The blanket was removed and the soap was left opened to dry well. The same procedure was adopted in the production of all the soap samples using all the fatty acid oils mentioned previously.

2.3 Hardness Tests of Soaps

2.3.1 Brinell Hardness Principles

The hardness test conducted on the soap samples was based on the Brinell hardness principle. Hardness is defined as the ability of the material to resist wear, abrasion, cutting and indentation of an object pressed onto it [6]; that is, to the impression that the object causes on the materials' surface. Hardness can also be considered as that which represents the material's capacity of resisting permanent deformations. The Brinell hardness principle was based on pressing a hard spherical indenter (normally steel ball of diameter 10mm) with an exactly defined force onto the test piece for 10-30 seconds and obtaining the depth of indentation of the test piece. After that the surface area of the indentation is evaluated using the Brinell equation below [7], and the value obtained gives the measure of the hardness of the test material.

2.3.2 Brinell Hardness Principles

$$\frac{2xF}{\prod Dg(D-\sqrt{}(D^2-d^2)} \quad (1)$$

Where,

F=defined force in kgf or Newton

D = diameter of the indenter

d= diameter of indentation

g = acceleration due to gravity

$\prod$ = pie (3.14)

In this investigation the test equipment used to test the hardness of the soaps was made up of a locally improvised manual indenter. The indenter consists of three parts. A plunger made up of a metallic tubing material, a load bearing container produced from an aluminium electrical socket box, and a steel ball indenter of diameter 10.9mm. All the three major parts were welded together to produce the indenter test piece depicted in figure 3 below

Fig 3. Improvised manual indenter used to test the hardness of the soaps: (A) aluminium weight pan (B) plunger, (C) steel ball holder, (D) steel ball indenter and (E) retort stand

2.3.3 Hardness Test Procedure

The test procedure involves clamping the indenter onto a retort stand in an upright position. There after the indenter was then suspending on top of the soap sample and adjusted until it just touches the soap surface leaving a faint impression on the soap sample. A load of 6.585kgf or 64.5N was then introduced into the weight pan and allowed to dwell for 10-15 seconds. After that, the indenter was removed leaving a round indentation on the soap surface.

Next the size of the soap indentation was determined manually by measuring the horizontal and vertical diameters of the indentation using a ruler.

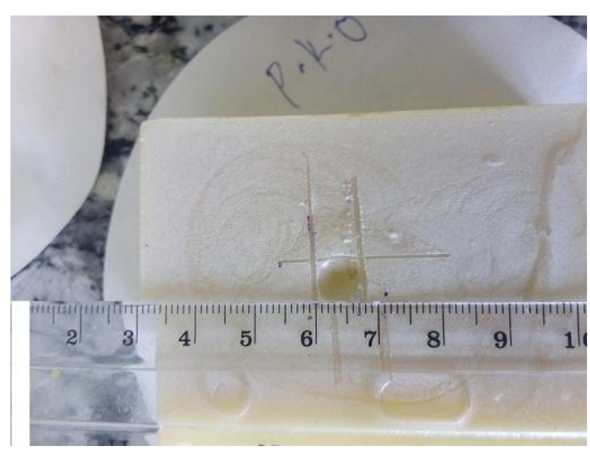

Fig 4. Measurement Indentation Diameter of Soap Samples

The Brinell hardness number (BHN) is a function of the test force divided by the curved surface area of the indentation [7]. The indentation was considered to be spherical with a radius equal to half the diameter of the indentation. The indentation tests were carried out for each soap sample for a period of 10 days. The average diagonal distances of the impression made by the indenter on the soap surface was calculated and used as the diameter of the indentation (d).

2.4 Weight Measurement

The study also conducted the measurements of the soap samples to compare and ascertain the curing process and also the progression of moisture loss for each of the soap sample. Weight measurement was carried out after every 24 hours period from the initial time each soap sample was produced. It was carried out by weighing each soap sample on a weighing balance and the weight loss for each time interval was determined.

3. Results

3.1 Groundnut Oil

Figure 5 shows the three weeks curing and hardening process for groundnut oil soap. Curing was measured as the loss in moisture content of the soap. Each moisture loss for the soap was measured at an interval of 24 hours (1 day) beginning from the time the soap paste was put into a plastic mould. Before recording the moisture loss the soap was removed from the mould and put on a balance to measure the loss in weight. By subtracting the weight of the soap sample after an interval of one day the moisture loss of the soap was obtained.

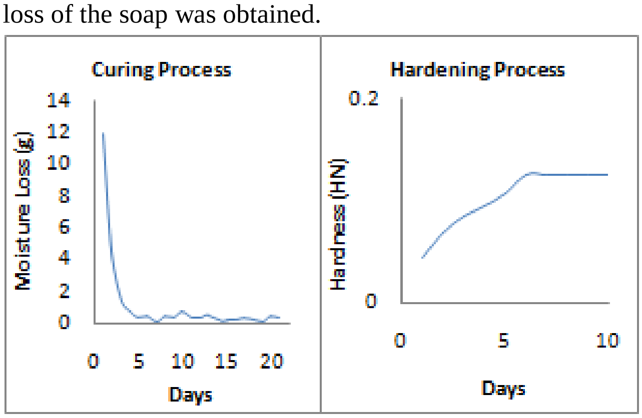

Fig 5. Groundnut Oil Curing and Hardening Processes

As can be seen from the first graph the curing process of the soap displayed an exponential decay features during its curing process. At initial stages, the rate of moisture loss was very rapid and the rapidness in moisture loss decreased exponentially with time to lower values throughout the curing duration. After the first 24 hours of curing time the moisture loss of soap was 11.9g. This dropped sharply to 4.5g after two days of curing. The rate of moisture loss continues to fall rapidly until at day four when it registered a value of 0.715g. The rapid rate of decrease slowed at the fifth day and the moisture loss decayed exponentially to an almost constant value for the remaining period of the investigation. Observation has shown that the decrease in the rate of moisture loss at the constant region of the curve was characterised by wavy fluctuations.

The reason for the waviness may be attributed to the sensitivity of the curing process to changes in atmospheric weather conditions, which varies on daily basis. As such, the rate at which moisture is lost through evaporation from the soap surface depends on the peculiarities of the weather condition for that day. Consequently this is bound to cause the small fluctuations observed at the constant region of the curve. It could also be explained that as the soap was being transformed from the liquid paste to solid mass the thermodynamic driving force that favours evaporation was the dominant process taking place. However, as the liquid paste changes to solid mass the moisture content was finding it difficult to diffuse through the body to get evaporated into the atmosphere and the thermodynamic force was decreasing. This explains the reason why most of the moisture loss was observed at the early stages of the curing process.

Meanwhile, as indicated by the second graph, as moisture was being lost to the atmosphere the hardness of the soap was increasing, initially, very rapidly. For instance, just after 24 hours of exposure the hardness of the soap was 0.044 HN. In contrast to the moisture loss, while the rate of moisture loss was declining the hardness of the soap was increasing rapidly. After day five of hardening process the hardness of the soap became 0.1HN, which is an increase of 150% from day one.

The increases in hardness reached a maximum value 0f 0.13HN at day six of the tests, and thereafter the hardness levelled to a constant value for the remaining days of the investigation. At the constant level the hardness of the soap was 0.13 HN. It may be pointed out at this juncture that the final hardness value of the soap recorded for this test is the ultimate hardness of the soap within the limit of the test load taken (64.533N).

Higher test loads will be expected to give different hardness values for the soap. But we could not go beyond this test load as doing so would not accommodate the hardness of the soap at the early stages of the hardening process.

3.2 Palm Oil

Figure 6 represents the curing and the hardening process of palm oil soap sample. As shown in the first graph a similar characteristics curve observed for groundnut oil soap was also observed for this sample. In general, the curve is said to exhibit an exponential decay in the values of the moisture loss throughout the duration of the curing process. From the curve it can be seen that after 24 hours of curing the soap recorded a moisture loss of 4.25g. There was a sharp increase in moisture loss of 12.24g after the second day of curing. This is in contrast to the groundnut oil soap, where after the second day a decrease in the moisture loss was observed. However, after day two, the moisture loss starts to decrease in an exponential manner until it recorded a value 0.67g at day six of the curing process. Thereafter, the rapid decrease in the moisture loss begins to slide to reach a near constant value, but for the small differences due to the variation in atmospheric weather conditions.

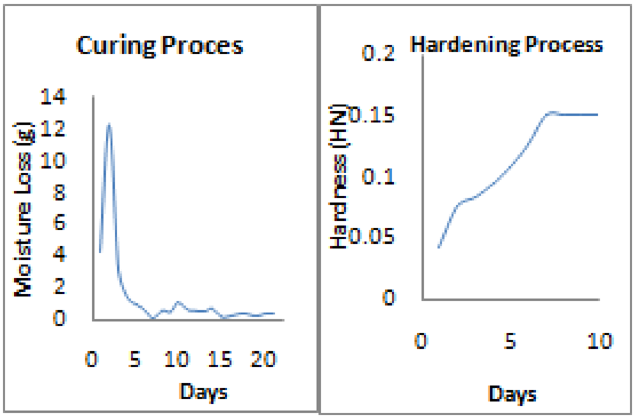

Fig 6. Palm Oil Curing and Hardening Processes

At the same time that the moisture was being lost the hardness of the soap was increasing steadily (see second graph of Figure 5). The hardness of the soap recorded after day one was 0.004 HN. From day one we witnessed a progressive increase in the hardness of the soap, which continue to increase until it reached a value of 0.11HN at day seven of the hardening process. After day seven of curing, the hardness of the soap reached a maximum value of 0.13HN that remained constant for the remaining period of the investigation.

3.3 Palm Kernel Oil

Figure 7 below is the graphical depiction of the curing and hardening process of palm kernel oil soap. As indicated from the first graph, the moisture loss also exhibited a similar trend demonstrated by groundnut and palm oil samples. The moisture loss was characterized by moisture decay. At the first 24 hours of curing the rate of moisture loss of the soap was 2.95g. The rate of moisture loss then increased to 4.92g after 48 hours of curing. However, at day three there was a sharp decrease in moisture loss from 4.92g to 2.37g. The decrease continues until a value of 0.66g was attained at day six of the curing. Subsequently, the sharp decrease in moisture loss slowed down and between the sixth day and the last day of the study the moisture loss decreased exponentially to a near constant value, if not for small irregularities observed here and there.

While the decay in rate of moisture loss was taken place simultaneously the soap hardness was increasing in a non-linear fashion (as indicated by the second graph of Figure 6). The hardness of the soap obtained after 24 hours of hardening for this soap was 0.04HN.

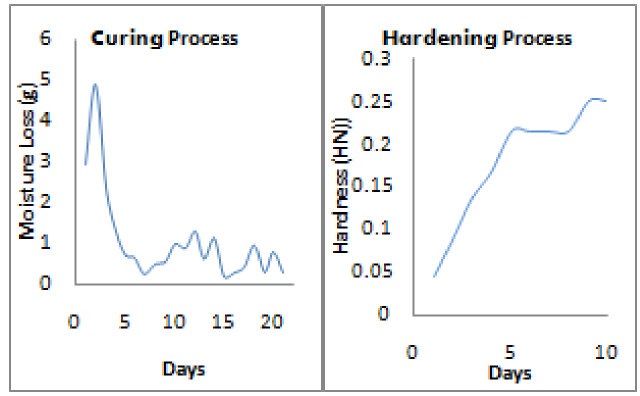

Fig 7. Palm Kernel Oil Curing and Hardening Processes

3.4 Shea-Butter Oil

Figure 8 displays the curing and hardening process of Shea butter oil soap. The curing process followed the same pattern observed for groundnut, palm oil and palm kernel oil soaps. The moisture loss from the beginning to the end showed an exponential decay in its curing behaviour. After day one the moisture loss was 2.76g. At day two, the moisture loss increased to 8.79 in contrast to what was observed for groundnut oil soap. Beginning from day three there was a rapid and large decrease in the moisture loss that continued up to day six where a moisture loss of 0.67g was recorded. Thereafter, the decrease in the moisture loss converged towards a constant value characterized by an exponential decay behavior.

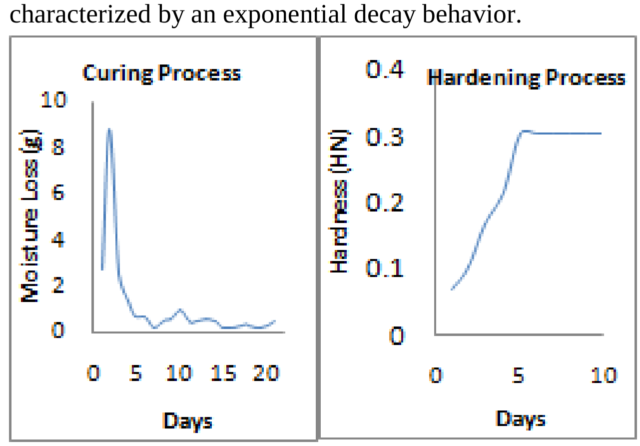

Fig 8. Shea Butter Oil Curing and Hardening Processes

At the same time, the hardening process, depicted in the second graph, showed that the hardness was increasing progressively as the moisture was being lost. Initially, the hardness of the soap, after one day of hardening, was 0.068HN. The hardness rapidly increased to higher values and at day six it reached a peak with a maximum value of 0.3HN. From day six to the final day of the study, the hardness of the soap remained constant within the limit of the test load applied (64.553N).

3.5 Tallow Oil

The curing and hardening process for tallow oil soap is displayed in Figure 9 below. Graph 1 showed that the curing process followed similar trend exhibited by the rest of the previous soaps. As shown from the graph, the curve of the moisture loss has the characteristics feature of an exponential decay function. The moisture loss after day one of curing was 9.68g. After day two the rate of moisture loss decreased sharply to 1.8g reaching a low value of 0.38g at day three of the curing process. Thereafter, the value of moisture loss began to flatten out to a near constant value characterized by an exponential decay behavior; if not for the fluctuations here and there attributable to the effect of atmospheric conditions on the daily rates of evaporation of the moisture. Thereafter, the moisture loss equate to a very near constant value for the rest of the investigation period.

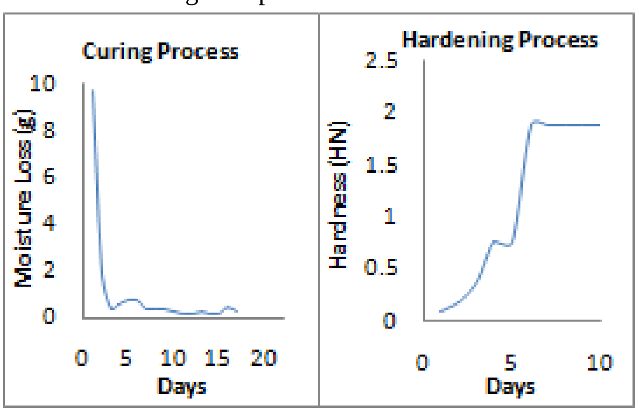

Fig 9. Tallow Oil Curing and Hardening Processes

From graph 2 (Figure 9) the hardening curve showed that as the soap was curing its hardness was increasing. Initially, after day one (24 hours), the hardness of the soap reached 0.08HN. This was followed by a steady steep rise in the hardness of the soap. After day four, the hardness of the soap approached a constant maximum value of 1.89HN within the limit of the test load and for rest of the investigation period.

3.6. Graphical Comparative Analysis of the Five Soaps

Figure 10 below is the plot of the curing and hardening processes of the five soaps investigated. From the first graph it can be seen that there were very close fittings of the curves, signifying that the curing process of the soaps fare similar in trends.

Close fitting of the curves was very pronounced towards the tail end of the curves where moisture loss of all the soaps was approaching a constant value. Although there was convergence in the values of the moisture loss there were still little variations among the soaps during early stages of the curing process. For instance, from the plots it was obvious that palm oil, palm kernel and shear butter oils lost small quantity of moisture during the first day of curing but lost large quantities of moisture after the second day.

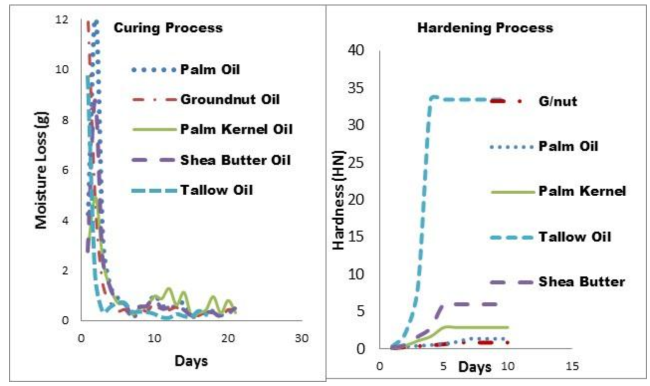

Fig 10. Curing and Hardening Processes of the Soap Samples

On the other hand, groundnut and tallow oils lost large quantities of moisture after the first day and small quantities of moisture during the second day of the curing process. During the mixing process it was observed that the process of dehydration of the tallow oil and the lye mixture was so pronounced that almost 35.5g of moisture was lost before the commencement of the curing process. This may explain why the soap was the most rapid hardening soap of all the other soaps.

The curves of palm oil, palm kernel oil and shea-butter oil soaps showed that the soaps have the same characteristics behaviour in terms of how rapid moisture was being lost. Nevertheless, these soaps lost moisture less rapidly than groundnut oil and tallow oil soaps. While tallow soap and groundnut soap took two and two and a half days respectively to lose 1.8g of moisture it took three and a half days for palm oil, shea-butter oil and palm kernel oil soaps to lose the same quantity of moisture.

With regards to the softness of the soaps, groundnut oil soap was the softest soap from the beginning to the end of the curing process. The next soaps in order of softness were palm oil, palm kernel and shea-butter soaps. The tallow oil soap, which lost about 35.5g of water upon mixing of the ingredients, has the lowest softness of the soaps from the beginning to the end of the investigation.

In terms of the hardening process, graph 2 of figure 9 showed that the rapid hardening of the soaps follow the following order: tallow > shea-butter soap > palm-kernel soap > palm oil soap > groundnut oil soap. Tallow soap had the highest overall hardness of all the soaps. The soap took only one day to reach a hardness of 0.08HN, which is more than 100% higher than the other soaps. While it took 2 days for palm kernel and 3 days for groundnut oil and palm oil soaps to achieve the same hardness. Shea butter oil soap had the next overall hardness, followed closely by palm kernel, palm oil soaps and lastly groundnut oil soap with highest softness of all the soaps.

Statistical analysis of variance for the means of the moisture loss among the various soap samples revealed that there was no significant difference ($p>0.05$) among the soaps.

The result for the means of daily moisture loss between the various soap samples showed that there was no significant difference ($p>0.05$).

Statistical analysis for the effect of duration on the ability of the soaps to lose moisture revealed that moisture loss was not dependent on the duration of the curing process ($p<0.05$).

The result of the statistical analysis for the means of the hardness among the soaps confirmed that there was significant difference ($p>0.05$).

Also, the result of the means of the daily change in the hardness between the soaps showed that there was no significant difference ($p>0.05$).

4. Conclusions

In this study, the soap samples were locally produced from the saponification of unsaturated (plant) and saturated (animal) fats with synthetic sodium alkaline (lye) solution. In order to ensure that optimum saponification reaction takes place for each soap sample a standard saponification value for each oil/fat was used to calculate the volume of the alkaline solution needed to exactly saponify the fat/oil used. For all of the soap samples the soaps were produced using a lye solution of 25% concentration. It was expected that by using maximum saponification stoichiometric quantities of fatty acid and alkaline the soaps produced will contain few, if at all, quantities of un-reacted ingredients. This will ensure that the soaps are produced at optimum saponification levels with standard properties that can be analysed with great accuracy. The study found that all the soaps samples displayed similar trends in both their characteristic curing and hardening processes respectively. The curves exhibited non-linear characteristics behaviour at the initial stages and a constant behavior at the later stages. Previously [3], it has been shown that soaps with the lowest moisture content saponify first and are harder than soaps with higher moisture content at initial stages of the curing process. However, our investigation revealed that the tallow oil soap with second largest moisture content was the most rapid in saponification than the rest of the soaps at initial stages. Saponification of the tallow oil started as soon as the lye and the oil were mixed together. This was followed with the released of large volume of moisture of about 35cm3. Due to the rapid moisture loss, tallow oil soap was considered to be the most rapid hardening soap of all the other samples. The study also revealed that groundnut oil soap containing the next lowest moisture content was the next most rapid moisture lost soap after tallow. Its moisture loss was accompanied with the release of 12cm3 of moisture after 24 hours of curing. Despite this, groundnut oil soap was discovered to be the softest soap amongst all the soap samples investigated a**t** early stages of hardening. From this investigation, it can conclusively be deduced that curing process and hardness are two interwoven processes that take place simultaneously so as to cause changes in the physical and natural strength of soap samples. However, the investigation also uncovered that when the saponification process was optimized by using standard saponification values in determining the stoichiometric ratio of the alkali that would exactly saponify the given quantity of the oil the moisture content does not play a major positive role in determining the initial hardness of the soaps. Rather, other factors such as the properties of the raw-mix may play a significant part in determining the initial hardness of the soap samples.

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Conflict of Interest

The authors declare that they have no conflict of interest

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