UTILIZATION OF AGRICULTURAL WASTE (RUBBER SEED OIL, KOLANUT PODS AND ALMOND LEAVES) FOR SOAP PRODUCTION

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ABSTRACT
Soap is very important commodity with high demand across the world for domestic and industrial cleaning. This research work was aimed at producing soap using agricultural raw materials (Rubber seed oil, Kolanut pods and Almond leaves). The kolanut pods and almond leaves were air-dried, burnt to ashes and alkali was extracted from them by dissolving the ashes in water and later filtered. The kernels from the rubber seed were pounded with mortar and pestle to form a paste. Oil was extracted from the rubber seed paste using n-hexane. The alkali from the kolanut pods and almond leave ashes were made to react with the prepared RSO to form the soaps. The soap samples so produced were analyzed to determine their chemical composition and efficacy. The values obtained from the analysis of the soap samples has revealed that soap produced from locally source agro based waste has a quality very similar to any standard soap. The deviation in the values of the prepared soaps from the standard soap (Joy soap) may be due to the crude nature of the prepared soaps and presence of impurities. These impurities can be removed through purification of the soap. The result obtained from this study has revealed that soap produced using locally source material from agricultural waste can compete favourably with any standard soap.

Keywords – Kolanut pods, Almond leaves, Rubber seed oil, Soap, Saponification

INTRODUCTION
The making of soaps from ash-derived alkalis has been an age-old craft in Nigeria and many West African countries (Nwoko, 1982). Ash - derived alkalis offer cheap alternatives to imported ones. According to Irvine (1965), agricultural waste materials contain a good percentage of potash. These materials include palm bunch waste, kolanut pods, cocoa pods, plantain peels, banana leaves, maize cob, wood, sugar beet waste and many others. When these materials are burnt in air, the resulting ashes contain oxides of potassium and sodium which when dissolved in water yield hydroxides of sodium and potassium.

Rubber tree (Hevea brasiliensis) is exploited in Nigeria mainly for latex in view of its economic importance. The ancillary products namely, wood and the seeds are mostly neglected (Hosen et al., 1981). Of these two products, the seeds have been reported to contain about 42% oil
The yield of seeds per annum in the plantations is estimated to be from 100 to 150 kg/ha (Abdullah et al., 2009). The seeds have been found to be rich in oil. Its content in the dried kernel varies from 35 to 45% (Nwankwo et al., 1985). Rubber seed oil (RSO) has been found to have potential applications in many areas amongst which are in the production of biodiesel as fuel for compression engines (Ramadhas et al., 2005; Ikwuagwu et al., 2000; (Njoku et al., 1995), foaming agent in latex foam (Reethamma et al., 2005), in the synthesis of alkyd resin used in paints and coatings (Aigbodion et al., 2005) and most importantly as oil in soap production (Iyayi et al., 2007).

Natural rubber plant and the RSO are biodegradable, renewable resource and above all environmental friendly. Rubber seed oil (RSO) is unique for soap production because of its relative abundance, level of unsaturation and possession of similar properties with palm oil, which is traditionally employed in the manufacture of soap (Aigbodion, 2000). In addition, due to non use of RSO for edible purposes, it stands out as a veritable substitute for the palm oil which is currently being overexploited in the country.

Commercially, the fat is sourced from tallow, lard, palm oil, palm kernel oil, coconut oil, marine oil, etc. Potassium hydroxide is obtained from electrolysis of potassium chloride using a mercury cathodic cell (Kirk and Othmer, 1954); Sodium hydroxide is obtained from a similar electrolytic decomposition of sodium chloride. Although at present, palm oil and palm kernel oil for the local soap making in Nigeria are readily available, nearly all the alkali for soap making is imported. Furthermore, Nigeria at present lacks the resources to build a modern alkali plant. Following the call by the Federal government of Nigeria, for industries to as much as possible source their raw materials locally, a need has arisen for local supply of the alkali needed in soap making. Sourcing of the alkali from agricultural wastes to replace imported ones has therefore become attractive.

Moreover, Edewor (1984) estimated an annual availability of over 15,000 tonnes of KOH derivable from kolanut pod wastes alone in Nigeria; which more than met the importation requirements of KOH and NaOH of 26,000 tonnes in 1985. Also Onifade (1994) asserted that the dumping of kolanut pod wastes in concentrated heaps on the farms (the usual practice in Nigeria) was adverse to soil fertility and that hogs and other livestock could not completely remove the total wastes available, as fodder. Thus the mentioned agricultural wastes needed to be removed from the farms and were indeed potentially viable resources which should be harnessed.

Manufacture of soap and detergents in Nigeria with the use of imported caustic potash as a source of alkali for soap production has caused price of soap to become very high and expensive due to high exchange rate. In developing countries such as Nigeria, small scale industries have been encourage through micro financing to source for local substitutes for imported raw material. Although at present, palm oil and palm kernel oil for the local soap making in Nigeria are readily available, nearly all the alkali for soap making is imported. Furthermore, Nigeria at present lacks the resources to build a modern alkali plant. It therefore becomes pertinent for local industries in Nigeria to source their raw materials locally; thus a need has arisen for local supply of the alkali needed in soap making. This research work is therefore undertaken to explore the production of solid soap from rubber seed oil (RSO) using kolanut pod and almond leave as source of alkali.
Soap is an anionic surfactants used in conjunction with water for washing and cleaning. It consist of sodium or potassium salts of fatty acids and is obtained by reacting common oils or fats with a strong alkaline solution in a process known as saponification. Soap belongs to the family of detergents which is a substance which improves the cleaning properties of water. The production of crude soap was initiated 3000 years ago in the Nile valley and other early centres of civilisation. The Romans were also known to be considerable users of soap to the extent that urine was used as a source of ammonium carbonate for cleaning purposes (Wigner, 1940).

Fats and oils obtained from animals, olive oil, and vegetable oils, have been the source of raw materials in modern both soda and potash soaps are readily soluble in either alcohol or hot water. In cold water they dissolve more slowly, and owing to slight decomposition, due to hydrolysis (*vide infra*), the solution becomes distinctly turbid. Sodium oleate is peculiar in not undergoing hydrolysis except in very dilute solution and at a low temperature. On cooling a hot soap solution, a jelly of more or less firm consistence results, a property possessed by colloidal bodies, such as starch and gelatine, in contradistinction to substances which under the same conditions deposit crystals, due to diminished solubility of the salt at a lower temperature (Nuttall, 1987).

Soda soaps are insoluble in concentrated caustic lyes, and, for the most part, in strong solutions of sodium chloride, hence the addition of caustic soda or brine to a solution of soda soap causes the soap to separate out and rise to the surface. Addition of brine to a solution of potash soap, on the other hand, merely results in double decomposition, soda soap and potassium chloride being formed.

The solubility of the different soaps in salt solution varies very considerably. Whilst sodium stearate is insoluble in a 5 per cent solution of sodium chloride, sodium laurate requires a 17 per cent solution to precipitate it, and sodium caproate is not thrown out of solution even by a saturated solution (Nuttall, 1987).

Soap making (Laszio,1978). Although local tallow was the major source of fat used in England (Wigner, 1940; Albright and Wilson, 1974), the development of chemical industries has led to the use of alkalis (such as sodium carbonate and sodium hydroxide) for complete saponification in soap making. Soap is produce through a process called saponification. Saponification is the reaction of basic solution, with fats and oils to produce glycerol and salts of fatty acids. The naturally occurring fat is glycerol tristearate. When this is heated with a base such as sodium hydroxide or potassium hydroxide or potassium carbonate conversion occurs forming glycerol and a salt that is soap.

\[
\text{fat/oil} \quad + \quad \text{base} \quad \rightarrow \quad \text{glycerol} \quad + \quad \text{salt (soap)} \\
\text{e.g. glycerol} \quad \text{e.g. sodium} \quad \text{e.g. sodium} \\
\text{tristearate} \quad \text{hydroxide} \quad \text{tristearate}
\]

\[
\text{CH}_3(\text{CH}_2)_{16}\text{COOCH}_2 + 3\text{NaOH} \rightarrow \text{CH}_2 - \text{OH} + 3\text{CH}_3(\text{CH}_2)_{16}\text{COO}^- \text{Na}^+ \\
\text{CH}_3(\text{CH}_2)_{16}\text{COOCH} \quad \text{CH} - \text{OH} \\
\text{CH}_3(\text{CH}_2)_{16}\text{COOCH}_2 \quad \text{CH}_2 - \text{OH}
\]
In its strict chemical sense, soap refers to combinations of fatty acids with metallic bases. Technically speaking, however, the meaning of the term soap is considerably restricted, being generally limited to the combinations of fatty acids and alkanes, obtained by treating various animal or vegetable fatty matters, or the fatty acids derived there from, with soda or potash, the former giving hard soaps, the latter soft soaps.

Other definitions of soap have been given, based not upon its composition, but upon its properties, among which may be mentioned that of Kingsett (1978), who says that "Soap, considered commercially, is a body which on treatment with water liberates alkali," and that of Nuttall (1987), who defines soap as "an alkaline or unctuous substance used in washing and cleansing".

The objective of this study is to explore the use of agricultural raw materials (Kolanut Pods and Almond Leaves) as a source of alkali in soap production. This study also aimed at investigating the use of (Rubber Seed Oil) in place of palm oil as source of fat and oil in soap production.

**EXPERIMENTALS**

2.2  **Collection of samples**
Kolanut pods were collected from kolanut traders within Port Harcourt. The almond leaves were collected from almond plants located in Rivers State University of Science and Technology, Port Harcourt while the rubber (*Hevea brasiliensis*) seeds were obtained from a rubber plantation at Abua Town, Rivers State, Nigeria.

2.3  **Preparation of Sample**
The procedures for the preparation of rubber seed oil is as follows.
1. The rubber seeds were removed from it shells, cleaned and dried under the sun for a day.
2. The kernel will later be dried in the oven for three hours at 35°C to ensure that water and moisture were removed.
3. 1000g of the kernel was immediately ground using mortar and pestle into a paste in order to weaken and rupture the cell.
4. The paste so form was stored in a labeled airtight container for oil extraction.
5. n-hexane was added to the paste in the container and mixed for oil extraction
6. The mixture was then filter to separate the paste from the oil using separate funnel
7. The extracted oil was put in an evaporating dish for 24 hours to evaporate the n-hexane.
8. The oil was later put in an oven at 60°C for another 24 hours to remove any remaining n-hexane.
9. The oil was then collected and stored in a label airtight container for further used.

2.4  **Preparation of Alkali (Almond leaves and Kolanut Pods)**
Procedure for the Extraction of Alkali from Kolanut pods and Almond leaves are highlighted below:
1. About 1000g each of Kolanut pods and Almond Leaves were air dried for 20 days to constant weight.
2. The dried kolanut pods and almond leaves were broken into small pieces and later burnt gradually in an open air.
3. The ashes were left to cool and put in a polythene bag.
4. The ashes were collected and soaked in 1000ml distilled water for two days.
5. The ashes were separated from the liquid extract by filtration using a conical flask, funnels, and filter paper.
6. The resultant extracts/filtrates were kept safe for further use.

2.5. Determination done prior to Saponification of Ash Extract from Kolanut Pods and Almond Leaves

2.5.1 pH

The pH of the Ash Extract of Kolanut Pods and Almond Leaves were measured using a pH meter.

2.5.2 Alkali Concentration of the Ash Extract

Both samples were titrated against 0.1M standard solution of HCl.

2.5.3 Oil and Alkali Mixing Ratio

The oil and alkali was mixed in the ratio 50g:500ml (that is 50g of oil to 500ml of alkali).

2.6 Degumming of rubber seed oil

The degumming of the rubber seed oil was done using water degumming method. Here, hot water was added (2% volume of the total oil) to the oil at a temperature of 70°C to 75°C, and the water and oil was mixed for 10 to 15 minutes. During this period, the Phospholipids absorb water and their Lipophilic characteristics, thus becoming insoluble in the oil. The insoluble Phospholipids later combine to form the gum. Once formed, the gums were separated by centrifugation.

2.7 Physico-chemical characterization of crude and degummed rubber seed oil

2.7.1 Saponification Value

23 Physico-chemical characterization of crude and degummed rubber seed oil
Saponification Value Zg of RSO was dissolved into a 25ml ethanolic potassium hydroxide solution in 250ml round bottom flask with a reflux condenser. The flask was heated in a steam bath and occasionally swirled to effect saponification as the solution starts to boil, the heating was effected for 30mins, after heating: the soap solution formed was titrated with standard. O.5m hydrochloric acid using phenolphthalein as indicator. A blank titration was also done in the same manner. The saponification value (S.V) was expressed as:

\[ S.V = (56.1M \times (u-v))/W \]

Where:
M = Molarity of hydrochloric acid
W = Weight of samples in grammes used
U = Volume in ml of hydrochloric acid titrated in blank and
V = Volume in ml of hydrochloric acid titrated in test.

2.7.2 Iodine Value
The sample under determination was weighed and dissolved in 2Sml of carbon tetrachloride and agitated thoroughly and then allowed to stand for about 30 mins in dark. 100ml of distilled water and 20ml 10% (w/v) potassium iodide was added to the mixture and filtered with 0.1M standard sodium thiosulphate using.

2.7.3 Acid Value
2g of the Rubber seed oil was dissolved in 50lm of neutralized solvent of equal volume of diethyl ether and absolute ethanol. The resultant solution was titrated with 0.1m KOH solution using phenolphalein as indicator. The acid value was calculated using the expression;

\[
\text{Acid Value (AV)} = \frac{(V \times M \times 56.1 \times 0.142)}{2}
\]

Where
V = Volume of titrant (KOH) in ml
M = Molarity of standard KOH
W = Weight of grams of samples.

2.7.4 Oil Content
The oil content is the percentage of oil contain in the seed. The oil content of rubber seed was calculated using this expression in equation (4) in accordance with (Haque et al., 2009).

\[
\text{Oil Content (OC)} = \% \text{ oil} = (w_2 - w_1/w) \times 100
\]  

Where; \( w = \)weight of sample, \( w_1 = \) weight of beaker with glass ball, \( w_2 = \) weight of beaker with glass ball and oil, \( w_2 - w_1 = \) weight of oil.

2.7.5 Heating Value
Heating value, was estimated from iodine and saponification values of the rubber seed oil in accordance with (Fariiku, Ndonya, and Bitrus, 2007) and calculated using equation (5).

\[
\text{Heat Value (H_{fc})} = 47645 - 4.1871V - 38.31SV
\]  

Where = heating value of oil (MJ/kg), IV = iodine value (gI2/100g), SV = Saponification value (mgKOH/g).

2.7.6 Refractive Index
Refractive index was calculated in terms of the iodine, saponification and acid values of rubber seed oil using the expression by (Onwuka, 2005) as stated in equation (6).

\[
R_1 = 1.4643 - 0.0000665 \times 0.0096AV/SV + 0.00017111V
\]  

Where IV = iodine value (gI2/100g), SV = Saponification value (mgKOH/g), AV = acid value (mgKOH/g).

2.8 Preparation of soap
The procedure for the Preparation of Soap Sample (Saponification Process) is highlighted below:

1. 50g of the rubber seed oil was added to 300ml of the alkali extract in a beaker.
2. The mixture was heated to about 55°C with stirring for about 30 minutes.
3. NaCl (table salt) was added to the mixture to separate the soap mass from the glycerol.
4. The mixture was allowed to cool.
5. The soap formed a cake on the surface of the beaker while a solution of glycerol was below.
6. The solution of glycerol was removed by piercing the soap mass and pouring out the solution.
7. The soap sample was stored in a container for further analysis.
8. The pH of the soap was measured.

2.9 Physico-chemical analysis of soap sample

2.9.1 Procedure for the Determination of Total Fatty Matter (TFM) of Soap Sample

1. 5g of soap sample was dissolved in 50ml-distilled water and the volume adjusted to 10ml.
2. The solution was allowed to cool and then made acidic with 0.1m sulphric acid.
3. The solution was then extracted with 30ml diethyl ether and then with another three-15ml portions of diethyl ether.
4. The combined ether extracts was filtered into flask and the ether evaporated.
5. The weight of the total fatty matter was obtained by subtracting the weight of the ether extracts form initial weight of the soap sample.

2.9.2 Procedure for the Determination of Total Free Alkali (TFA) of Soap Sample

1. 5g of soap sample was added to 50ml of neutralized ethanol in a conical flask on steam bath until the soap sample was dissolved.
2. The solution was heated to boiling.
3. 2 drops of phenolphthalein was added to the solution and then titrated with 0.1M sulphuric acid to end point.
4. The total free alkali was calculated as Na2CO3 oxide using the relationship:
Weight (g) of TFA = molarity of acid * molar mass of oxide * volume of acid used.

2.9.3 Procedure for the Determination of Free Caustic Alkali (FCA) of Soap Sample

1. 5g of the soap sample was dissolved a 100ml volumetric flask containing 50ml of distilled water.
2. 5ml of barium chloride solution and 2 drops of phenolphthalein solution were added to the solution respectively and mix.
3. The precipitate was allowed to settle
4. 25ml of the clear liquid was draw off and titrated with 0.1Mol sulphuric acid.
5. The amount of free caustic alkali in the soap was calculated using the relationship: FCA = molarity of acid * formula weight of barium chloride * volume of acid used.

2.9.4 Procedure for Foamability tests on soap sample produced

1. About 5.0g of the soap sample was dissolved in a 1000ml measuring cylinder containing 10ml of distilled water.
2. The mixture was shaken vigorously so as to generate foams.
3. After shaken, the volume occupied by the foam in the cylinder was recorded.
4. The procedure was repeated a second time.

2.9.5 Procedure for Foam Stability tests on soap sample produced
1. 5g of soap sample was dissolved in a measuring cylinder containing 10ml of distilled water.
2. The mixture was shaken vigorously so as to generate foams.
3. After shaken, the cylinder was allowed to stand for some time.
4. The time taken for the foam/lather to completely dispersed/disappeared was recorded as a measure of the foam stability.
5. The experiment was repeated.

2.9.6 Procedure for the Determination of Free Carbonate Alkali of Soap Sample.

Free carbonate Alkali was determined by subtracting the free caustic alkali from total free alkali. Mathematically, Free carbonate alkali = TFA - FCA

2.10 Procedure for the Determination of Wash-Active-Substance (WAS) of Soap Sample
1. About 20g of the soap sample was put in a 100ml beaker.
2. 30ml of neutralized ethanol was added to the soap sample in the beaker.
3. The solution was refluxed for 50 minutes over steam bath and then allowed to settle down.
4. 2-4 drops phenolphthalein was added.
5. The solution was filtered and the resulting precipitate was washed with 25ml neutral ethanol then boiled and filtered into the beaker containing the filtrates.
6. The washing was repeated five times.
7. The combined filtrates were evaporated to dryness over a steam bath and the residue was dried in an oven at 80℃ constant weight.
8. The paste obtained is the W.A.S.
### 3.1 Results for physicochemical analysis of kolanut pods prepared soap sample

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value of prepared soap sample</th>
<th>Value of joy soap sample</th>
<th>Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foamability (ml)</td>
<td>342</td>
<td>384</td>
<td>Marginal differences due to crude nature of prepared soap.</td>
</tr>
<tr>
<td>Foam stability (hr)</td>
<td>7.1</td>
<td>7.5</td>
<td>Almost similar</td>
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<tr>
<td>Total Fatty Matter (TFM)</td>
<td>58</td>
<td>71</td>
<td>Different in oil used</td>
</tr>
<tr>
<td>Total Free Alkali (TFA)</td>
<td>12.2</td>
<td>10.2</td>
<td>Difference in alkalinity of the base</td>
</tr>
<tr>
<td>Free Caustic Alkali</td>
<td>5.2</td>
<td>4.8</td>
<td>Almost the same</td>
</tr>
<tr>
<td>Free Carbonate Alkali</td>
<td>7.0</td>
<td>5.4</td>
<td>Differences due multiple carbonate in ash extract</td>
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<tr>
<td>Wash-Active-Substance</td>
<td>30.8</td>
<td>38.71</td>
<td>Difference is due to presence of impurities in prepared soap</td>
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<tr>
<td>pH</td>
<td>11.1</td>
<td>12.2</td>
<td>Difference in base</td>
</tr>
</tbody>
</table>

### 3.2 Results for physicochemical analysis of Almond leaves prepared soap sample

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<th>Parameters</th>
<th>Value of prepared soap sample</th>
<th>Value of joy soap sample</th>
<th>Remark</th>
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<tr>
<td>Foamability (ml)</td>
<td>280</td>
<td>384</td>
<td>Marginal differences due to crude nature of prepared soap.</td>
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<tr>
<td>Foam stability (hr)</td>
<td>6.9</td>
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<td>Marginal different</td>
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<td>Total Fatty Matter (TFM)</td>
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<td>71</td>
<td>Different in oil used</td>
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<tr>
<td>Total Free Alkali (TFA)</td>
<td>11.5</td>
<td>10.2</td>
<td>Difference in alkalinity of the base</td>
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<tr>
<td>Free Caustic Alkali</td>
<td>4.9</td>
<td>4.8</td>
<td>Almost the same</td>
</tr>
<tr>
<td>Free Carbonate Alkali</td>
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<td>5.4</td>
<td>Differences due multiple carbonate in ash extract</td>
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<tr>
<td>Wash-Active-Substance</td>
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<td>Difference is due to presence of impurities in prepared soap</td>
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<tr>
<td>pH</td>
<td>10.5</td>
<td>12.2</td>
<td>Difference in base</td>
</tr>
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</table>

## CHAPTER FOUR
DISCUSSION, CONCLUSION AND RECOMMENDATION
4.1 Discussion

Formability: The result obtained from the analysis of the soap samples for formability gave 342ml for kolanut pods and 280ml for almond leave. The results are lower than the ones obtain from the analysis of standard Joy soap (384ml). These differences may be due to the crude nature of the soap sample and raw materials.

Foam Stability: The result obtained from the analysis of the soap samples for foam stability from gave 7.1 hrs for kolanut pods and 6.9 hrs for almond leaves. These values are difference from the result obtain from the analysis of standard Joy soap (7hrs 40mins).

Total Fatty Matter: The result obtained from the analysis of the soap samples for total fatty matter (TFM) gave 58% for kolanut pod and 55% for almond leave. The results are lower than the result obtained from the analysis of standard Joy soap (71%). The differences in values may be due to the differences in the composition of oil used for the soap production process.

Total Free Alkali: The result obtained from the analysis of the soap samples for total free alkali (TFA) gave 12.2% for kolanut pods and 11.5% for almond leaves. These values are higher than the result obtained from the analysis of standard Joy soap with 10.2%. This increase may be due to the differences in alkalinity of the soap samples.

Free Caustic Alkali: The result obtained from the analysis of the soap samples for free caustic alkali (FCA) gave 5.2% for kolanut pods and 4.9% for almond leave. These values are almost similar to the result obtain from the analysis of standard Joy soap with 4.8%.

Free Carbonate Alkali: The result obtained from the analysis of the soap samples for free carbonate alkali gave 7.0% for kolanut pods and 6.6% for almond leaves. These values are higher than the result obtained from the analysis of standard Joy soap with 5.4%. This increase may be due to the presence of multiple carbonates in the ash extract.

Wash – Active – Substance: The result obtained from the analysis of the soap samples for wash-active-substance gave 30.8% for kolanut pods and 24.60% for almond leaves. These values are lower than the result obtained from the analysis of standard Joy soap with a value of 38.71%. This increase may be due to the presence of multiple carbonates in the ash extract.

4.2 Conclusion

The result obtained from the analysis of soap produced in this work using rubber seed oil (RSO) as source of oil kolanut pod and almond leave as source of alkali shows that the soap samples produce from agro-base waste can compete favorably with other toilet soaps. The apparent deviation may be due to their crude nature and that of raw materials. Foaming efficiency can be taken care of by addition of water softeners and scum dispersant which will help in lowering the
surface tension of the water. These impurities are responsible for some of the differences seen in the soaps which were produced. These impurities can be removed by boiling the crude soap curds in water and re-precipitating the soap with salt.

4.3 Recommendation
The result obtained in the study has clearly shown that soap with similar characteristic and washing efficiency can be produced using locally source materials from agricultural waste and non edible oil. It is recommended that if the soap produce from locally source material are well purified, they can compete favourably with other toilet soap. It is therefore recommended that government at all levels in Nigeria should set up alkali processing plants to convert our agro-waste to wealth. Also, soaps, if well prepared can be used as inexpensive sources of soap for industrial cleaning.
The use of raw materials from agricultural waste for the production of soap have left so much room for manufacturers to explore and satisfy the tastes of soap users, create more jobs as well as make profits and further investments.

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