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Production of Biodegradable Detergent from Castor Oil

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I. Introduction

Castor oil has long been used commercially as a highly renewable resource for the chemical industry. It is a vegetable oil obtained by pressing the seeds of the castor oil plant (*Ricinus communis* L.) that is mainly cultivated in Africa, South America, and India. Major castor oil-producing countries include Brazil, China, and India. Even though castor oil accounts for only 0.15% of the world production of vegetable oils, worldwide consumption of this commodity has increased more than 50% during the past 25 years, rising from approximately 400,000 tons in 1985 to 610,000 tons in 2010 (Scholz, 2008). On average, worldwide consumption of castor oil increased at a rate of 7.32 thousand tons per year. In general, the current rate of castor oil production is not considered sufficient to meet the anticipated increase in demand (Ogunniyi, 2006, Mutlu, 2010, Thomas, 2000).

Soap industry, has roots over 2000 years in past, a soap factory having been found in the Pompeii excavation. Soap itself was never actually "discovered", but instead gradually evolved from crude mixture of alkaline and fatty acid (George, 1984). Scientifically, the term detergent covers both soap and synthetic detergent, or "syndets" but it is widely used to indicate synthetic cleaning compounds as distinguished from soap. Detergent differs from soap in their action in hard water. Although soaps are excellent cleansers, they do have disadvantages. As salts of week acids, they are converted by mineral acids into free fatty acids. These fatty acids are less soluble than the sodium or potassium salts and form precipitate or soap scum. Because of this, soaps are ineffective in acidic water. Also soaps form insoluble salts in hard water such as water containing magnesium, calcium or iron. The insoluble salts from bath rub rings, leave film that reduce hair luster, and gray/roughen textiles after repeated washings. Synthetic detergents, however, may be soluble in both acid and alkaline solutions and don't form insoluble precipitate in hard water (Hong, 2015, McKeon, 2016, Shrirame *et al.,* 2011, Tewari, 2012).

All the activities of man, starting from the primitive farming techniques to today's high technology industrial activities have in small or large ways impacted negatively on man and his environment while the various products developed are highly desirable for the enhancement of the citizenry's well being and sustenance of nations' economy, the negative impacts precipitated by the introduction of its unwanted by-products into the ecological systems may be catastrophic if allowed to build up and uncontrolled. Industrial revolution and evolution have been targeted principally at satisfying immediate changing demands rather than tailored towards a structured, wholesome and guided global program that will satisfy not only temporary human needs but are environmentally safe. The result of this is an increasing ecological degradation that has severely polluted water, land and air (Odigure, 1998).

The detergent and soap making industries are no exceptions to the above trends, for while they provide us with cleansing agents, their processing and by-products are also a cause of public nuisance. For instance, detergents, unlike soaps, have proved very effective cleansing agents in hard and cool water whereas soap is often wholly ineffective under such condition. It was observed, however that many of these detergents were neither soluble nor biodegradable, that is they were so stable that when they flow into the soil in laundry sewage water, they remain unchanged, resisting conversion into less complex and more soluble substances. They thus, create suds and foams in fresh tap water, naturally occurring ground and surface waters (Severino et al., 2012).

This paper is aimed at producing a biodegradable detergent from castor oil with the objectives of replacing the non-biodegradable

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detergents. Production of environmentally friendly biodegradable detergents (soft detergents) from castor oil has the dual advantage of using locally sourced raw material that can be grown and generating wastes that are appetizing to micro organisms (bacteria).

II. Materials and Methods

The castor seeds (or beans) were gotten from the Kawo market at Kaduna, Kaduna state. The operations involved in extraction of castor oil are:

- Pre-treatment of the castor seeds
- Solvent extraction of the castor oil

The pre-treatment of the castor oil and the solvent extraction was carried out in the department of Chemical Engineering, Ahmadu Bello University Zaria.

a) Pre-treatment of castor seeds

The pre-treatment involves the preliminary preparations of the seeds in the following:

Shelling: This involves the removal of the shells to obtain the seeds. It was done manually.

Clearing: This involves the removal of foreign matter introduced during the sun drying and any unshelled seed.

Drying: The moisture content of the seed was reduced using the electric oven. The oven was operated at 80°C for about three hours.

Crushing: Mortar and pestle were used to reduce the sizes of the dry seeds so as to increase the interfacial area between the solvent and the seeds.

b) Extraction of castor oil

100ml of n-hexane was poured into the round bottom flask. 10g of castor beans was placed in the thimble and inserted in the center of the soxhlet extractor as seen in the figure. The extractor was heated at 70°C when the solvent was boiling; the vapor rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the center which contains the solid sample to be extracted. The extract sips through the pores of the thimble and fills the siphon tube where it flows back down into the round bottom flask. This was allowed for 30mins after which the sample was removed from the tube, dried in the oven, cooled in the desiccators and weighed to determine the amount of oil extracted.

The experiment was repeated by placing 5g of the sample into the thimble again, and after every 30mins, the samples were withdrawn for drying and weighing. The miscella (extracted oil mixed with solvent) was heated at the end of the extraction to recover the solvent from the oil. The solvent free oil was then refined for further use.

c) Refining of castor oil

The refining was needed to remove gum from the extracted oil. Boiling water was added to the oil and the mixture stirred for 2 mins and allowed to stand in the separating funnel. The aqueous layer was then removed. The procedure was repeated to ensure removal of most gums. The de-gummed oil was collected and stored for use.

d) Determination of saponification value

2g of the oil was placed in a conical flask to which 25ml of ethanoic potassium hydroxide (0.1M) was added and the mixture allowed to boil gently for about 60 mins with shaking at regular intervals of 5mins.

Few drops of phenophthalin indicator, as specified by International Standards Organization (ISO 3657, 1988) was added to the warm solution and then titrated with 0.5M HCl. The end point was reached when the pink colour of the indicator just disappeared. The same procedure was followed for the blank.
The saponification value (sv) is given by:

$$sv = 56.1 \times N \times \frac{V_o - V_i}{m}$$

where: V_o = volume of HCl solution used for the blank test, V_i = volume of HCl solution for the determination, N = actual molarity of HCl used, and m = mass of sample

e) Production of detergent

0.1M sodium hydroxide solution was prepared by weighing 40g of NaOH Pellets into a beaker containing 100ml of water and shaking vigorously.

The electric hot plate was switched on; 30ml of castor oil in a stainless steel plate was placed on it and heated at 35°C for about 2 mins. Caustic soda (0.1M) was added and the mixture stirred with a glass rod. 18M sulphuric acid was then added with constant stirring, and the reaction allowed to completion after which hydrogen peroxide (Bleaching agent) was introduced into the reaction mixture. When the foaming had subsided, the heating was continued to allow for more vaporization before putting off the heating system. Finally, perfume was added and the system was allowed to cool. The powdered detergent formed was the subjected to foamability test to ensure the effectiveness of the process.

The same steps were then followed, but this time palm kernel castor oil was used as the base material. The resulting powdered detergent formed was again collected and tested as above. The results are as shown in Tables 1 - 3, while the equations of the chemical reaction of the process are:

Sulphation

$$CH_3(CH_2)_5CHOHCH_2CH = CH(CH_2)_7\,COOH + H_2SO_4 \rightarrow CH_3(CH_2)_5\underset{\displaystyle O-SO_3H}{\underset{|}{CHOH}}CH - CH(CH_2)_7CO_2H$$

Neutralization

$$\underset{\displaystyle O-SO_3H}{\underset{|}{CH_3(CH_2)_5CHOHCH}} - CH(CH_2)_7CO_2H + NaOH(\partial q) \rightarrow \underset{\displaystyle O-SO_3H}{\underset{|}{CH_3(CH_2)_5CHOHCH}} - CH(CH_2)_7CO_2N\partial + HOH$$

Vaporization

$$\underset{\displaystyle O-SO_3H}{\underset{|}{CH_3(CH_2)_5CHOHCH}} - CH(CH_2)_7CO_2N\partial + HOH + he\partial t \rightarrow CH_3CH_2)_5\underset{\displaystyle O-SO_3H}{\underset{|}{CHOHCH}} - CH(CH_2)_7COON\partial$$

f) Foamability tests on detergents produced

About 2.0g of the palm kernel based detergent was added to a 500ml measuring cylinder containing 100ml of distilled water. The mixture was shaken vigorously so as to generate foams. After shaken for about 2mins, the cylinder was allowed to stand for about 10mins. The height of the foam in the solution was measured and recorded.

The same steps were followed using the detergent produced with the castor oil so that the foamability of the two could be compared. The results obtained are as recorded in Table 1, 2 and 3.

III. Results and Discussion

The results obtained for the percent volume of oil extracted, determination of saponification value and the production as well as the foamability tests of the detergent produced are as seen in Table 1-3.

Table 1: Average percent of oil extracted

Weight of raw sample W_1(g)	Weight of raffinate W_2(g)	Weight of oil extracted (g)	Percent oil extracted W(g)
25.00	16.45	8.55	34.20
20.00	13.20	6.80	34.00
30.00	22.00	8.00	26.67
35.00	26.80	8.20	23.43
40.00	29.10	10.90	27.25

Table 2: Comparing the saponification value of refined oil with the standard

Property	Refined castor oil	Standard value
Saponification value	183.7275mgKOH/g of oil.	176 - 187 mg KOH/g of oil

Table 3: Height of foam formed by the detergents produced

Detergent base material	Height of foam formed in water (cm)	
	Sample 1 (2.0g)	Sample 2 (2.0g)
Castor oil	2.57	2.60
Palm kernel oil	2.10	2.00

IV. Discussions

The results obtained for the extraction showed an average percent oil extracted to be 29.11%. This value is low relative to similar works done by Isah, Alhassan and Garba, 2005 using n-hexane as solvnent with an average oil yield of 32.1%. The low yield could be attributed to the nature of the seeds and difference in solvent. The oil quality was very desirable as demonstrated from the saponification value of 183.7275 mg KOH/g of oil, which compares very favorably with that in literature (180.00 mg KOH/g of oil).

Moreover, the sulphation and neutralization reactions gave a powered detergent of high enough efficiently as seen from the result of the foamability tests. Usually, the efficiency of a washing powder is assessed through the amount of foam it is capable of producing. The presence of persistent foam exemplifies a good detergent (Bajar *et al*, 1995). The foam height of 2.6cm persisted for about 10 minutes and is higher than that formed by the palm kernel based detergent. The detergent formed was the result of the esterifications of the castor oil.

When ricinoleic acid (castor oil) is treated with concentrated H_2SO_4, its gives a complex mixture consisting of hydrogen sulphate (OSO_3H) of ricinoleic acid in which the hydroxyl group is esterified and a compound in which the H_2SO_4 has added to the double bond. Esterification and addition do not occur together in the same molecule of ricinoleic.

The product which is known as Turkey red oil (sulphated castor oil) has good wetting properties. Neutralization of this with aqueous NaOH gave a detergent plus water. The reaction proceeded at temperatures between 35 - 40%. The water was vaporized by further heating and a solid (powdered) detergent was the result. The bleaching agent (H_2O_2) added helped to bleach the color of the castor oil so that milk colored detergent was produced. pH tests showed that the detergent exhibited basic property. The detergent can thus be described as amphoteric. This classification is characteristic of the intrinsic property of

castor oil. This pH range is preferable to that of acidic as it is non - corrosive to the skin and cloths.

From the structure of castor oil (Ricinoleic acid) as shown under literature review, the sulphation reaction occurred at the hydroxyl group while the esterification reaction occurred at the ester linkages and this can be used to produce both soluble and insoluble soaps. Hence the detergent produced was the result of the esterification of the ricinoleic acid.

V. Conclusions

The extraction of biodegradable detergent was done using n-hexane as the solvent. The oil was refined by de-gumming to remove most gums. Sulphation and neutralization of the refined oil gave a detergent. Other operations like bleaching, perfuming and drying were done to improve color, scent and texture of the detergent. Although many other additives such as optical brighteners, extenders, re-deposition inhibitors, enzymes, etc. were not added, the active ingredients (surfactants) were used and as such the detergent efficiency was high.

The production of synthetics detergent from castor oil was successively done. The many desirable intrinsic qualities of castor oil makes it very useful in the detergent industry, thus castor oil can serve as a good substitute to petroleum and coal, the conventional detergent bases.

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