LEAD SALT-ETHER SEPERATION OF FATTY ACIDS FROM PALM OIL

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(Received, March 22, 2005)

ABSTRACT

This study resulted from the researcher’s awareness of scarcity of fatty acids in chemical laboratories and industries in Nigeria despite the fact that we have many Agricultural products containing these acids, for example, palm oil in which Nigeria is one of the leading producers in the world before the oil boom can serve as a source of saturated and unsaturated fatty acids. The oil was bleached using activated charcoal; the bleached oil was saponified and acidified to obtain the mixture of fatty acids. Out of 100g, 79g of bleached oil was obtained. The unsaturated fatty acids collected from 3g of mixture was 1.56g (52.0%) and that of saturated was 1.34g (44.6%). This shows that palm oil can serve as a drying as well as non-drying oils.

Key word:- Agricultural products, fatty acids, palm oil and lead salt-ether.

INTRODUCTION

Palm oil is believed to have originated from West Africa and later introduced in Congo, South East Asia and South America along the equatorial belt (Hartley, 1962). In the 1830s 11,000 - 14,000 tons of palm oil were exported per annum from West Africa. By 1870 export was 25,000 - 30,000 tons; in 1911 it was 87000 tons in which about 75 percent came from Nigeria. Nigeria, the largest exporter until 1965, took part in expanding world trade in palm oil. There was a set back in 1966 to 1973, in 1974 Nigeria came second as the largest producer of palm oil, thereafter, the oil boom came and paralysed the production of palm oil.

Available statistics show that vegetable oil imports into Nigeria rose from 57,911 metric tons valued at N93 million in 1991 to 84, 252 metric tons valued at N508 million in 1992, while in 1993, it was 77,577 metric tons valued at N957 million. Therefore, the search to expand the production of palm oil which have the highest yield of oil per unit area of all the known vegetable oil became imperative and the dual usage as drying and non drying oil. Despite the apparent popularity of petroleum products as raw materials in different areas of application, fats and oils are greatly favoured for use in surface coatings, soaps, cosmetics, pharmaceuticals, lubricants and polymer processing (13-18).

Palm oil is obtained from palm tree (Eleasis guinensis). The palm oil give the highest yield of vegetable oil per unit area than any other oil seed crop and also produces two distinct oils: palm kernel oil derived from the seed (kernels) and palm oil derived from the fleshy mesocarp of the fruit which contain 45-55 percent oil. Palm oil melts over a temperature range of 25-50 °C, the colour ranges from yellow to orange - red depending on the amount of carotenoids present; (Weiss, 1983) the amount of oxidation by lipoxidases in bruised fruit stored for various period before processing,
and oxidation, catalysed by iron during processing and bulking (Purseglone, 1972).

The operations involved in the processing of palm fruits to palm oil can be briefly summarized as follows:

STERILIZATION → STRIPPING →
MILLING → SEPARATION →
PRESSING → CLEARIFICATION →
STORAGE OR SALE
(Opeke, 1987)

The demand for vegetable oil has recently increased considerably as they are used as raw material for expanding industries. Our dependence on it can be expected to increase further owing to the fact that they are renewable resources and their products are more environmentally friendly (Thomes and Yu, 1996; Erhen and Bagby, 1995) as against the constant depletion of non renewable mineral resources e.g. crude oil (Choo, 2001).

They are recognised as vegetable oils and thus their derivatives and applications are fully acceptable to people whom animal fats may be subjected to religious taboos. The two main non food industrial uses of palm oil are in the manufacture of oleo chemicals and soap. Oleo chemicals refer to chemicals obtained from palm oil and can be divided into two groups of products; i.e. basic oleo chemicals and oleo chemical derivatives (Ong, 1990).

| RAW MATERIAL | BASIC OLEOCHEMICAL | OLEOCHEMICAL DERIVATIVES |
|--------------|---------------------|--------------------------|
| palm oil     | fatty acids         | fatty alcohol sulphates, alcohol thioxylylates |
|              | fatty alcohols      | fatty alcohol sulphates, alcohol ether sulphates, fatty alcohol thioxylylates |
|              | methyl esters       | alpha-sulphonated fatty acids, methyl esters, alkylamides, sucrose esters, soap |
|              | Glycerol            | alkyd resins, glycerides |
|              |                     | Soaps, epoxides, fatty acids, Alkanol amides |
|              | quaternary ammonium compounds, amine oxides, diamines |

Oleochemicals from palm oil (Ong, 1990).
Palm oil contains nearly the same amount of saturated and unsaturated fatty acids. The saturated fatty acids proportion in palm oil is 43 percent of palmitic acid and 4.5 percent of stearic acid. The monosaturated portion in palm oil is composed by oleic acid and being 41 percent of its composition. 10 percent of palm oil are composed by linoleum acid (polyunsaturated fatty acid) which is also an essential fatty acid (Eckey, 1970).

The objective of this work is to reveal to some extent how fatty acids could be extracted from palm oil and to separate it to saturated and unsaturated. The work is to bring to light the technology which can be locally developed for the utilization of farm products like palm oil which contain nearly equal percentage of saturated and unsaturated fatty acids which are used differently in chemical industries and laboratories. In the literature, various work has already been reported starting from breeding to its utilization and its composition by GC and GC/MS but much has not been reported in getting them separated in commercial scale to saturated and unsaturated as reported here.

MATERIALS AND METHODS

The palm oil used was obtained from a farm where it is being prepared in Isinbode-Ekiti of Ekiti State, Nigeria.

Bleaching of the Palm Oil:

The bleaching was performed by weighing 100g of palm oil into a 500cm$^3$ flask and heated over a hot plate to 90$^\circ$C, 40g bleaching material used (activated charcoal) was added and the temperature was further raised to 120$^\circ$C, this temperature was maintained constant for 30 minutes. It was then cooled and filtered at a temperature of 75$^\circ$C.

Saponification of the bleached palm oil:

12g of palm oil was mixed with 25cm$^3$ of concentrated sodium hydroxide in a porcelain dish. The mixture was heated on a Bunsen burner for 30 minutes until it started to boil gently. The mixture was stirred with a rod to prevent bubbling. Distilled water was added from time to time to maintain the initial volume of the mixture. Completeness of saponification was tested for at the end of 30 minutes as follows: a few drops of the soap prepared was transferred into a test tube and 5cm$^3$ of distilled water was added. The mixture was heated with shaking on water bath until all dissolved without fat drops, showing that saponification was completed. 30cm$^3$ of hot saturated mixture was allowed to settle and cool. The soap floated to the surface of the aqueous solution and was then separated.

Isolation of mixed acids from soap:

5g of salted-out soap was weighed into a test tube, this was dissolved with 20cm$^3$ of distilled water under heating in boiling water bath. Excess 1M H$_2$SO$_4$ was added top to the soap solution formed in the test tube to acidify it. A separating funnel was used to separate the layers formed. The oil layer containing fatty acid was on the top and this was collected after draining off the first layer which contains an aqueous solution. The sample collected was then washed with distilled water and was separated using a separating funnel before being put inside a dessicator for drying.

Separation of saturated and unsaturated fatty acids from the mixture of fatty acids:

Lead salt-ether separation was used to separate saturated fatty acids and unsaturated fatty acids from the mixture of fatty acids.

3g of fatty acid mixture was neutralised with 15cm$^3$ of warm alkali solution (NaOH). After neutralisation, excess saturated
lead acetate solution was added to the soap solution in a round bottom flask. The mixture was heated in a water bath the lead salts of the fatty acids were precipitated all over the inner surface of the round bottom flask. The flask was then cooled and the precipitate was allowed to settle completely. After settling, distilled water was used to wash the precipitate three times. After draining off as much water as possible from the flask, excess ether was added (diethyl ether). The lead salt and ether were boiled together on a water bath under reflux for two hours, then the whole was cooled in a refrigerator at 0°C for 24 hours, after this period the solution was filtered. The filtrate contains the lead salt of the unsaturated fatty acids. The lead salts were obtained by removing the ether by heating in water bath in the fume cupboard. Later the fatty acids (unsaturated) were obtained by adding 15 cm³ 1 M HCl into the mixture of lead salts under boiling. There was a precipitate of lead chloride and the acids float on top. Separating funnel was used to separate out the mixture of the acids.

RESULTS AND DISCUSSION

The amount of oil got from 100 g of palm oil after bleaching was 80 g, this shows lost of 23 g due to the presence of impurities, these are compounds which are not straight chain compounds, they are called bulky molecules, they are highly conjugated compounds, examples are carotenoids, tocopherols e.t.c. They cannot pass through it (Purseglone, 1972, Weiss, 1983 and Ucko, 1988).

The unsaturated fatty acids collected from 3 g of mixture of fatty acids after the experiment was found to be 1.56 g (44.6%). This is in agreement with the percentage composition of fatty acids in palm oil by GC analysis¹⁷, Anonymous, 1991. The colour, physical state and mass in gram is as shown below.

| Colour | Physical State | Mass (g) | % |
|--------|----------------|---------|---|
| Saturated Fatty acids | Butter Solid | 1.34 | 44.6 |
| Unsaturated Fatty acids | Yellow Liquid | 1.56 | 52.0 |

The reaction taking place in the bleaching of palm oil is adsorption. The bulky molecules are adsorbed to the surface of the activated charcoal whereas the straight chain molecules (fatty acids) pass through. The efficiency of adsorption depends on the surface area of the charcoal, the more it adsorbs the bulky molecules (Robert and Marvin, 1982, Weiss, 1981).

The conversion of palm oil to soap is alkali hydrolysis known as saponification.

\[
\text{H}_2\text{C} - \text{O} - \text{C} - \text{R}_1 + 3\text{NaOH} \rightarrow \text{CH}_2\text{OH} + 3\text{RC} - \text{ONa}
\]

The soap formed is hydrolysed with acid to give fatty acid.

\[
\text{RCOONa} + \text{HCl} \rightarrow \text{RCOOH} + \text{NaCl}
\]

In the separation of saturated and unsaturated fatty acids from the mixture of fatty acids using lead salt-ether separation method, what happens is that lead salt of unsaturated fatty acid is soluble. The reaction taking place are:

For saturated fatty acid:

\[
[\text{CH}_3\text{COO}]_2\text{Pb} + 2\text{CH}_3[\text{CH}_2]_n\text{COONa} \rightarrow
\]
\[ 2\text{CH}_3\text{COONa} + [\text{CH}_3(\text{CH}_2)_n\text{COO}]_2\text{Pb} \rightarrow 3 \]

For unsaturated fatty acid:

\[ [\text{CH}_3\text{COO}]_2\text{Pb} + 2\text{CH}_3\text{CH}=\text{CH}(\text{CH}_2)_n\text{COONa} \rightarrow 2\text{CH}_3\text{CH}=\text{CH}(\text{CH}_2)_n\text{COOH} + \text{PbCl}_2 \downarrow \]

When the lead salt of both saturated and unsaturated fatty acids is hydrolysed with HCl, free fatty acids are set free and lead chloride is precipitated.

\[ [\text{CH}_3(\text{CH}_2)_n\text{COO}]_2\text{Pb} + 2\text{HCl} \rightarrow 2\text{CH}_3(\text{CH}_2)_n\text{COOH} + \text{PbCl}_2 \downarrow \]

The present results indicate that palm oil is a good source of both saturated and unsaturated fatty acids and can be considered as a balanced fat because it contains nearly equal quantities of saturated and unsaturated fatty acids. Also, the lead chloride precipitate from the reaction can be utilised as another path of getting lead chloride for our industry.

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