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# Extraction and characterization of seed oils

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A b s t r a c t. All the seeds examined in this project have been shown to contain varying levels of oils, mainly in the range of 26-42%, with the exception of *Detarium microcarpum* which contains about 7% of oils. Characterization of the oils by standard techniques suggest that they contain high levels of saturated fatly acids, judging by their low iodine values (*IV*) which did not exceed 88 in all cases. They are, hence, not suitable as alkyd resins for paint formulation but may, however, be used for soap production judging by their high saponification values (*SV*) in the range of 199-261. *Lophira lanceolata* showed considerable reduction in *IV* and increase in *PV* over a period of one month under storage conditions of light , darkness and refrigeration. In light, the *IV* value of 65 dropped by 50% at the end of one month, while under the same conditions the *PV* increased by almost tenfold. Less profound changes in both *IV* and *PV* were observed for oil stored in darkness and under refrigeration. The observed profound changes were explained as arising from oxi- dative rancidity of the oils. The nutritional non-oil residue of *Lophira lanceolata* may be suitable as animal feed judging by the balance of its nutrient composition.

K e y w o r d s: seed oils, iodine value, saponification, value, peroxide value, storage

## INTRODUCTION

A lot of work has been carried out on analysis of seed oils by a number of workers, primarily because of extensive demands for oils both for human consumption and for industrial applications; consequently there is an increasing need to search for oils from non-conventional sources to augment the available ones and also to meet specific applications.

Abakaka *et al.* (1989) studied oils from rubber for their peroxide value and iodine value (*IV*). They also gave values for the saponification number and moisture content. They concluded that the values were comparable to those of palm oil and groundnut oil. The iodine values and the peroxide value were 65 and 27.5 (ml $kg^{-1}$), respectively.

The composition of the seed oil of *Chlorophora excelsa* was analysed with a view to determining the nutritional value of the oil Idigo (1989).

Adesomoju and Akinbo (1996) reported that the esterified fatty acids of the seed oil of *Chlorophora excelsa* were examined by gas chromatography (GC) and gas chromatography mass-spectrometry (GC-MS), and palmitic linoleic stearic acids were found to be the component acids of the seed oil of *C. excelsa*. The high percentage of linoleic and palmitic acids in the oil indicated several potential uses for the oil of *C. excelsa*.

Kar and Mital (1999) reported that shea butter is a natural fat obtained from the seeds of the shea tree *Butyrespermum parkii,* The shea butter fat was extracted from the seeds with various organic solvents, namely petroleum ether, n-hexane, chloroform and benzene. It was concluded that these solvents, particularly petroleum ether and n hexane, could be used for the extraction of shea butter that is free from any oxidized fat and colouring impurities. Akpan *et al.* (1999) carried out extraction characterization and modification of castor seed oil and reported that tested parameters which include specific gravity, refractive index, acid value, saponifiation value and iodine value for both crude and refined castor oil produced were within the ASTM standard specifications. In fact the iodine value obtained (84.8) for the refined oil indicates that the oil could certainly be used as a lubricant, hydraulic brake fluid and protecting coatings. Cary *et al.* (2000) investigated the fatty acid and natural product content of hemp seed oil by GC-MS and LC-MS. The presence of linolieic acid (LA) and x-linoleic acid (LNA) were confirmed in the ratio of 3:1 LA: LNA. The presence of B-caryophyllene (740 mg $l^{-1}$), myrcene (160 mg $l^{-1}$), B-sitostrol (100-148 g $l^{-1}$) and trace amounts of methyl salicylate was observed in the oil.

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Deferne and Pate (1996) reported that hemp seed oil (cannabis) has largely focused on its content of psychoactive substances (cannabi noids) or its potential industrial use as a source of cellulose fibre. While the whole seed has long been used as a source of food, its potential health contribution has never gained much attention. Hemp seed shares with no other plant resource, both in high content of easily digestible complete protean and a rich endowment of oil providing a favourable ratio of the linoleic (C18:2w6) and linoleic (C18:3w3) essential fatly acids required for proper human nutrition, in addition to a significant contribution of gamma linoleic (C18:3w6) acid of potential therapeutic efficiency.

The aim of presented investigation was to search for oils from non conventional sources, because of increasing needs for oil both for human consumption and industrial applications.

## METHODS

### Operation of soxhlet extractor

300 ml of petroleum ether was poured into a round bottom flask. 10 g of the sample was placed in the thimble and was inserted in the centre of the extractor. The soxhlet was heated at 40-60°C. When the solvent was boiling the vapour rose through the vertical tube into the condenser at the top. The liquid condensate dripped into the filter paper thimble in the centre which contained the solid sample to be extracted. The extract seeped through the pores of the thimble and filled the siphon tube, where it flowed back down into the round bottom flask. This was allowed to continue for 30 min. It was then removed from tube, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted. Further extraction was carried out at 30 min intervals until the sample weight at further extraction and previous weight became equal. The experiment was repeated by placing 5 g of the sample into the thimble again. The weight of oil extracted was determined for each 30 min internal. At the end of the extraction, the resulting mixture containing the oil was heated to recover solvent from the oil.

### Determination of the percentage of seed oil extracted

30 g of the sample was placed in the thimble and about 150 ml of petroleum ether was poured into the round bottom flask. The apparatus was heated at 40-60% and allowed for 3 h of continuous extraction using soxhlet apparatus. The experiment was repeated for different weights of the sample, 35, 40 and 50 g. At the end the solvent was distilled and the percentage of oil extracted was determined.

### Determination of acid value

25 ml of diethyl ether and 25 ml of ethanol was mixed in a 250 ml beaker. The resulting mixture was added to 10 g of oil in a 25 ml conical flask and a few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1 m NaOH to the end point with consistent shaking for which a dark pink colour was observed and the volume of 0.1 m NaOH (*Vo*) was noted. Free fatty acid (FFA) was calculated as *Vo*/*Wo* = 82-100, where 100 ml of 0.1 m NaOH= 2.83 g of oleic acid, *Wo* = sample weight ; then acid value = FFA-2 (Laboratory Handbook, 1997).

### Determination of saponification value

The indicator method was used as specified by ISO 3657 (1988). 2 g of the sample was weighed into a conical flask; 25 ml of 0.1 N ethanoic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 60 min. A reflux condenser was placed on the flask containing the mixture and a few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5 M HCl to the end point until the pink colour of the inculcator just disappeared. The same procedure was used for other samples and a blank. The expression for saponification value (*SV*) is given by:

$$SV = 56.1\ \mathrm{N}\ (Vo\text{-}Vi)/\mathrm{m},$$

where: *Vo* – the volume of the solution used for the blank test, *Vi* – the volume of the solution used for determination, N – actual normality of HCl used, *m* – mass of the sample.

### Determination of iodine value

The method specified by ISO 3961 (1989) was used. 0.4 g of the sample was weighed into a conical flask and 20 ml of carbon tetra chloride was added to dissolve the oil. Then 25 ml of Dam reagent was added to the flask using a safety pipette influenced chamber. A stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 h and 30 min. At the end of this period, 20 ml of 10% aqueous potassium iodide and 125 ml of water were added using a measuring cylinder. The content was titrated with 0.1 M sodium-thiosulphate solution until the yellow colour almost disappeared. A few drops of 1% starch indicator were added and the titration continued by adding thiosulphate drop-wise until blue coloration disappeared after vigorous shaking. The some procedure was used for the blank test and for other samples. The iodine value (*IV*) is given by the expression:

$$IV = 12.69\mathrm{c}\ (V_1\text{-}V_2)\ \mathrm{m},$$

where: $c$ – concentration of sodium thiosulphate used, $V_1$ – volume of sodium thiosulphate used for the blank, $V_2$ – volume of sodium thiosoulphate used for determination, $m$ – mass of the sample.

**Determination of peroxide value**

To 1 g of the oil sample, 1 g of potassium iodide and 20 ml of solvent mixture (glacial acetic acid/chloroform, 2/1 by volume) were added and the mixture was boiled for one minute. The hot solution was poured into a flask containing 20 ml of 5% potassium iodide. A few drops of starch solution were added to the mixture and the latter was titrated with 0.025 N sodium thiosulphate and the peroxide value was determined as follows:

$$PV = \frac{S\ N\ 10^3}{W}.$$

where: $S$ – ml of $Na_2S_2O_3$, $N$ – normality of $Na_2S_2O_3$, $W$ – weight of oil sample (g).

**Determination of refractive index**

A refractometer was used in this determination. The sample were transferred into the glass slide of the refractometer. In order to carry out determination of the refractive index of oils, the prism box was open and a few drops of the oil were placed on the ground surface of the lower prism. It was then closed and the box flattened again, making sure that the oil did not flow away. The cross wires of the telescope was focused by rotating the eye piece and adjusting the mirror so as to get good illumination. By means of the lower knob, the prism box was turned slowly backwards and forwards until the field of view became coloured fringe. By means of the upper knob the compensator was rotated until the coloured fringe disappeared and the lighted image showed a sharp edge. The prism box was rotated until the sharp edge was in coincidence with the intersection of the cross-wires in the telescope. The index of refraction was then read off on the scale through the eye piece. The third decimal place in the refractive index could be read directly and the fourth was estimated with an accuracy of about ± 0.0002.

## RESULTS AND DISCUSSION

Some physical and chemical properties of oils/fats extracted from various seeds are shown in Table 1.

Apart from *Detarium microcarpum* with percentage oil content of 7.42%, the percentage oil yields from seeds is in the range of 26 to 42% which may be considered to be reasonable yield levels. The value of 34% for *Butyrospermum parkii* is in good agreement with the value of 32.80% reported by Kar and Mital (1999).

The acid values are lower than 1% in all cases of the oils investigated, being only 0.5% in the case of *Sterculia setegera*. Thus, in all cases there are corresponding low levels of free fatty acids in the oils, which also suggests low levels of hydrolytic and lipolytic activities in the oils.

The Saponification value (*SV*) is in the range of 199-261, with the exception of the value of 123.3 of *Detarium microcarpum*. The range for *SV* conforms to those reported for common oil (lab. 1960); for example, the *SV* for palm oil is 200, for groundnut is 193 and for coconut oil is 257. Thus the oils in Table 1 may be used for soap making.

The iodine values for all the seeds are in the range of 55 to 87g 100 $g^{-1}$, with the lowest value from *Detarium microcarpum* and the highest value from *Blighia sapida*. The iodine value of 61 for *Butyrospermum parkii* confirms the value of 60 reported in the literature (Kar *et al*., 1999) Apart from the oil from *Blighia sapida*, the iodine values for other seed oils suggest that they are highly saturated. The iodine value of 89.6 for *B. sapida* is compa- rable to the literature value of castor oils and olive oils, both of which are non-drying oils. A good drying oil should have iodine value of 180 and above. Thus, all the oils in Table 1 are not suitable as alky resins for paint formulation or use as varnishes; they may, however, found uses in conjunction with amino resins as finishes for certain appliances, and in this case, the oils can also act as plasticizers.

The peroxide values (*PV*) are in all cases high, but comparatively low in the case of *Sterculia setegera* and *Schorocarya birrea*. The high values of *PV* are indicative of high levels of oxidative rancidity of the oils and also suggest absence or low levels of antioxidant; certain antioxidants

**T a b l e 1.** Chosen properties of oils/fats extracted from various seeds

| Parameters | *Butyrospermum parkii* | *Lophira lanceolata* | *Sterculia setegera* | *Detarium microcarpum* | *Blighia sapida* | *Schorocarya birrea* |
|---|---|---|---|---|---|---|
| Oil content (%) | 34 | 40 | 33 | 7.42 | 26 | 42 |
| Acid value (mg NOH $g^{-1}$ of oil) | 0.463 | 0.034 | 0.504 | 0.204 | 0.340 | 0.250 |
| Saponification value (mg KOH $g^{-1}$ of oil) | 223 | 219 | 212.8 | 123.3 | 261 | 199.3 |
| Iodine value ($I_2$ g 100 $g^{-1}$ of oil) | 61.0 | 65.0 | 67.3 | 55.9 | 87.6 | 69.0 |
| Peroxide (ml $g^{-1}$ of oil ) | 77.5 | 95.0 | 35.0 | 150.0 | 135.0 | 25.0 |
| Refractive index (30-40°C) | 1.453 | 1.459 | 1.465 | 1.465 | 1.449 | 1.422 |

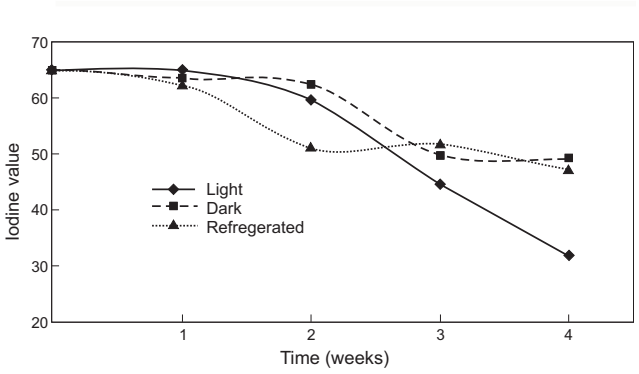


**Fig. 1.** Iodine value versus storage time of *Lophira lanceolata* oil.

**T a b l e 2.** Iodine and peroxide values of *Lophira lanceolata* during storage

| Method of storage | Iodine value ($I_2$ g 100 $g^{-1}$ of oil) | Peroxide value (ml $kg^{-1}$ of oil) |
|---|---|---|
| | Control | |
| Light | 65 | 90 |
| Dark | 65 | 90 |
| Refrigerated | 65 | 90 |
| | after 1 week | |
| Light (ambient) | 64.77 | 207.5 |
| Dark | 63.50 | 57.5 |
| Refrigerated | 62.23 | 72.5 |
| | after 2 weeks | |
| Light (ambient) | 59.42 | 890.0 |
| Dark | 62.23 | 75.0 |
| Refrigerated | 50.90 | 80.0 |
| | after 3 weeks | |
| Light (ambient) | 44.45 | 1110 |
| Dark | 49.53 | 80.0 |
| Refrigerated | 51.44 | 80.0 |
| | after 4 weeks | |
| Light (ambient) | 31.75 | 597.0 |
| Dark | 49.00 | 55.0 |
| Refrigerated | 46.99 | 100.0 |

may, however, be used to reduce rancidity such as propygadlate and butyl hydroxyl anisole. The refractive index values are in the range found for common oils.

Figure 1 is a plot of the iodine value versus time of storage of oil of *Lophira lanceolata*. In the first two weeks of storage, the *IV* dropped much more rapidly for the refrigerated oil, but at the end of the fourth week the *IV* was much lower for the oil exposed to light.

The overall results indicate considerable loss of instauration of oil by photo-catalysed reactions, suggesting that the oil is better stored in the dark (Table 2). Psychotropic organisms secreting oxidative enzymes can grow at low temperatures (even at 5°C), which may account for drop in *IV* in the cold.

The increase in the corresponding peroxide values (*PV*) is much greater for the oil which was stored in the light. This suggests a high level of photo-catalysed oxidation of the oil.

The values of *IV* and *PV* for both oils stored in the dark and refrigerated do not differ significantly.

## CONCLUSIONS

1. All the seeds examined in this work have been shown to contain oils in reasonable levels except *Detarium microcarpum* which contains about 7% oil (w/w).

2. It suggests that they contain mainly saturated fatty acids judging by their low iodine value which did not exceed 88 and are therefore not suitable as alkyd resins for paint formulation.

3. They may however be useful for other purposes such as soap judging by their high saponification values in the range of 123-261.

4. Storage properties for seed oils from *Lophira lanceolata* showed considerable changes in iodine and peroxide values under different conditions of storage.

5. The increase peroxide values of the oil with time under light was attributed to oxidative rancidity probably due to low levels of antioxidant in the oil.

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