International Journal of Biological, Life and Agricultural Sciences
ISSN: 2415-6612
Vol:8, No:11, 2014

Physicochemical Analysis of Soxhlet Extracted Oils from Selected Northern Nigerian Seeds

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Abstract**—**The aim of the present study is to investigate the potential use of the selected seed oils. The oil was extracted using Soxhlet apparatus and the physicochemical characteristics of the oil determined using standard methods. The following results were obtained for the physicochemical parameters analysed: for Egusi seed oil, Oil yield 53.20%, Saponification value 178.03±1.25 mgKOH/g, Iodine value 49.10±0.32 g I_2/100g, Acid value 4.30±0.86 mgKOH/g, and Peroxide value 5.80±0.27 meq/kg were obtained. For Pawpaw seed oil, Oil yield 40.10%, Saponification value 24.13±3.93 mgKOH/g, Iodine value 24.87±0.19 g I_2/100g, Acid value 9.46±0.40 mgKOH/g, and Peroxide value 3.12±1.22 meq/kg were obtained. For Sweet orange seed oil, Oil yield 43.10%, Saponification value 106.30±2.37 mgKOH/g, Iodine value 37.08±0.04 g I_2/100g, Acid value 7.59±0.77 mgKOH/g, and Peroxide value 2.21±0.46 meq/kg were obtained. From the obtained values of the determined parameters, the oils can be extracted from the three selected seeds in commercial quantities and that the egusi and sweet orange seed oils may be utilized in the industrial soap production.

Keywords**—***Carica papaya, Citrus sinensis*, iodine value, peroxide value, physicochemical.

I. Introduction

SEEDS have nutritive and calorific values, which make them necessary in diets. They are also good sources of edible oils and fats [1]. In addition, seed oils were found to be of nutritional, industrial and pharmaceutical importance [2]. Various vegetable oils are usually obtained from various sources. These include the common seed/vegetable oils such as soybean oil, cottonseed oil, peanuts oil and sunflower oil. They also include oils such as palm oil, palm kernel oil, coconut oil, castor oil, rapeseed oil, etc. Other less common but equally important vegetable oils include rice bran oil, tiger nut oil, patua oil, kome oil, niger seed oil, piririma oil and many others. The utilisation of oil in various applications is largely determined by the yield, composition, physical and chemical properties of the oil [3].

In Nigeria, the major sources of edible oils are peanut (*Arachis hypogoea*) and oil palm (*Eloesis guineensis*). These oils are used mainly as cooking oils, for the production of soap, margarine, and cosmetics [4]. However, with the increase in demand, which has led to increase in the importation of cooking oils, there is need to source for local oil-bearing-seeds which can be used in production of oils, both for consumption and industrial applications.

Melon (*Citrullus colocynthis* L.) is a widely cultivated and consumed oil seed crop in West Africa. The seeds popularly called 'egusi' contain about 53% oil, 28% protein and some other important mineral nutrients. They are consumed in 'egusi soup', melon ball snacks and 'ogiri' (a fermented condiment) [2]. The regions of its cultivation are Middle East, West Africa (Nigeria, Ghana, Togo, Benin) and other African countries for the food in the seeds and as a crop inter-planted with maize, cassava and yam. In Nigeria only, 'egusi' is cultivated over an area of 361,000 hectares with a production figure of 347,000 tonnes (as seeds) in 2002. It is used both as condiment and thickener in Nigerian local soup, and the industrial scale production of the oil yet to be utilized despite the huge potential. The crop had been in cultivation for at least 4000 years mainly for seeds schippers. The crop does well on a sandy free draining soil; it can also be planted as an intercrop with crops like maize, okro and also with cassava or yam because they are weed suppressor When planted it can be harvested between two and half to three months and with good management, there can be a seed yield of 350-400 kg per hectare [3].

The papaya or pawpaw is the fruit of the plant *Carica papaya*, the only species in the genus Carica of the plant family Caricaceae. It is native to the tropics of the Americas. The papaya is a large, tree-like plant, with a single stem growing from 5 to 10 m (16 to 33 ft) tall, with spirally arranged leaves confined to the top of the trunk. The leaves are large, 50–70 cm in diameter, deeply lobed, with seven lobes. The tree is usually un-branched, unless lopped [1]. The flowers appear on the axils of the leaves, maturing into large fruit. The fruit is ripe when it feels soft and its skin has attained amber to orange hue. Papaya is a rich source of iron, magnesium, potassium and calcium. The plant is also a good source of vitamins A, B and an excellent source of vitamin C (ascorbic acid). It also a good source of pantothenic acid, fiber and folate. The extracts of unripe *Carica papaya* contain a number of secondary metabolites or phytochemical constituents such as terpenoids, alkaloids, flavonoids, carbohydrates, glycosides, saponins, steroids, etc [4]

The sweet oranges (*Citrus sinensis*) is the most widely grown of the Citrus trees and the fruits are either eaten fresh or made into endocarp which contain the thin-walled juice vesicle. Surrounding the endocarp is the peel, which comprises 20 to 50% of the weight of the fruits and consists of the flavedo and alebedo [2].

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The present work is therefore aimed at the extraction and physicochemical analysis of egusi (*Citrullus colocynthis* L.), sweet orange (*Citrus sinensis* L.) and pawpaw (*Carica papaya* L.) seed oils in order to justify their industrial applications.

II. Materials and Method

A. Sample Collection and Identification

The sweet orange (*Citrus sinensis*) seeds were collected from Bgokpo, Benue state, Nigeria in March, 2013, while pawpaw (*Carica papaya*) fruit and egusi (*Citrullus colocynthis*) seeds were bought from Aliero central market, Aliero, Kebbi State, Nigeria. The seeds were identified by a plant Scientist in the Department of Biological Science, Kebbi State University of Science And Technology, Aliero, Kebbi State. Nigeria. The sweet orange and egusi seeds were selected and damaged ones were discarded. The seeds were cleaned, de-shelled and well dried and ground using pestle and mortar prior to extraction.

While the pawpaw fruit was cut in to four and the seeds were scoop out with a spoon, washed with distilled water to remove all the fruit flesh, allowed to dry and the epicarp was removed and dried under the sun for five days. The seeds were then pulverized with the aid of a blending machine and store in a plastic container for further use. All reagents were of analytical grade unless otherwise stated. The physicochemical analyses were carried out in triplicates unless otherwise stated.

B. Oil Extraction

The oil content of *Jatropha curcas* seeds were obtained by complete extraction using the Soxhlet extractor (Konte, USA). Extraction of oils from seeds was carried out by Soxhlet extraction method. The 70 g of each powdered seed sample was put into a porous thimble and placed in a Soxhlet extractor, using 150 cm^3 of n-hexane (with boiling point of 40-60^oC) as extracting solvent for 6 hours. The oil was obtained after the solvent was removed under reduced temperature and pressure and refluxing at 70^oC to remove the excess solvent from the extracted oil. The oil was then stored in freezer at –2^oC for subsequent physicochemical analyses [5].

C. Oil Yield Determination (%)

The oil gotten after the extraction was transferred into a measuring cylinder which was placed over water bath for 30 min at 70^oC to ensure complete evaporation of solvent and volume of the oil was recorded and expressed as oil content (%) [6].

The oil content was calculated as follows:

$$\text{Oil content} = \frac{weight\ of\ the\ oil}{\text{weight of the sample}} \times 100\%$$

D. Physicochemical Analysis

Physicochemical analysis of the oils was conducted using the standard methods reported [7]–[9]. The parameters analyzed were the iodine value (IV), saponification value, acid value and peroxide value as follows:

E. Saponification Value

About 2g of the oil sample was added to a flask with 30ml of ethanolic KOH and was then attached to a condenser for 30 minutes to ensure that the sample is fully dissolved. After sample has cooled 1ml of phenolphthalein was added and titrated with 0.5 M HCl until a pink colour appeared, indicated the end point.

The expression for saponification value (S.V.) is given by:

$$\text{Saponification Value} = \frac{56.1N(VO-V1)}{\text{M}}$$

where V0 = the volume of the solution used for blank test;
VI = the volume of the solution used for determination;
N = actual normality of the HCl used;
M = Mass of the sample.

F. Iodine Value

0.4g of the sample was weighed into a conical flask and 20ml of carbon tetrachloride was added to dissolve the oil. Then 25ml of Dam's reagent was added to the flask using a safety pipette in fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours 30 minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 125ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium-thiosulphate solutions until the yellow colour almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples.

The iodine value (I.V) is given by the expression:

$$\text{Iodine Value} = \frac{12.69C\ (V1-V2)}{\text{M}}$$

where C = Concentration of sodium thiosulphate used;
V1 = Volume of sodium thiosulphate used for blank;
V2 = Volume of sodium thiosulphate used for determination,
M = Mass of the sample.

G. Acid Value

100 ml of neutral ethyl alcohol was heated with 10g of oil or fat sample in a 250ml beaker until the mixture began to boil. The heating was stopped and the solution was titrated with N/10 KOH solution, using two drops of phenolphthalein as indicator with consistent shaking for which a permanent pink colour was obtained at the end point.

The Acid value was calculated using the expression

$$\text{A.V} = 0.56 \text{ x No. of ml. N/10 KOH used.}$$

H. Peroxide Value

Exactly 1.0g of KI and 20ml of solvent mixture (glacial acetic acid: chloroform, 2:1 v/v) were added to 1.0g of the oil sample and the mixture was boiled for one minute. The hot solution was poured into a flask containing 20ml of 5% KIO_3 solution. Few drops of starch solution were added to the

mixture and the latter was titrated with 0.025M $Na_2S_2O_3$ solution.

III. Results

TABLE I
PHYSICOCHEMICAL CHARACTERISTICS OF THE SELECTED SEED OILS*

Selected seed oil	Egusi	Pawpaw	Sweet orange
oil yield (% wt/wt)	53.20	40.10	43.10
Saponification value (mgKOH/g)	178.01±1.25	24.13±3.93	106.30±2.37
Iodine value (gI2/100g)	49.10±0.32	24.87±0.19	37.08±0.04
Acid value (mgKOH/g)	4.30±0.86	9.46±0.40	7.59±0.77
Peroxide value (meq/kg)	5.80±0.27	3.12±1.22	2.21±0.46

*Values are mean and standard deviation (n=3).

The following results were obtained for the physicochemical parameters analysed: for Egusi seed oil, Oil yield 53.20%, Saponification value 178.03±1.25 mgKOH/g, Iodine value 49.10±0.32 g I_2/100g, Acid value 4.30±0.86 mgKOH/g, and Peroxide value 5.80±0.27 meq/kg were obtained. For Pawpaw seed oil, Oil yield 40.10%, Saponification value 24.13±3.93 mgKOH/g, Iodine value 24.87±0.19 g I_2/100g, Acid value 9.46±0.40 mgKOH/g, and Peroxide value 3.12±1.22 meq/kg were obtained. For Sweet orange seed oil, Oil yield 43.10%, Saponification value 106.30±2.37 mgKOH/g, Iodine value 37.08±0.04 g I_2/100g, Acid value 7.59±0.77 mgKOH/g, and Peroxide value 2.21±0.46 meq/kg were obtained.

IV. Discussion

The result for the physicochemical analysis of the three soxhlet extracted seed oils is presented on Table I. The percentage oil yield of egusi, pawpaw and sweet orange seed oils was 53.20%, 40.10% and 43.10% (wt/wt) respectively. The values are closely similar to that of percentage oil yield of *M. peregrina* seed oil (49.80%) reported from Saudi Arabia [10]. However, the oil contents of the seed oils in the present study are higher than that of some conventional oil seed crops: cotton (15.0 – 24.0%), soybean (17.0 – 21.0%), safflower (25.0 – 40.0%) and mustard (24.0-40.0%) [11]. Such variation in oil content across species and locations might be attributed to the environmental and geological conditions of varied regions [12]. With this relative high percentage oil yields in the present study, the processing of the oils for industrial, primarily soap production, as well as edible purposes would be viable.

The Saponification value for egusi, pawpaw and sweet orange seed oils was found to be 178.01mgKOH/g, 24.13mgKOH/g and 106.30mgKOH/g respectively. The saponification values for egusi and sweet orange seed oils (178.01% and 106.30%) are lower than the saponification value of 213mgKOH/g in neem seed oil [13] and 253mgKOH/g in coconut oil [14]. However, the values obtained were higher than that of beeswax (93 mgKOH/g), which is commonly used in soap making [15]. This indicates that egusi and sweet orange seed oils could be used in soap making since their saponification values fall within the range of these oils. Higher saponification value justifies the usage of fat or oil for soap production. However, pawpaw seed oil, with lower saponification value (24.13%) may not be very good in soap production.

The iodine value for egusi, pawpaw and sweet orange obtained was 49.10gI_2/100g, 24.87gI_2/100g and 37.08gI_2/100g respectively. The iodine values obtained for the three seed oils are lower than that of the other common seed oils such as safflower and soyabean oil with iodine values of 145 gI_2/100g and 132 gI_2/100g [16] respectively. The iodine values obtained for the three seed oils are all less than 100gI_2/100g which qualifies them as non-drying oils useful in soap manufacture.

The acid value for egusi, pawpaw and sweet orange seed oils was found to be 4.30mgKOH/g, 9.46mgKOH/g and 7.59mgKOH/g respectively. The values are lower than that of olive oil 17mgKOH/g but higher than 1.20mgKOH/g reported for Jatropha seed oil [17] which signifies a maximum purity and suitability of the oils for soap production.

The peroxide value for egusi, pawpaw and sweet orange seed oils was found to be 5.80meq/kg, 3.12meq/kg and 2.21meq/Kg respectively. These values are relatively low compared with the peroxide values of other oils of wild plants [18]. According to reference [19], high peroxide value is associated with high rancidity rate. Thus, with this fact, the low peroxide values obtained from these oils is simply an indication the oils are less liable to rancidity at room temperature.

V. Conclusion

From the result obtained, the three seeds are good source of oil. In addition, egusi and sweet orange seeds could be utilised in the industrial soap production.

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