International Research Journal of Pure & Applied Chemistry

21(8): 12-17, 2020; Article no.IRJPAC.47365
ISSN: 2231-3443, NLM ID: 101647669

Extraction and Characterization of Oil from Sorrel (Yakuwa Seed) *Hibiscus sabdariffa*

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Authors' contributions

This work was carried out in collaboration among all authors. Author JI designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author GC managed the analyses of the study. Author MN effected corrections on the revised manuscript. Author VC managed the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/IRJPAC/2020/v21i830184
Editor(s):
(1) Dr. Richard Sawadogo, Group Cell Death & Natural Compounds, Laboratoire de Biologie Moléculaire et -Cellulaire du Cancer Hôpital Kirchberg, Luxembourg.
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Reviewers:
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(2) Uchendu, Mbah Okuwa, Michael Okpara University of Agriculture, Nigeria.
(3) S. Murugesan, University of Madras, India.
Complete Peer review History: http://www.sdiarticle4.com/review-history/47365

Original Research Article

Received 24 March 2020
Accepted 31 May 2020
Published 03 June 2020

ABSTRACT

Hibiscus sabdariffa (yakwua seed) is one of economic value in tropical and subtropical regions around the world due to diverse industrial, medical and food uses. In this work, oil was extracted from the seed of *Hibiscus sabdariffa* using normal hexane as extracting solvent. The extraction was carried out at temperature of 70°C at 3-4 hours to ensure optimum oil yield. At a range of 3-4 hours extraction time and at a temperature of 70°C, the average oil yield obtained was 17.8%. The physical state of oil extracted is liquid/yellow greenish in colour with characteristic pleasant smell, relative density of (0.92 g/m^3) , refractive index of (1.46),specific gravity of (0.91), and viscosity of (43.67mm^2/sec) and the chemical parameters were also determine, the saponification value of (166.20 mgKOH/kg), acid value of (1.40 mgKOH/g), free fatty acid is (0.55 mgKOH/g), peroxide value of (6.90 mg/kg), iodine value of (80.03 g I_2/100 g), and ester value of (164.79 mgKOH/g),are comparable with other oils extracted from other sources suitable for consumption. This reveals that

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the oil extracted from *Hibiscus sabdariffa* can be used both domestically and industrially, as the seed is already used by michika community for production of traditional maggi called dadawa in Hausa.

Keywords: Sorrel seed; solvent extraction; physico-chemical parameter and oil yield.

1. INTRODUCTION

Hibiscus sabdariffa locally known as “Yakuwa” is a well-adapted crop in the semi-arid zone of West Africa including Nigeria and it is generally planted as a border crop. The leaves are used as vegetable and the floral parts in the preparation of “Sobo”, a local drink. The stem provides fibre and the seeds are eaten by scavenging poultry [1]. The seed of *Hibiscus sabdariffa* is said to have high protein value and it is on account of its protein content that it is well priced for human consumption in French West Africa (Mali, Guinea, Burkina Faso, Cote D’ Voire). However, in Nigeria larger quantities of the seeds of this crop are wasted on the farm annually and just enough being collected and stored for planting.

However new low-cost oil seed crops are needed to produce inexpensive oils suitable for food, pharmaceuticals and industrial applications. One of the possible alternative crops is *Hibiscus sabdariffa,* also known as sorrel or Roselle because some parts of the plant such as leaves, stem, calyxes, had been used hence the seed could be a source of vegetable oil. It is an herb belonging to the Malvaceae family, which is grown in Nigeria, India and West Indies and to some extent in tropical America [2].

Vegetable oils and fats are an important renewable source of raw materials in the chemical industries as they are used in the manufacture of various industrial products. Thus, there is a need to expand the existing supplies of vegetable oils and fats for both domestic and industrial use. It is especially important to pay more attention to the underutilized tropical seed oils in Nigeria. These works review some areas where vegetable oils can be modified or used as industrial raw material [3].

Generally, there has been a surge in research interest aimed at harnessing various seed oils that are used in chemical industries. The need is as a result of the ever increasing world demand for oil and the challenge to expand the existing supplies of oils for human consumption and industrial utilization. It is even more compelling now when renewable resources are preferred to petroleum based raw materials in the chemical industries; furthermore they are more environmentally friendly [4]. The use of vegetable fats and oils for both edible and industrial uses dates back to antiquity. They are obtained mainly from the seeds of a plant. Traditionally, vegetable oils are used for cooking, medicinal uses, soap and polish production, as insecticide, for lighting etc. Today, vegetable oil has found a wider application in the chemical industries leading to a higher demand for these oils.

However, in Nigeria, there abound a vast number of seeds produces by the available tropical plants that are potential sources of industrial oil which if well harnessed can enhance the supply of vegetable oil as raw materials. The popular seed oil such as palm oil, groundnut oil, soya bean oil, palm kernel oil has been widely used for both edible and industrial applications. The dual usage of some of these oils has caused a kind of scarcity and higher demand leading to relatively higher price of crops like soya bean [4] whereas, a lot of the locally available seeds that are potential sources of industrial oil are still largely under-utilized or neglected. Sorrel seeds, which until now have not have any commercial applications, are a source of a vegetable oil that is low in cholesterol and rich in other phytosterols and tocopherols, particularly β-sitosterol and 8-tocopherol. The overall characteristics of roselle seed oil allow for important applications and represent added value for its cultivation [5].

The Oil seed crops are vital sources of oils of nutritional, pharmaceutical and industrial importance. The characteristics of oils from different source depend mainly on their compositions and no oil from a single source can be suitable for all purpose [6].

2. MATERIALS AND METHODS

2.1 Sample Collection

Sorrel (yakuwa seed) *Hibiscus sabdariffa* was purchased from Jimeta modern market yola, Adamawa state of Nigeria. The seed was washed thoroughly (4 to 5 times) then was sun dried for five days. Finally the cleaned seed was milled into powder.

2.2 Solvent Extraction (using n-hexane)

Soxhlet apparatus and n-hexane as solvent was used in this work. 400 g of sample was weighed out using a weighing balance. The apparatus was charged with a known weight oil seed powder in a muslin cloth placed in the thimble of soxhlet extractor. 200 ml of the solvent was measureed into a round bottom flask containing known volume of the sample was fixed to the end of the apparatus and condenser was tight fixed at the bottom end of the extractor.

The whole set up was heat up in the heating mantle at the temperature of 70°C. The extraction was carrying out for 3-4 hours to ensure optimum oil yield. The quality of the oil extract will deter- mine by using gravimetric analysis. The oil yield was evaluated as the ratio of the weight of the extracted oil to the weight of the sorrel oilseed powder sample. The oil was stored appropriately for further work.

2.2.1 Percentage yield determination

$$\% \text{ Yield} = \frac{(W_1 - W_2)}{W_1} \times 100$$

W_1 = Initial weight of sample (g)
W_2 = Final weight of sample (g)

2.2.2 Free fatty acids (FFA)

25 ml of diethyl ether was mixed with 25 ml alcohol and 1 ml phenolphthalein solution (1%) and carefully neutralized with 0.1 M NaOH. 2 g of the oil is was dissolved in the mixed neutral solvent and titrated with aqueous 0.1 M NaoH which was shaken constantly until a pink colour which persisted for 15 seconds was obtained [7].

2.2.3 Iodine value

5 ml of the prepared Dam's solution was added to 2 g of oil sample in a conical flask and mixed thoroughly. The mixture was allowed to stand in a dark cupboard for 5 minutes to allow for hallogenation of double bond. 5 ml of 10% KI solution was then added followed 20 ml of distilled water. The KI reduced the Dam's iodine to free bromine. The liberated bromine was titrated with 0.1m solution thiosulphate using starch indicator as well using chloroform instead of the oil sample [7].

2.2.4 Peroxide Value

1 g of oil was weighed out into a clean dry boiling tube and while liquid, 1 g of powdered potassium iodide and 20 ml of solvent mixture (2 vol glacial acetic acid + 1 vol chloroform). The tube was placed in water at 100°C for 30 seconds. The contents was quickly poured into a flask containing 20 ml of potassium iodide solution (5%), the tube was washed out twice with 25 ml water and titrated with 0.002 sodium thiosulphate solution using starch. A blank was performed at the same time [7].

2.2.5 Saponification Value

A portion (2 g) of the sample was measured into a flask and 25 ml of alcoholic potassium hydroxide solution was added to it. A reflux condenser was attached to the flask and the solution heated for one hour with occasional shaking. 3 drops of phenolphthalein solution was added and titrated with 0.5 m HCL. A blank was carried out with water and recorded as b.(bml). [7]

2.2.6 Acid value

$$= \frac{\text{Titre (ml)} \times 5.61}{\text{Weight (g) of sample used}}$$

2.2.7 Ester value

Ester value represents the number of milligrams of potassium Hydroxide required to saponify the ester present in 1 g of the oil. It is obtained as the difference between the saponification value and the acid value [7].

2.2.8 Moisture content

A portion (2 g) of the oil was measured into a preweighed porcelain allowed to cool in a desiccators and the weight measured. Moisture content was placed in a laboratory oven at 105°C for 2 hours. [7]. The sample was removed and calculated thus;

$$\text{Moisture contentment} = (M_1 - M_2) \times 100$$

Where M_1 = initial weight of sample (g)

M_2 = final weight of sample (g)

2.2.9 Refractive index determination

The refractive indices of the oil extracts were determined with Abbe refractometer. Average value of three readings was taken for each sample [7].

2.2.10 Density determination

Density was measured by taking the weight of a known volume of oil extracts inside a density bottle using metler (Model, AT400) weighing balance [7]. Average value of three readings was taken for each sample.

2.2.11 Viscosity determination

Viscosity measurements were carried out using 50 ml graduated glass macro-syringe. The apparatus were standardized with 20% (W/V) sucrose solution with the viscosity 2.0 mPa.s at 30°C. The viscosity of the oil extracts were evaluated in relation to that of the standard sucrose solution at 30°C. Three different readings were taken for each sample and the average value calculated.

3. RESULTS AND DISCUSSION

Physiochemical characteristics of *Hibiscus sabdariffa* sorrel oil is discussed in Table 1.

3.1 Results

Results of the various experiments are shown in Table 1 below.

3.2 Discussion

Result of saponification value as can be seen in the table above is 166.20 mg/KOH/g. the result was compared to cashew nut oil 137.00 mg/KOH/g [8]. According to "International standard for edible oil" (FAO/WHO, 2009) a saponification value of 181.4 mg/KOH/g indicates a high proportion of fatty acid of low molecular weight [9]. This shows that the oil may have a potential for use in making soap and cosmetics industry and for thermal stabilization of poly vinyl chloride (PVC); these properties) makes them useful as sources of essential fatty acids required in the body [10].

Peroxide value is the index of rancidity, thus the higher peroxide value of oil indicates a poor resistances of the oil to per oxidation during storage [11] The peroxide values of *Hibiscus sabdariffa* seed oil is 6.90 meq/KOH/g which is below the maximum acceptable value of 10 meq/KOH/ g set by the Codex Alimentarius commission for such oils as groundnut seed oil [12] Peroxide value is an indication of level of deterioration of the oil. The low peroxide value further confirms the stability of the oil. The fresh oil has values less than 10 meq/KOH/kg. Higher values between 20 and 40 results to rancid taste [13]. The low acid and peroxide values are indicators of the ability of the oil to lypolitic hydrolysis and oxidative deterioration [10].

Acid value is a direct measure of the percentage content of free fatty acids in a given amount of oil. Acids value depends on the degree of rancidity which is used as an index of freshness [14].The acid value of *Hibiscus sabdariffa* seed oil is 1.40 mg/KOH/g .The value is compared to that of butter oil, 1.79 mg/KOH/g [15] and cashew nut 10.7 mg/KOH/g [8] which are higher than it. The low value of *Hibiscus sabdariffa* seed oil (1.403) suggested that the oil may be of advantage for paint making and also edible [16].

Table 1. Results obtained

Physicochemical parameters of *Hibiscus sabdariffa* sorrel oil	**Value (X)**	**Standard deviation** $s = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(x_i - \overline{x})^2}$	**International standard for edible oil (FAO/WHO, 2009)**
Saponification value	166.20(mgKOH/g)	0.17	181.4 ± 2.60
Peroxide value	6.90 (meq/kg)	0.1	10
Iodine value	80.03 ($gI_{2/100g}$)	0.11	80 – 106
Free fatty acid	0.55 (%)	0.01	5.78 – 7.28
Acid value	1.40 (mgKOH/g)	0.017	4
Ester value	164.79(mgKOH/g)	0.017	-
Relative density	0.92 g/cm^3	0.026	0.9 – 1.16
Refractive index	1.46	0.01	-
Viscosity	43.67s/cm^2	0.046	-
Percentage yield	17.80%	0.017	38 – 40
Moisture content	0.07%	0.01	-
Specific gravity	0.91	0.02	-

Iodine value obtained for *Hibiscus sabdariffa* seed oil is 80.03 I_2/100 g. The value is compared with palm oil and cottonseed oils which is 105-115, according to "International standard for edible oil" (FAO/WHO, 2009) [17]. Cottonseed oils have the iodine value of 104 I_2/100 g higher than that of *Hibiscus sabdariffa seed* oil. While palm oil has an iodine value of 60 I_2/100 g which is lower than that of *Hibiscus sabdariffa* seed oil. The low iodine value of the oil may contribute to its greater oxidative stability during storage. Oxidative and chemical changes in oils during storage are characterized by a decrease in the total unsaturation of the oil and increase FFA content [18].

The FFA value of *Hibiscus sabdariffa* seed oil is 0.55 KOH/g. In refined vegetable oils, the lower the FFA more acceptable the oil to the human palate. The low FFA content of sorrel seed oil obtained indicates that this oil is good resistances to hydrolysis [19].

Ester value represents the number of milligrams of potassium hydroxide requires to saponify the ester presents in 1 g of oil. It is obtained from the difference between the saponification and acid value. Ester value obtained from *Hibiscus sabdariffa* seed oil is 164.79 KOH/g. The value obtained in this study is in line with those of avocado pear 172.8 mgKOH/g and castor seed oil 174.09 mgKOH/g [16].

Physical properties of oil extracted from *Hibiscus sabdariffa* sorrel seed. The oil extracted is yellow greenish color, odour is pleasant smelling. It has a specific gravity of 0.91 which shows that it is less than water. This value agrees with those obtained by [20] and Tint and mya [21] from *Hibiscus sabdariffa* seed oil. The refractive index shows level of optical clarity of the crude oil sample relative to water. A refractive index obtained is 1.46 which is in agreement with the value of 1.46 obtained from African star apple seed oil [14] which shows that the is not thick as most drying oils whose refractive indices fall between 1.475 and 1.485 [8]. The oil yield from *Hibiscus sabdariffa* seed oil is 17.8%. This show that a fraction of the seed are made of oil. The result of this study was compared with that of cash nut oil [8] 49.1% and [16] Avocado pear oil 52.4%. This indicates that the seed may not be a good source of abundant oil. However, genetically modified breeds may be developed which produce seeds with more oil yield. The low moisture content obtained will encourage the storage stability of the *Hibiscus sabdariffa* sorrel seed. The value is 0.07%. The oil showed a similar trend for free fatty acid content. This implies that the higher the value of the moisture content of the oil, the greater the value used for food texturing, baking and frying and industrial manufacture and the oil paint [22]. The viscosity, which is a measure of the resistance of oil to shear, is 43.8mm^2/sec. The viscosities of vegetable oil shows that different oil has different viscosity. Increase in viscosity is due to increase molecular weight and cross-linking density will decrease as a result of low molecular weight and low cross-linking.

4. SUMMARY AND CONCLUSION

This result shows that *Hibiscus sabdariffa* sorrel (yakwua seed) may be viable sources of oil going by its oil yield. Furthermore, the physicochemical properties of this oil extracted shows that it could have many domestic applications. Importantly, *Hibiscus sabdariffa* sorrel (yakwua seed) are one fruit of economic value in tropical and subtropical regions around the world due to diverse industrial, medical and food uses.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history:
The peer review history for this paper can be accessed here:
http://www.sdiarticle4.com/review-history/47365