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Extraction and physicochemical analysis of *Citrus sinesis* seed oil (sweet orange)

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ABSTRACT

The aim of this study was to extract oil from sweet orange seeds and determine potential applications by investigating the physicochemical parameters of the oil. The seeds of Citrus sinesis were collected, washed and prepared for uses such as decocting, drying and pulverizing. The seed oil was extracted using soxhlet apparatus with diethyl ether and petroleum spirit as solvents. The extract was centrifuged to remove particulates and the percentage yield of oil extraction with diethyl ether was (56.3%) and (56.0%) with petroleum spirit. Physicochemical analysis of the seed oil was conducted using standard procedures and the following results were obtained; relative density (0.88 12g/cm^3), moisture content (4.0%), refractive index at 25^{o}C (1.457), ash content (5.0%), organic matter (95.0%), iodine value (54.19 g/100g), saponification value (190.32mgKOH/g) and peroxide value (5.8meq/kg). The use of Citrus sinesis seed oil in chemical industries for the production of soap, perfumes, candles, lubricant, flavoring and insecticides has been justified.

Keywords: *Citrus sinesis*, extraction, physicochemical, parameters

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INTRODUCTION

Citrus sinesis commonly known as sweet orange belongs to the *Rutaceae* family. Sweet orange originated from south-east Asia and it is the most widely used species of citrus fruits [1]. Sweet orange tree is a medium size plant often grown to a height of 6.0m – l00m. The broad, glossy, ever green leaves are medium sized and ovate with crenulated margins and about 4-12cm in diameter; the petioles (leaf stalks) have narrow rings [2], [3]. The peel is 0.5cm thick and tightly adheres to the segments [4]. Upon ripening, it changes into an oranges color but often remains green or pale yellow in tropics. The pulp is very juicy and slightly acidic. The central line is solid and it may contain no seeds or many seeds. Sweet orange seeds occur with the berry and are embedded in juice sacs of the loculus and very close to the central axis [3], [4].

The sweet orange (*Citrus sinesis*) is a fruit-bearing plant that provides edible fruits through the whole tropical and subtropical lands [4]. Oranges are citrus fruits consisting in a pericarp or the peel, a thin white membrane and a pulp including some pits. The world production of oranges is stated 62 709 636 tons.

The aim of this research was to extract oil from sweet orange seeds and carry out physicochemical analysis in order to determine whether or not the oil can compete with other vegetable oils like ground nut, soya beans etc. and to find out if it can serve as a source of cheap vegetable oil for the growing population of this country.

MATERIALS AND METHODS

Seed material

The sample (sweet orange seeds) was collected from Muda lawan and Wunti market areas of Bauchi state, Nigeria. The seeds were identified by Mal. Ibrahim Shuaibu, Department of Forestry, College of Agriculture, Bauchi state. The seeds were washed, dried and pulverized prior to extraction.

Oil extraction

The extraction of 250 g of powdered seeds of *Citrus sinesis* was conducted in a soxhlet apparatus using diethyl ether and petroleum spirit respectively, at 40-60^{o}C for 6hrs. Solvent was removed from the oil under reduced temperature, pressure and refluxing at 70^{o}C. The obtained oil was stored in a freezer for subsequent analysis.

***Oil yield determination* (%)**

Solvent was freed from the oil obtained after extraction was placed over a water bath at 70^{o}C for 30mins and the volume of oil was recorded and expressed as oil content (%) as calculated below;

$$\text{Oil content} = \frac{\textit{Weight of oil}}{\textit{Weight of sample}} \times 100$$

Physicochemical parameters

Moisture content determination

A crucible was washed and dried in the oven, after cooling in the dessicator and weighed (W_1). 2.0g of the sample was carefully weighed in the crucible and the weight was taken as (W_2). The crucible containing the sample was then placed in an oven at temperature of 105°C for 1 hour. It was cooled and weighed. The crucible was then introduced into the oven again and process of cooling and weighing continued at intervals until a constant weight was obtained (W_3). Percentage moisture content was calculated below:

$$\%\ moisture\ content = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

Ash content determination

A porcelain crucible was washed and dried in an oven then cooled in a desiccator and weighed. 2.0g of the sample was carefully weighed in the crucible containing the sample and was heated gently on a Bunsen burner until the smoke ceased. It was then transferred to a muffle furnace and heated at a temperature of 550°C - 570°C for 2 hours to burn all organic matter. The crucible was taken out of the muffle furnace after a white was observed and placed in a dessicator to cool and weighed.

$$\%\ of\ Ash\ content = \frac{Weight\ of\ Ash}{Weight\ of\ sample} \times 100$$

Relative density determination

A specific density bottle was washed, dried and weighed (W_1). It was filled with distilled water and weighed (W_2). The water was poured off and the bottle was dried to it previous constant weight and then filled with the oil sample and weighed (W_3).

$$Relative\ density = \frac{W_3 - W_1}{W_2 - W_1}$$

Refractive index principle determination

The prism of the instruments was cleaned thoroughly and two drops of the oil sample was placed on the prism. The temperature of the oil was allowed to equilibrate with that of the thermo stated fluid and reading of the thermometer was noted.

The knobs of the instrument were set and the fluid was demarcated by a sharp line dividing the field of view in to two equal halves and when line conceded with the spot marks “X” in the field of the view. The reading was taken at this point that is at normal temperature of 25°C but 40 — 60°c is for high melting fats [5], [6].

Refractive index (Ri) = R + K ($T_1 - T_2$)

Where
Ri = Refractive index reduced to standard temperature (25°C).
R = Reading obtained at temperature T^3
T_1 = Standard temperature 25°C
T_2 = Temperature at which reading was taken.
K = Substituting factor (Constant) 0.000385 for Oils 0.000365

Iodine value determination
Wij's solution was prepared; 8.0g of iodine monochloride was dissolved in 200cm^3 glacial acetic acid. 9.0g of Iodine crystals was dissolved in 300cm^3 of carbon tetrachloride (CCl_4) the two solutions were then mixed and made up to the mark with glacial acetic acid.

10g of Oil sample was weighed into a dry 250cm^3 conical flask and 10cm^3 of carbon tetrachloride was added followed by 20cm^3 of the prepared Wij's solution. The flask was stopped and kept in the dark cup board for 30 minutes at room temperature; 15ml of 10%of potassium iodide solution and 100cm^3 of distilled water was added. This was titrated against 0.2ml Sodium thiosulfate solution using starch as an indicator. A blank titration was also conducted under the same conditions without the sample.

$$Iodine\ value = \frac{(A-B) \times (N\ of\ N_{a2}S_20_35H_20) \times 12.69}{Q}$$

Where;
A = volume of 0.1 in $Na_2S_2O_3.5H_2O$ solution used for the black titration.
B = Volume of 0.lm of $Na_2S_2O_3$ $5H_2O$ solution used for the Sample titration.
Q= Weight in gram of the oil sample
12.69 = Conversion factor.
N = Normality

Saponification value determination
The method described by Pearson [6] was employed. 2.0g of the oil sample was weighed in to 200ml conical flask and 25ml of 0.5M of ethanolic potassium hydroxide solution was added. The flask was configured to a condensing set-up and heated on a water-bath for 1 hour with frequent shaking and the content was allowed to cool. The solution was then titrated with warm O.5M Hydrochloric acid using 1% Phenolphthalein indicator. Equivalent titration was performed for the blank and generated values were employed for computation according to the following relation;

$$Saponification\ value = \frac{A-B}{Q} \times 28.05$$

Where
A = Volume of 0.5M of Hydrochloric acid used in the blank titration.
B = Volume of 0.5M of Hydrochloric acid used in the sample titration.
Q = Weight in grams of the oil sample.
2805 = Conversion Factor

Peroxide value determination
Exactly, 2.0g of the oil sample was transferred into 250cm^3 flask and 1g of powdered potassium iodide (KI) and a solvent mixture (2:1 of glacial acetic and trichloromethane) were then added. The solution was then placed on a water bath for a few minutes for complete dissolution. 20cm^3 of 50% potassium iodide were introduced and the sample titrated with 0.1M $Na_2S_2O_3$. The indicator was a regular starch solution. Blank experiment was similarly performed.

$$\text{Peroxide value} = \frac{(R \times B) \times \text{morality of } Na_2S_2O_3.}{W}$$

Where, R and B stand for oil and blank samples in term of titre values, respectively.

RESULTS AND DISCUSSION

The result of physiochemical analysis of *Citrus sinesis* seed oil is presented in Table 1. The percentage yield of the seed oil was 56.0 and 56.3% for petroleum spirit and diethyl ether respectively. The values are higher compared to that of percentage oil yield of M. peregrine seed oil (49.80%) reported from Saudi Arabia [7] and that of egusi, pawpaw and sweet orange seed oils was 53.20%, 40.10% and 43.10% (wt/wt) reported from northern Nigeria respectively [8]. The variation in content of the oil might be attributable to geographic and environmental conditions which solely dependent on various region [9]. This relative high percentage oil yield in the study, processing of industries especially soap production is viable.

The saponification value for the *Citrus sinesis* seed oil was found to be 190.32mgKOH/g. The saponification values are higher than the saponification values for egusi, pawpaw and sweet orange seed oils 178.01mgKOH/g, 24.13mgKOH/g and 106.30mgKOH/g respectively [8] and are lower than the saponification value of 213mgKOH/g in neem seed oil [10] and 253mgKOH/g in coconut oil [11]. This is an indication that sweet orange seed oil could be used in soap making. Hence, higher saponification value justifies the usage of fat or oil for soap production.

The iodine value of *Citrus sinesis* (54.l9g/100g) is higher than the value (28.6 g/100g) of *citrullus lanatus* seed oil, Hausa melon seed (38.50 g/100g and 44.4 g/100g for cashew nut oil [12], egusi, pawpaw and sweet orange obtained was 49.10gI2/100g, 24.87gI2/100g and 37.08gI2/100g respectively [8]. The value obtained for *Citrus sinesis* seed oil qualifies it to be non-drying oil because drying oils have an iodine value above 100 g/100g [13]. This nondrying quality means the oil can be used effectively in the paint industry [13].

The peroxide value for sweet orange was found to be 5.8meq/kg. The value was similar to that of egusi 5.80meq/kg and higher than the peroxide value for pawpaw and sweet orange seed oils 3.12meq/kg and 2.21meq/Kg respectively [8], [14]. High peroxide value is associated with high rancidity rate [15] [16]. Thus, the values between 20 and 40meq/kg result in rancid taste. The low peroxide value (5.8meq/kg) indicates that the oil is stable.

The relative density and the refractive index of the *Citrus sinesis* seed oil was found to be 0.8812g/cm^3 and 1.457 at 25°C respectively, which falls within the range of recommended values of 1.445-1.470 refractive index for edible vegetable oil [6].

CONCLUSION

The seeds of *Citrus sinesis* contain high level of oil that has great potential use as industrial as well as domestic oil.

Table 1: Physicochemical parameters of *Citrus sinesis* seed oil

Properties	Results
Percentage yield (%)	
Extraction with petroleum spirit	56.0
Extraction with Diethyl ether	56.3
Colour	Light yellow
Texture at 37°C	Liquid
Moisture content (%)	4.0
Relative density (g/cm^3)	0.8812
Refractive index	1.457
Ash content (%)	5.0
Percentage organic matter (%)	95.0
Iodine value (g/100g)	54.l9
Saponification value (mgKOH/g)	190.32
Peroxide value (meq/kg)	5.8

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