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EXTRACTION AND CHARACTERIZATION OF WATERMELON SEED OIL

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Abstract

Oil extracts from a number of many fruits, nuts and seeds are being used in soap making, cooking, pharmaceutical products and other industries. The seeds of numerous fruits are thrown out as waste despite of their nutritional values. Many of them represent an important source of high quality protein and vitamins. Watermelon seed oil has a large concentration of unsaturated fatty acids. The low acid, peroxide and iodine values of the oil together with some favourable functional properties makes the watermelon seed and seed oil suitable for human use rather than as a waste where the crop is grown in abundance. The study was realized using Soxhlet method and micro-Kjeldahl. Citrullus lanatus seed oil has a lot of nutritional compounds. As mineral content is shown that is rich in phosphorus 30.8 mg, magnesium 2.98 mg and sodium 2.3 mg. The fatty acid composition is predominant by linoleic acid 65.7%, polyunsaturated fatty acid 66.03% and saturated fatty acids 17.87%.

Key words*: watermelon seed, oil characteristics, nutritional quality.*

INTRODUCTION

Vegetable oils are essential in satisfying global nutritional requests and are exploid in industrial and food purposes. Although the broad range of sources of vegetable oils, the world comsumption is dominated by sunflower, soya bean and rapeseed oils. These sources are conventional and no longer meet the increasing demands of industrial and domestic sectors. From this point of view, non-conventional seed oils are much approachable to confront with this challenge. It was reported that seeds of some species of *Cucurbitaceae* family can be the edible oil sources to meet the increasing demands for vegetable oil (Stevenson, 2007).

Citrullus lanatus is grown all over the warm parts of the globe and is one of the under-utilized fruits. Watermelon is almost exclusively consumed as a fresh fruit. The pulp can be also found in the composition of juices, jams, salads, jellies and even sauces. In some regions of Middle East and in China seeds are consumed. In other countries the peel is pickled. Some beers have in the ingredients watermelon juice. In India make bread using the seed flour. But in most parts the seed and rind remaining major wastes (Dias, 2010).

The largest producers of watermelon are China with 79.2 million tons, Turkey with 3.9 tons and followed by Iran with 2.8 million tons. Total production volume is aproximative 117.01 million tons.

Watermelon seeds remain intact after removing the pulp and rind making them usable in the food industry.

The seeds consist in 57.1% fat, 31.9% protein, 8.2% fiber, 6.2% ash, 4.4% carbohydrates, 456 mg phosphorus, 130 mg calcium and 7.5 mg iron. It also contains aminoacids as Isoleucine, Leucine, Tryptophan and Valine (Adejumo, 2015).

According to the results obtained by Hassan et al. in 2013 in the oil are predominant high levels of fatty acids (78.35%) such as linoleic acid (59.6%), stearic, palmitic and oleic acids (18.1%). The seeds contain protein (35.66-36.47%) and oil (50.10-51.01%) making them good to use in food formulations.

To extract lipids from seed oil are used organic solvents, but this has some disadvantages because of the possibility of thermal degradation of the functional compounds and the unsaturated fatty acids, depending on the need to eliminate the residues of organic solvent from the oil (Costa de Conto, 2011).

The main objective of this study is to determine the physicochemicals proprieties and the fatty acid composition of the *Citrullus lanatus* seed oil practicing the solvent extraction method.

MATERIALS AND METHODS

A number of 10 pieces of watermelon (*Citrullus lanatus*) were purchased from a local producer (Călărași, Romania) during the summer season of 2019. The selection of the seeds was done in a random way.

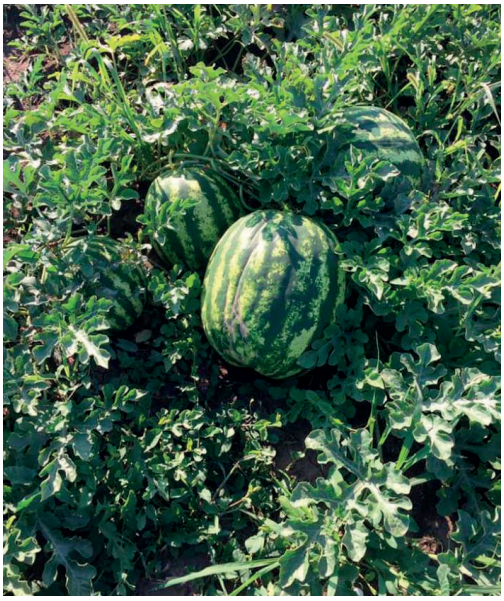

Figure 1. Landscape of watermelon producer

Watermelons were cutted into slices and the seeds were collected using the hands and then washed with distilled water. They were put to dry in the sun at aproximative 28°C for a week. The seeds were peeled manually after being shelled by breaking them with a metal cylinder to remove the kernels. Fine flour of kernels was obtained after they were ground with a coffee grinder and stored in the refrigerator at 6°C until analyses were made.

Extraction

The crude fat was extracted using Soxhlet method and total nitrogen with micro-Kjeldahl. The protein was calculated N x 5.3. The refractive index at 25°C, the acid value Cd 3a-63, the peroxide value Cd 8-53, the saponification value Cd 3-25, the iodine value Cd 1-25 and the unsaponificable matter Ca 6a-40 of the samples of oil were determined.

Using the 743 Rancimat at 110°C was evaluated the oxidative stability of the *Citrullus lanatus* oil. The oil sample weighing 3 g was placed in the Rancimat apparatus and heated under an airflow rate of 4 L/h. After 30 minutes the temperature reached 110°C, the vessels head outlets were connected to the conductivity cells, the air flow rate was increased to 20 L/h so the measurement started.

All analyses were repeated three times and expressed as ± SD.

Using boron trifluoride the methyl ester of crude oil was prepared. A gas chromatograph was used to separate the methyl esters using helium as the carrier gas.

Mineral contents and physicochemical properties were characterised by using various standard methods.

Percentage oil yield

The percentage of oil yield of the seeds was determinated and calculated using the equation:

$$\text{Percentage yield} = \frac{\text{weight of extracted oil}}{\text{weight of seed}} \times 100$$

Specific gravity

The specific gravity of the watermelon oil was identified emplying the ratio weight of the oil to the equivalent weight of water corresponding to the next formula:

$$\text{Specific gravity} = \frac{W1}{\text{W2}}$$

Where: W_1 is noted the weight of the oil and W_2 is weight of equivalent volume of water.

Acid value

For the measurement of acid value some ethanol was heated on a water bath for a few number of minutes to eliminate dissolved gases. After it was then neutralised by including small drops of phenolphtalein and potassium hydroxide till a light pink colour was achieved. A quantity 6 g of oil was weighed into a conical bottle and 50 ml of boiling already neutralized ethanol was supplementary added. The fusion was then titrated with potassium hydroxide solution before the pink colour recurred.

The acid value was calculated using:

$$\text{Acid value} = \frac{\text{titre value(ml)x N x 56.1}}{\text{weight of sample}}$$

Where: N is noted normality of KOH is 0.1M and 56.1 is molar mass of KOH.

Free fatty acid

Oleic acid as the percentage of free fatty acid was calculated by multiplying the acid value with the 0.503.

Saponification value

For saponification value was used 2 g of oil into a conical cilinder and 25 ml of alcoholic KOH was combined. A blank was also processed by setting 25 ml of alcoholic KOH in a identical cilinder. Reflux condensers were implemented to bottles and the constituents were heated in a water bath for exactly 60 minutes, rolling the bottle from time to time. The bottles were then let to cool for awhile and after the condensers washed down with some distillated water. The surplus KOH has been titrated with HCl acid utilizing phenolphthalein indicator. Then was calculated applying the upcoming formula:

$$\text{Saponification value} = \frac{(b-a)\text{x F x } 28.05}{\text{weight of sample}}$$

Where:
b is notted titre value of blank (ml)
a is notted titre value of sample (ml)
F is factor of 0.46 M HCl = 1
28.05 = mg of KOH proportionate to 1 ml of 0.46 M HCl

Peroxide value

Peroxide value of the *Citrullus lanatus* oil was calculated using 2 g of oil sample scaled into a 500 ml conical cylinder and 10 ml of chloroform was mixed to disslolve the sampling. This was succeded by including of 15 ml of acetic acid plus 1ml of freshly processed saturated potassium iodide suspension. The cylinder then was rapidly closed, stirred for 1 minute and stored at room temperature for 10 minutes away from sunlight. A quantity 75 ml of distilled water was mixed to the content of the cylinder and next shaken energetically. Several drops of starch solution were joined as indicator. The solution of liberated iodine was titrated across 0.01 N sodium thiosulphate solution. The alike procedure was applied out for blank and the peroxide value indicated in milliequivalent of active oxygen per kg of sample was notted:

$$\text{Peroxide value} = \frac{V1-V0\ x\ T\ x\ 1000}{M}$$

With V_0 is indicated the volume of the sodium thiosulphate solution needed for blank.
With V_1 is the volume of the sodium thiosulphate solution needed for determination of the sample.
With T is notted normality of the sodium thiosulfate used.
And with M is represented the mass of the test sample in grams.

Iodine value

Iodine value of the watermelon oil was asessed using to the titrimetric method. A value 2 g of oil specimen was weighed into a moistureless 250 ml glass bottle and 10 ml of carbon tetrachloride was mixed with the oil. Exactly 20 ml of Wij's solution was then combined and allowed to stay in the dark for 20 minutes. A quantity of 15 ml potassium iodide (10%) plus 100 ml of water was joined and the resulting blend was then titrated with 0.1 M sodium thiosulphate solution taking starch as indicator just before the final point. A blank determination was made out parallel to the oil samples. The formula is:

$$\text{Iodine value} = \frac{(V2-V1)x\ 1.269}{weight\ of\ sample\ (g)}$$

Where: V_2 is titer value for blank, and V_1 is titer value for sample.

Mineral contents

Determination of mineral contents of oil: The oil sampling were digested individually for mineral analysis by wet digestion method expressed by Oluremi in 2013. Amount of 0.5 g of sample was scaled and moved into 75 ml micro digestion tubes. Concentrated solution of H_2SO_4 in 4 ml quantity and 2 ml H_2O_2 solution were combined delicately. The tubes were warmed in a block digester that was preheated to 270°C for a half an hour. They were then removed out and let to lose heat. Another division of 2 ml H_2O_2 was added extra and warmed more to accomplish complete digestion that was indicated by a clear solution aspect.

Magnesium, copper, zinc, iron and calcium quantities were identified using an Atomic Absorption Spectrophotometer. Sodium and potassium were determined using Flame Photometer while phosphorus using a Spectrophotometer.

RESULTS AND DISCUSSIONS

The physicochemical properties of the *Citrullus lanatus* seed oil tested in this article are featured in Table 1 in correlation with the literature.

Table 1. Physicochemical properties of watermelon seed oil

	Watermelon seed oil	Literature according to Adejumo, 2015
Colour	Orange	Golden yellow
Odour	Fruity	Fruity
State at room temperature	Liquid	Liquid
Specific gravity (g/ml)	0.92±0.10	0.918±0.002
Percentage oil yield	33.02±0.22	30-34%
Acid value (mg KOH/g)	3.93±0.11	2.283
Percentage of free fatty acids (oleic acid)	1.97±0.12	1.15
Saponification value (mg/KOH/g)	65.30±1.00	201
Peroxide value (mg O2/g)	8.51±0.90	3.4
Ester value	62.56±0.20	
Iodine value (GI2/100 g)	5.07±0.10	115

The specific gravity is 0.92 g/ml and in the literature in comparison to Adejumo 0.918 g/ml. Percentage oil yield is is between the range. The acid value in this study is big.

The peroxide value is 8.51 mg O_2/g and according to The Codex Alimentarius Commision the specified permitted maximum peroxide level that does not exceed 10 mequiv of peroxide oxygen/kg of oil, for example coconut oil, soybean and cotton seed.

Minerals are very important for the human health. The body uses minerals for different functions that include hormone production, bones construction and regulation of heart functions. Macrominerals are minerals that the organism needs in big amounts and these include calcium, magnesium, phosphorus, potassium, sodium, chloride and sulfur. The body also needs some small amounts of iron, copper, manganese, cobalt, zinc, selenium and fluoride. The best way of getting these minerals in the body is consuming food that have them in their composition. The daily recommended dose of calcium is 1000 mg, magnesium 310 mg, potassium 4.7 mg. Watermelon seed oil mineral content is presented in Table 2.

Table 2. Mineral content of watermelon seed oil

	mg/100 g
Phosphorus	30.87
Calcium	2.07
Magnesium	2.98
Potassium	0.86
Sodium	2.35
Iron	1.6
Copper	0.75
Zinc	1.26

Table 3 shows the fatty acid composition of the watermelon seed oil.

Table 3. Fatty acid composition for watermelon seed oil

Fatty acid	%
Myristic acid	0.07±0.0
Palmitic acid	10.01±0.11
Palmitoleic acid	0.10±0.00
Stearic acid	6.98±0.01
Oleic acid	15.89±0.15
Linoleic acid	65.73±0.07
Linolenic acid	0.20±0.03
Arachidic acid	0.21±0.00
Gadoleic acid	0.12±0.01
Behenic acid	0.10±0.02
Erucic acid	0.05±0.04
Lignoceric acid	0.05±0.00
Saturated fatty acids	17.87
Monounsaturated fatty acids	15.98
Polyunsaturated fatty acids	66.03

Oleic, palmitic and linoleic acids are the main fatty acids present in the seed oil, with a predominance of linoleic acid (65.73%), similar to the results found by Okunrobo (65.85%). Oleic acid is utilized as an excipient in pharmaceutical products and is also a emulsifying agent. Palmitic and linoleic acids

are used in the cosmetic industry and soap making.

The values however, are an indication that the *Citrullus lanatus* oil has a valuable number of long chain fatty acids in structure.

CONCLUSIONS

The results obtained in this research work in comparison with literature have shown that the percentage yield of the watermelon seed oil is high enough to be commercialized. If harvested, it will reduce the amount of agricultural waste and serve as a valuable source.

The *Citrullus lanatus* oil has a lot of nutritional values and can be consumed and also utilized for soap making because of its physicochemical properties.

Because of the high level of phosphorus in the oil it can be used also in pharmaceutical industry. The peroxide value indicated that is stable to auto-oxidation and can be stored for a long time under normal conditions. It is less susceptible to rancidity.

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