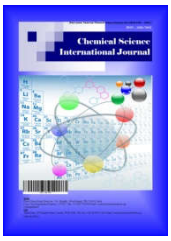

Chemical Science International Journal

29(6): 29-37, 2020; Article no.CSIJ.59021
ISSN: 2456-706X
(Past name: American Chemical Science Journal, Past ISSN: 2249-0205)

Comparative Study of Extraction Methods on the Composition and Physicochemical Properties of Vegetable Oil from the Seeds of *Ricinus communis*

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Authors' contributions

This work was carried out in collaboration by all authors. Authors RDTN, AGBA and JCN designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors DRTT, GMM, MAC and RDTN managed the analyses of the study. Authors DRTT, RDTN, AGBA, MAC, GMM and JCN managed the literature search. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CSJI/2020/v29i630188
Editor(s):
(1) Prof. Akmal S. Gaballa, Zagazig University, Egypt.
Reviewers:
(1) A. V. L. N. S. H. Hariharan, Gitam University, India.
(2) Le Van Tan, Industrial University, Vietnam.
Complete Peer review History: http://www.sdiarticle4.com/review-history/59021

Original Research Article

Received 10 May 2020
Accepted 17 July 2020
Published 27 August 2020

ABSTRACT

This paper carries out the effects of the extraction methods on the quantities and qualities of vegetable oil from *Ricinus communis*. The same initial quantities of *Ricinus communis* were used to extract oil by the physical methods, hot and cold hydraulic press, and screw press; and the chemical method of solvent extraction using various solvents at the same temperature. The resulting oils were characterized to obtain the acid index, saponification index, iodine index,

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peroxide index, water content, specific density, kinematic viscosity at 40°C, color, hydrogen potential (pH), refractive index, the fatty acid profile of the oils by gas chromatography (GC) and the density at 15°C; in accordance with the ASTM standard specification. The results obtained show that, quantitatively, the hydraulic press has the best yield (46.25%) and qualitatively, the screw press extract has the best properties on a wide range of properties. This result allows us to conclude that the extraction methods depend on the use or application of the vegetable oil.

Keywords: Extraction; Ricinus communis; characterizations; chromatography; yield.

1. INTRODUCTION

The castor plant *(Ricinus communis L.)*, is a non-edible plant of the E*uphorbiaceae* family, which produces seeds known for their high oil content. It is essentially a tropical plant, although it can grow in temperate regions [1]. The use of edible oils in industry such as soybean oil or palm oil risks generating new humanitarian and ethical problems. So, the inedible castor seeds make it a prime raw material for the industry. Several works have been carried out on castor oil for industrial applications. Castor oil is one of the few naturally occurring triglycerides, close to being a pure compound, since the fatty acid portion is nearly nine-tenths ricinoleic [2]. Its crude form has a pale straw colour that turns colourless or slightly yellowish after refining and bleaching. According to Marter (1981), castor oil, a pale amber viscous liquid derived from the seeds of the plant *Ricinus communis*is is sometimes known as ricinus oil. Castor oil is one of the few naturally occurring glycerides that approaches being a pure compound, since the fatty acid portion in it is nearly nineteenths ricinoleic acid [3]. Castor oil has been widely investigated as a raw material for the production of biofuel [4]. Nkouetcha et al. (2019) used castor oil as an alternative to mineral oil in power transformers [5]. Amdebrhan et al. (2015) has shown that castor oil can be used as a bio-lubricant [6]. Castor oil can be extracted by different processes (mechanical and solvent extractions) or combination of processes [7,8]. Castor oils studies have shown increasing growth with the number of manuscripts increasing six fold since 1980. While alternative breeding programs and marketing can lead to economic growth of castor oil production, at the commercial level, various projects fail due to the lack of knowledge about novel processing methods and parameters used in castor oil production. In 2008, three countries, India, China, and Brazil produced 93% of the world's supply of castor oil. However, the method used had an impact on the physicochemical properties and the quality of the oils obtained. The aim of this work is the extraction by chemical and mechanical methods followed by physico-chemical characterization of castor oil from wild *Ricinus communis* L. seeds found in Douala, Cameroon, with the view of bringing out its biofuel potentials. To achieve this, two mechanical methods and four chemical methods were used for extraction.

2. MATERIALS AND METHODS

2.1 Vegetable Material

The castor seeds used in this work were collected in Douala, Littoral region of Cameroon. Several operations are involved in the production process of the vegetable oil of *Ricinus communis*: the ripe castor fruits harvested were manually cleaned and sun-dried for a week on an aluminum plate, until the fruit capsules open to discharge the coated seeds. This was followed by the removal of pods and winnowing from the tray to separate the shells from the beans.

2.2 Oil Yield

Yield of the extracted oil is expressed in percentage, which is defined as weight of oil extracted over weight of the sample taken. The percentage oil yield was calculated as follows [9].

$$\text{Oil yield, \%} = \frac{\text{Weight of oil obtained}}{\text{Weight of seeds used for extraction}} \times 100$$

2.3 Oil Extraction Methods

2.3.1 Mechanical method

Two extraction methods; hydraulic and screw press were used to extract castor oil from the seeds.

2.3.1.1 Screw press method

A manual DIY vertical screw press was used for this method. It has a cylindrical cage perforated

at the base and a tapered rod with a bar (serving as a handle) at its upper end. 80 g of crushed seeds were placed in a tissue which was immediately introduced into the cylindrical cage of the press. The oil was produced by compressing the pulp from the crushed seeds. The extraction took 20 to 40 minutes with the pressure gradually increased after every 5 minutes.

2.3.1.2 Hydraulic press method

The extraction equipment was a 10 ton DIY hydraulic press. 80 g of crushed seeds, placed in a cotton cloth, was immediately introduced inside the press by its upper inlet. Once the inlet was closed, the hydraulic cylinder increased the pressure in the pressure chamber, thereby causing the crude oil to flow out. The extraction lasted for 30 to 60 minutes with the pressure being gradually increased every 5 minutes.

2.3.2 Solvent extraction

Two chemical extraction techniques, soxhlet and infusion were used.

2.3.2.1 Soxhlet extraction

80 g of ground grains were introduced into a cartridge before being introduced into the cage of the Soxhlet. It was connected to a flask comprising 750 mL of an extraction solvent hexane, acetate or methanol, and attached to a heating device. When the solvent was boiling to 50-80°C, the vapor rose through the vertical tube in the condenser at the top. The liquid condensate dripped into the filter paper cartridge in the center, which contained the solid sample from which oil was to be extracted. The extract seeped through the pores of the filter paper into the siphon tube and descended into the round bottom flask. This operation took 4 hours. It was then removed from the tube, dried in the oven, cooled in the desiccator and weighed again to determine the amount of oil extracted. The solvent/oil mixture was separated from each other using a rotary evaporator.

2.3.2.2 Infusion

80 g of previously crushed sample was immersed in water, boiled, homogenized and filtered. The filtrate was allowed to cool and then brought to a boil in order to evaporate the water and obtain the oil.

2.4 Chemical Analysis

2.4.1 Acid value (ASTM D664)

The free fatty acid in oil was estimated by titration using potassium hydroxide (KOH). The acid value is defined as the mg of KOH required to neutralize the free fatty acids present in 1 g of sample. Acid values of ricinus commuis seed oil was determined by titration method [10].

2.4.2 Saponification value (IP 136)

The saponification value in oil was estimated by titrating the excess KOH from a mixture of 2 g of oil with 0.5 N solution of KOH-ethanol and boiled for at least 60 minutes, with 0.5 N hydrochloric acid (HCl) solution. A blank test was prepared by following the same procedure. The saponification index (IS) was determined as follows:

$$IS = 56.1(V_0-V_1)C/m$$

V_0: is the volume of hydrochloric acid (mL) necessary to titrate the blank,
V_1: is the volume of hydrochloric acid (mL) necessary to titrate the test,
C: is the exact concentration, in moles per liter, of the standard solution of hydrochloric acid used,
m: is the mass (g) of the test portion.

2.4.3 Iodine value (ISO 3961)

The iodine value is a measure of the degree of unsaturation of fatty acids and is used to characterize oils and fats. The ricinus commuis seed oil contains both saturated and unsaturated fatty acids. Halogens add across the double bonds of unsaturated fatty acids to form additional compounds. Iodine monochloride (ICl) is allowed to react with the fat in the dark. Iodine gets incorporated into the fatty acids chains wherever the double bond exists. The amount of iodine consumed is then determined by titrating the iodine released after adding KI with standard thiosulphate and comparing with a blank in which the fat is omitted. Hence, the measure of iodine absorbed by an oil or fat gives the degree of unsaturation. Iodine value of garden cress seed oil was determined by titration method [11].

2.4.4 Peroxide value (ISO 3960)

The peroxide value (PV) was determined by iodometric titration, which measures the iodine produced from potassium iodide by the peroxides

present in the fat sample. A sample of 2.0 g oil was dissolved in 30 mL mixture of glacial acetic acid and chloroforms in the ratio 30:70 v/v. Then 0.5 mL saturated potassium iodide solution was added. After 1 min under darkness, 30 mL of distilled water was immediately added the I_2 liberated was titrated with 0.01 N sodium thiosulphate.

2.4.5 Gas chromatography ASTM D-6584

Gas chromatography measurements were carried out using the method described by ASTM D-6584. The methyl esters in castor oil were analyzed using GC-MS (Agilent 5975 GC - MSD) with software in Chem station 6890 scale mode. This method was carried out, followed by the standard method ASTM D-6584 equipped with " an FID detector and a capillary column (30 m × 0.25 mm). The sample was introduced into the 7683-B injector. The initial temperature was set at around 130°C. Then it was increased to 220°C at the ramp speed of 3°C/min and held for 15 minutes. The carrier gas used was helium, at a flow rate of approximately 1 mL/min. Using a fractionated injection system, 2 µL of samples were injected into the column. Then, 70 eV were used in a range of 50 to 550 m/z and finally, the different chromatograms of methyl ester samples were obtained.

2.5 Physical Analysis of *Ricinus communis* Oil

2.5.1 Specific gravity (IP 190)

Specific gravity was measured as described by AOCS (1998). An empty pycnometer bottle (W1), filled with water (W2), and oil (W3) were weighed separately. The specific gravity (SG) of the oil was calculated using mathematical relationship:

$$\text{Specific gravity (SG)} = \frac{\text{W3} - \text{W1}}{\text{W2} - \text{W1}}$$

2.5.2 Density (ASTM D3505)

The density of an oil characterizes the mass of that oil per unit volume. The density of oils was measured using a 0.01 g digital analytical balance, a 25 mL pycnometer and a thermo-regulated vessel. The density was determined from the weight of the oil introduced into the pycnometer.

2.5.3 Viscosity (ASTM 445) [12]

The kinematic viscosity was investigated with regard to standard ASTM 445, by means of a viscometer, capillary, made of UBBELOHDE type glass, of a viscometer support which makes it possible to maintain in a vertical position, a thermostated bath, which contains water of sufficient depth, a stopwatch for measuring time and a clean and dry filter paper. For a given test temperature, the viscosity was determined by the formula:

$$\eta c \left[\frac{\text{mm}^2}{\text{s}}\right] = \text{k} * \text{t}$$

$$\text{Where, } \begin{cases} \text{k} = \text{Viscometer constant} \\ \text{t} = \text{liquid flow time} \end{cases}$$

2.5.4 Hydrogen potential (pH) (ASTM D1293)

The pH of a 2.30 g dispersion of the oil in 15 cm^3 hot water was determined with the aid of a glass electrode pH meter (pH-2601).

2.5.5 Refractive Index (ASTM D1218)

Refractive index was measured at ambient temperature using a refractometer. The AOAC (1990) procedure was followed.

2.5.6 Colors (ASTM D1500)

The color is often used as a qualitative method. The technique is based on the comparison of oil color to a standard colored and numbered disc. An increasing or high color number is an indication of contamination, deterioration, or both [6]. It can be seen in Table 1; guideline for Quality Index Number (QIN) and eventual actions to be taken on the oil.

Table 1. Quality index number and effect of oil quality on transformer's condition

Colors	Good Oils	Proposition A Oils	Marginal Oils	Bad Oils	Very Bad Oil	Extremely Bad Oils	Disastrous Oils
Index	1	2	3	4	5	6	7
quality	Pale Yellow	Yellow	Bright Yellow	Amber	Brown	Dark Brown	Black

3. RESULTS AND DISCUSSION

3.1 Moisture Content of Seeds

The result of the moisture content of seed are given in Table 2.

The moisture content of *Ricinus communis* seed was 6.62%. This result is acceptable because to produce a good quality oil, the moisture content must be between 5 and 7% [13].

3.2 Result of Extractions

Table 3 shows the results of the extraction of oil from castor beans.

It shows the extraction yield obtained using different extraction methods. The best yield was obtained by the cold hydraulic press (46.25%). All these methods allow to have a maximum yield which is between 30 and 55% of castor oil, whether pressed mechanically or by solvent [4]. In general, the extraction yield of oil from the seeds depend on plant species, genetic and climatic variations within the same species [14]. For *Ricinus communis* seed oil, our results showed that the extraction method was the main factor that could significantly influence the extraction yield.

3.3 Characterization Results

3.3.1 Chemical properties

The effect of extraction procedure on the chemical characteristics of the oil, acidity, iodine value (Iv), saponification value and peroxide index, is shown in Table 4.

3.3.2 Iodine index (Ii)

The iodine index provides information on the degree of unsaturation of the oil and makes it possible to classify non-drying (Ii < 100), semi-drying (100 < Ii < 130) and drying (Ii > 130) oils. The oils extracted would be non-drying oils consisting of a small amount of polyunsaturated fatty acids (PUFA) [15]. The significant difference between these oils is due to the auto-oxidation (catalyzed by temperature) of the unsaturated fatty acids. These values obtained between (31.73 -68.74) g I_2/100 g of oils) were lower than the limit (82-88) g I_2/100 g of oil) fixed by the ASTM standard for the qualitative classification of castor oil proof that this oil is good quality and not been subjected to any alteration.

Table 2. Moisture content of seeds

Time in hours	0	2	4	6	8	12
Mass in grams	66.24	64.51	63.27	62.56	62.3	62.3

Moisture content = 6.32%

Table 3. Yield of oils extracted from castor seeds

Extraction Type	**Soxhlet**				**Mechanical**		
Solvent	HEX	MET	AC	Water	PHY	PHC	PV
Oil yield in %	30.75	37.87	33.03	36.31	46.25	44.47	25.62

HEX: oil extracted with hexane; MET: oil extracted with methanol; AC: oil extracted with ethyl acetate; Water: oil extracted with water (infusion); PHY: oil extracted with a hot hydraulic press; PHC: oil extracted by hydraulic press; PV: oil extracted with a screw press

Table 4. Chemical index for oils

Extraction	**(Av) (mg KOH/g)**	**% FFA (mg KOH/g)**	**Ii ($g\ I_2$/100g)**	**PI ($m_{eq}O_2$/ Kg)**	**SI (mg KOH/g)**
HPY	2.36 ± 00	1.18 ± 00	60.74 ± 7.48	8.33 ± 2.89	176 ± 2.04
HC	3.74 ± 0.32	1.87 ± 0.16	58.16 ± 7.48	11.67 ± 2.89	190 ± 7.48
HPV	1.50 ± 0.32	0.75 ± 0.16	52.88 ± 14.96	12.50 ± 3.54	178 ± 1.86
HEX	2.81 ± 00	1.40 ± 00	68.74 ± 22.43	15 ± 10	187 ± 2.57
AC	1.32 ± 0.31	0.75 ± 0.16	31.73 ± 29.91	11.67 ± 2.87	181 ± 2.23
MET	0.56 ± 00	0.28 ± 00	63.45 ± 00	17.5 ± 10.61	185 ± 3.06
INF	2.43 ± 0.32	1.21 ± 0.16	47.59 ± 7.48	10 ± 5	184 ± 7.25

Av: acid value, Ii: Iodine Index, PI: peroxide index, SI: saponification index, FFA: Free Fatty acid

3.3.3 Acid value (Av)

The acid values obtained are all different according to the extraction technique and vary from (0.5 to 3.74) mg KOH/g of oil. The acidity was significantly higher in the oil extract with the method of hot hydraulic press (3.74%) in comparison to the other methods. This could be explained by the presence of immature sheaths. The low fatty acid content of the oils will result in a low percentage of free fatty acid. These values of 0.5-3.74 mg KOH/g of oil were lower than the upper limit 0.4-4 mg of KOH/g set by the ASTM standard for the qualitative classification of castor oil.

3.3.4 Saponification index (SI)

The saponification index of the extracted oils was different and ranged from (176-190) mg KOH/g oil. Some oils (HC, HEX, MET and INF) had a saponification value greater than 182 mg KOH/g oil [4]. This could be explained by the polarity of the solvent used and the extraction method.

3.3.5 Peroxide index (PI)

The peroxide index of the oils obtained was different and range between (8.33 -17.5) meq O_2/kg of oil. These values are lower than the standards defined by the codex alimentarius (10 to 20 meq O_2/Kg of oil) and show that the oils studied had not yet undergone any alteration. Fresh oils have a peroxide index of less than 10 meq O_2/Kg of oil and they degrade gradually when the peroxide index is between 20 and 40 meqO_2/k [16].

3.4 Physical Properties

Table 5 presents a comparison between the physical properties of the castor oil extracted.

Table 5 shows that there are significant differences between the physical properties of these oils.

3.4.1 pH

The pH of the oils is between 5.5 and 6.1. This result shows that castor oil is made up of fatty acid, which justifies its acid character.

3.4.2 Color

The color of the extracted oils is slightly different. The difference is in the castor oil which had a bright yellow color. This color was due to the temperature because the extraction was done in a hot medium. Nevertheless, this oil was of good quality [5].

3.4.3 Specific gravity and density

The specific gravity and density are closely linked, the density of the oils extracted is between 0.903-0.943. This difference in densities could be due to the extraction method, the presence of particles in the oil and the extraction temperature.

3.4.4 Refractive index

The refractive index of the extracted oils is between 1.476 - 1.480. This slight difference can be attributed to the presence of impurities in the oil from the extraction method.

3.4.5 Kinematic Viscosity

Differences were observed between the values obtained for the viscosity of the oils extracted (804-1048) mm²/s. This large difference could be attributed to the presence of impurities, the extraction temperature and the polarity of the solvent used.

3.5 Fatty Acid Composition of Extracted Oils

Fatty acid profile of castor vegetable seed oil extracted by different method is presented in Table 6.

Table 5. Results of physical properties of extracted oils

Extraction type	Soxhlet			Infusion	Mechanical		
Solvent	**HEX**	**MET**	**AC**	**Water**	**PHY**	**PHC**	**PV**
Specific density	0.913	0.939	0.922	0.903	0.915	0.943	0.920
Specific gravity g/cm^3	0.864	0.888	0.872	0.854	0.866	0.892	0.87
Refractive index	1.479	1.477	1.477	1.480	1.478	1.480	1.476
Viscosity (mm²/s)	864	1004	804	1048	858	910	886
pH	6.02	5.92	5.70	6.05	6.00	5.50	6.10
Color	1	1	1	1	1	3	1

Table 6. Fatty acid composition (%) of castor oils extracted by all methods

Extraction type	Soxhlet			infusion	Mechanical		
Solvent	**HEX**	**MET**	**AC**	**Water**	**PHY**	**PHC**	**PV**
Hexadecanoic acide methyl ester	3.15	2.69	3.8	3.04	2.61	2.37	2.72
9,12-octadecadienoic acid (Z, Z)-, méthylester	8.02	8.46	7.62	7.24	8.46	8.18	7.04
9-octadecenoic acid (Z)-,methylester	4.48	4.95	3.94	3.98	4.92	3.98	3.89
Trans-13-octadecenoic acid, methyl ester	0.62	0.52	0.53	0.4	0.70	5.47	0.47
Octadecanoic acid, methyl ester	2.97	2.45	3.47	2.91	2.4	2.31	2.53
9-octadecenoic acid,12-hydroxy-,methyl ester,(Z)-	77.42	78.82	75.97	78.43	79.08	77.66	81.24
9-octadecenoic acid, 12-hydroxy- 2,3dihydroxy-propyl ester	1	1.13	/	1.44	/	2.15	/
Octadecanoic acid,9,10-epoxy,methyl ester	1	/	/	1.12	0.77	0.8	/
10,13-eicosadienoic acid methyl ester	1.34	/	/	1.43	1.07	1.25	1.36
Octadecanoic acid,9,10 dihydroxy,methylester	/	0.97	1.46	/	/	/	0.75
Phthalicacid,di(2-propylpentyl) ester	/	/	1.3	/	/	/	1.3
9-hexadecenoic acid, methyl ester,(Z)-	/	/	1.92	/	/	/	/

Table 7. Comparison of fatty acid contents of extracted oils and other oils

Fatty acid	Percentage		
	Malaysia [16]	Brazil [17]	This work
Palmitic (C16:0)	1.3	0.7	2.37 - 3.80
Stearic (C18:0)	1.2	0.9	2.31 - 3.47
Oleic (C18:1 ω 9^e)	5.5	2.8	3.89 - 4.9
Linoleic (C18:2 ω6)	7.3	4.4	7.04 - 8.4
Linolenic (C18:3 ω3)	0.5	0.2	/
Ricinoleic (C18:1ω OH)	84.2	90.2	75.97 - 81.2
13-octadecenoic (C18:1ω 13^e)	/	/	0.4 - 4.6
10, 13 Eicosadienoic (C20:2ω)	/	/-	1.07 - 1.4
Saturated fatty acids	2.5	1.6	4.68 - 7.2
Unsaturated fatty acids	97.5	97.6	93.05 - 99.1

The *Ricinus communis* vegetable oil extracted by different methods did not have the same fatty acid composition. We note the presence of seven fatty acids whose composition varies. We distinguish palmitic acid (2.37 - 3.80%); stearic acid (2.31 - 3.47)%; oleic acid (3.89 - 4.9%); linoleic acid (7.04 - 8.4)%; Ricinoleic acid (75.97 - 81.2)%; 13-octadecenoic acid (0.4 - 4.6)%; and 10.13 eicosadienoic acid (1.07 - 1.4)%. This difference could be attributed to the extraction method used.

A comparison between the extracted oils and other castor oils was made and the results recorded in Table 7.

It can be seen that the oils extracted using several methods were different in composition and fatty acid content of the castor oil from other countries (Malaysia and Brazil). This difference could be attributed to climatic conditions, the sensitivity of the acid profile determination devices and the extraction method used.

Regarding the physicochemical properties studied and the content of ricinoleic acid (which gives castor oil good adhesion to metal surfaces), the oils extracted comply with international standards and can be used for multiple purposes. An in-depth analysis shows that the oil extracted with hexane (HEX) and screw press (PV) could be used as biodiesel; oil extracted with methanol (MET) and hydraulic press (PHY) as FR3 insulating liquid (ENVIROTEMP FR3); the oil extracted with a hot hydraulic press (PHC) and ethyl acetate (AC) as Rhodorsil insulating liquid; oil extracted with water (WATER) as biolubricant/biodiesel. In general, all the oils extracted have characteristics required in industry and can therefore be used as industrial oils.

4. CONCLUSION

This study allowed a comparison of extraction methods and evaluated the effect of the method on the composition and physicochemical properties of a vegetable oil from *Ricinus communis* seeds. We varied the extraction methods, and each product obtained was subjected to the analysis of a chromatographic profile and to a physical and chemical characterization in order to evaluate the effect of the method on the ricinoleic acid composition, on the physicochemical properties of the oils extracted and on the use of the oil. It emerges from these studies that quantitatively, the hydraulic press offers a good yield (44.47%) and qualitatively, the screw press offers excellent physicochemical properties and a better yield of ricinoleic acid compared to the other methods. Thus, the choice of the extraction method leads to a very precise use. An improvement of the screw press would make it very efficient, inexpensive and easily accessible. An improvement in viscosity by chemical treatment will increase the range of use of the oils.

ACKNOWLEDGEMENTS

The authors greatly thank assistance rendered by M. Emeric Tchamdjio Nkouetcha for the Laboratory of Technology and Applied Sciences, University of Douala and all the members of our research group in the Analytical, Structural and Materials Chemistry Laboratory, Department of Chemistry, Faculty of Sciences, University of Douala.

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/59021