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Studies on Ethiopian Castor Seed (*Ricinus communis* L.): Extraction and Characterization of Seed Oil

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ABSTRACT

In this study, Ethiopian castor seeds were collected from a local market and studied for their oil yield and physicochemical characteristics including fatty acid composition. Their oil was extracted using mechanical method and using hexane, ethyl acetate, and methanol solvents and its physicochemical properties were studied. It was done by varying solid-to-solvent ratio (1:9, 1:12 and 1:15) and time (1, 3, 5, 6, and 7 h) for the different solvents used for the extraction. A maximum yield of 72.3% for methanol, 68% for ethyl acetate and 61.6% for hexane was obtained at optimum conditions of 1:12 ratio and 7 h extraction. Specific gravity, viscosity, moisture content, refractive index, acid value, saponification value, iodine value, and peroxide value were measured for the different types of oils. Gas chromatography-mass spectrometry analysis of the four types of oils showed that polar solvents give slightly higher ricinoleic acid content than hexane-extracted oil. Hence, use of polar solvents gave the highest yield, good quality, and purer fatty acid composition for the oil prepared from Ethiopian castor seed, making it suitable for industrial applications due to ease of modification of the chemical structure of the oil.

1. Introduction

Castor seed plant (*Ricinus communis* L.) is the source of castor oil, a nonedible oil that has a wide variety of uses in the chemical industry [1]. This plant belongs to the family Euphorbiaceae and grows in different regions of the world. But the origin of the plant is believed to be in eastern Africa, the Abyssinia region [2, 3]. Now the plant is well distributed in tropical and warm temperate regions throughout the world. It grows wildly over a wide range of geographical regions and different climatic conditions [4].

The castor seed contain about 45–60% oil [5] containing approximately 90% ricinoleic acid [1]. The oil is unique because of its high ricinoleic acid content and the hydroxyl functionality of the ricinoleic acid gives the oil good oxidation stability, shelf life, and a point of reaction for various chemical reactions [6]. These properties make it a valuable feedstock for the chemical industries such as paints, inks, cosmetics, plastics, and lubricants [7].

The world castor seed production is increasing throughout the years, the major producers being India, China, and Brazil. India is the leading producer accounting for 75% of the total production, followed by China (12.5%) and Brazil (5.5%) [8]. The major importers of castor oil are the USA, European countries, Russia, Japan and China [9].

Castor oil can be produced from the castor seed by various methods such as mechanical pressing and solvent extraction using various solvents [1]. Oggunniyi gave an indication that mechanical pressing give yields less than the soxhlet extraction method and also that oils produced using different methods have different physicochemical properties [1]. Patel et al. [7] further showed that mechanical pressing can be improved significantly by increasing the working temperature and the leftover oils can be extracted using organic solvents. Muzenda et al. [10] studied and tried to optimize the solvent type, extraction time, and solvent-to-solid ratio. Similarly, Rani and Goud [11] conducted a comparative study of extraction using small solvent-to-solid ratio, extended time, and different solvents. Perdomo et al. [12] studied different varieties of Mexican castor seed extracted using mechanical pressing at different temperatures and a single solvent for chemical extraction. The authors also studied the differences in physicochemical characteristics of the oil obtained at

different extraction conditions. Mohammed et al. [13] worked on optimization of particle size, solvent type, and extraction time for high oil yield and also reported the difference in physicochemical characteristics of the oil extracted using different solvents.

The yield and characteristics of castor oil obtained from different parts of the world gave different results due to different geographical and climatic conditions. Most of the castor oil obtained in the world has been studied based on yield, properties, and fatty acid composition. In this work, a study on Ethiopian castor seed was carried out to determine the yield and physicochemical characteristics of the oil extracted using different mechanisms. Oil extracted mechanically and chemically (soxhlet) were characterized to qualitatively and quantitatively differentiate the effect of extraction process on the characteristics of the oil, especially on the fatty acid composition. In addition, effect of solvent polarity on fatty acid composition of the resulting oil was studied.

2. Experimental Methods

2.1 Materials

Castor seeds were obtained from an Ethiopian local market (Ehele Berenda) Addis Ababa. The seeds can be assumed to be the average of all seed species available in Ethiopia. All chemicals used in this work were of analytical grade.

2.2 Sample Preparation

The purchased seeds were de-hulled manually and the de-hulled seeds were cut into pieces using a knife in order to minimize oil loss in a size reduction machine. The cut seeds were then dried in an oven at 105 °C for 3 h. Finally, the seeds were stored inside a refrigerator at 4 °C until extraction.

2.3 Oil Extraction Study

Two kinds of oil extraction methods were used: one, soxhlet extraction and the other, mechanical extraction. Soxhlet extraction was done using three solvents, namely n-hexane, ethyl acetate, and methanol. In this method, 150 mL of the solvents and a mass of seed were used to give solid-to-solvent ratios (g/mL) of 1:9, 1:12, and 1:15. The extraction process was also studied at different durations: 1, 3, 5, 6, and 7 h. Mechanical extraction was conducted using a mechanical press via cold pressing, which was used

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for only characterization purposes (than yield determination) due to inefficiency of the machine.

2.4 Yield Calculation

The yield of the soxhlet extraction was calculated based on the extracted castor seed cake. The extracted seed cake was oven-dried at the boiling point of the solvent used for 3 h. The equation used for the calculation of the yield is as follows:

$$\text{Yield (\%)} = \frac{\text{Weight of seed before extraction} - \text{Weight of seed after extraction}}{\text{Weight of seed before extraction}} \times 100\%$$

2.5 Characterization of Oils

2.5.1 Physicochemical Characterization

Both physical and chemical characterization analyses of all types of oils obtained were conducted to compare the quality of oil extracted using different techniques. The moisture content and specific gravity were determined according to AOAC (1990) [14]. Refractive index was measured using a DR6000 Digital Refractometer (KRUS) and viscosity was measured using a vibro-viscometer.

The chemical properties, acid value, saponification value, peroxide value, and iodine value were also determined for the four types of oils obtained. The analysis was conducted according to AOAC 993.20 for iodine value, AOAC 965.33 for peroxide value, AOAC 969.17 for acid value, and AOAC 920.160 for saponification value.

2.5.2 GC-MS Analysis

2.5.2.1 Fatty Acid Methyl Ester Preparation

Initially 200 mg of the oil sample was weighed in a screw-capped tube (teflon-lined) and then 5 mL of 2 N methanolic HCl was added. The tube was closed tightly and shaken on a water bath at 60 °C for 20 min. The sample was allowed to cool and 5 mL hexane and 5 mL water was added. The tube was shaken and the hexane layer was collected after a short centrifugation. The solvent layer was washed with dilute potassium bicarbonate solution to remove excess acid and dried over anhydrous magnesium sulfate. The solvent was recovered by evaporation under reduced pressure on a rotary film evaporator. Finally, the extract was transferred to a GC vial and injected.

2.5.2.2 Fatty Acid Methyl Ester Analysis

The fatty acid methyl ester (FAME) was analyzed by a gas chromatography-mass spectrometry (GC-MS) system (Agilent 7890B) equipped with a mass spectroscopy detector (Agilent 5977B). The column HP-88 has a dimension of 30 m, 0.25 mm, 0.25 µm. The inlet temperature was 250 °C. The oven temperature was initially set at 125 °C for 1 min and then increased to 145 °C at a rate of 10 °C/min, kept at 145 °C for 3 min, increased to 153 °C at 2 °C/min, kept at 10 min, and further increased to 168 °C at 1 °C/min and kept for 1 min. Finally, the temperature in the oven was increased from 168 to 230 °C at a ramp of 40 °C/min and kept constant for 3 min. The split ratio was 50. Helium was used as a carrier gas at a column flow of 1 mL/min and a methylated sample of 1 µL was injected into the system.

3. Results and Discussion

3.1 Effect of Solid-to-Solvent Ratio on Yield

The effect of solid-to-solvent ratio on the yield of the extraction process was studied by working on the three chosen solvents at an average extraction time of 5 h. The result is shown in Fig. 1. The graph shows that as the amount of the solvent increases, the yield improves until an optimum is found. After that the yield remains constant. Hence, it can be seen that 12 mL solvent is sufficient for 1 g castor seed. Mohammed et al. [13] applied a large solid-to-solvent ratio of 1:25 compared to this study and ethanol as a solvent with an extraction time of 2.65 h, but this study showed larger yield probably due to longer extraction time and nature of seeds. Rani and Goud [11] found an optimum extraction time of 12 h, solid-to-solvent ratio of 1:9 using hexane as a solvent. In the study, the optimum ratio using hexane as a solvent was found to be 1:12 and the yield became constant after 6 h of extraction. Muzenda et al. [10] obtained a maximum extraction yield of 50.16% using solvent-to-solid ratio of 6:1 and a solvent of isopropanol. Hence, the difference may have come from a local optimum value of solid-to-solvent ratio and difference in seed variety.

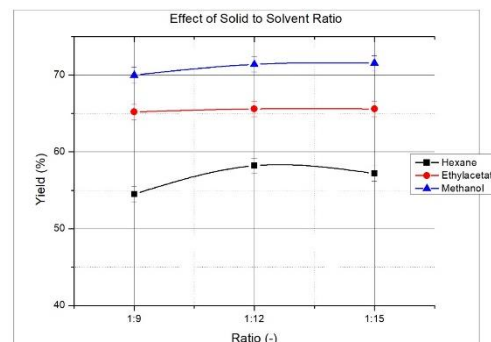


Fig. 1 Effect of solid-to-solvent ratio on yield of extraction process

3.2 Effect of Solvent Type and Extraction Time on Yield

Extraction of the oil using hexane, ethyl acetate, and methanol were carried out in a soxhlet apparatus at 1:12 solid-to-solvent ratio and different times. The result illustrates the variation of the yield for 1:12 solid-to-solvent ratio as function of time for all solvents. As can be seen from the graph (Fig. 2), the yield of the process increases gradually until all the oil in the seed is exhausted. It shows that after 6 h of extraction, the yield becomes constant and that methanol has a better extraction performance than other solvents. The maximum yields obtained using methanol, ethyl acetate, and hexane were 72.3%, 68% and 61.6%, respectively. These yields are high compared to all previously reported data and this difference may have arisen from geographical and whether conditions or seed type.

All the previous studies showed better castor oil yield when extracting with an alcohol functional group compared with other solvents [10, 11, 13]. This is also verified in this study. Although different optimum extraction times were obtained for different conditions, it was probably due to difference in the method of determining the optimum time. In this study, we determined the optimum extraction time by running the extraction in multiple consecutive hours until the yield becomes constant, and it resulted in average values of the reported times [10, 11, 13].

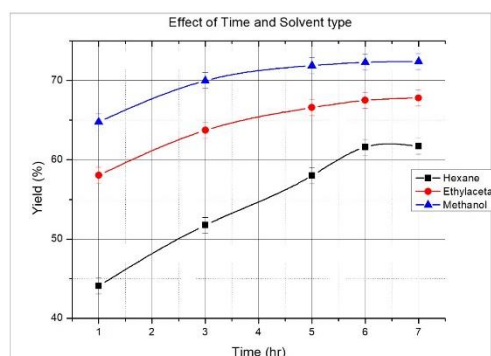


Fig. 2 Extraction of castor oil using hexane as a solvent in a Soxhlet apparatus

3.3 Characterization of Oils

3.3.1 Physicochemical Characterization

The physicochemical properties of the oils extracted using different methods were characterized to determine the effect of the extraction method on the properties of the different oils obtained. Hexane-extracted oil gave results close to the mechanically extracted oil in specific gravity, viscosity, and refractive index, which are relatively higher than those of the other solvent-extracted oils. The ethyl acetate-extracted oil had the least dense and viscous oil of the four oils, as can be seen from Table 1. The moisture content of the oils varies between 0.4% and 0.8%. Methanol and ethyl acetate have 0.8% moisture content each, which is probably due to similarity in their polarity, whereas hexane has the lowest moisture content of 0.4%. The refractive index of the ethyl acetate-extracted oil has the least value correlating with the trend of the viscosity.

Table 1 Physical properties of the oils

Method of Extraction	Solvent of Extraction	Specific Gravity	Viscosity @ 40 °C (mPa.s)	Moisture Content (%)	Refractive Index @ 20 °C
Sohxlet	Hexane	0.9485	229	0.4	1.4783
	Ethyl acetate	0.9057	137	0.8	1.4744
	Methanol	0.9285	190	0.8	1.4781
Mechanical press	-	0.9515	258	0.66	1.4786

The chemical properties of the oils also gave different results due to difference in extraction method (Table 2). High acid value was obtained from ethyl acetate-extracted oil and lowest from methanol-extracted oil. Saponification values of the different oils were not very different from each other but mechanically extracted oil showed slightly higher values. The result obtained is in good agreement with that reported by Ogunniyi [1] as the mechanically extracted oil gave higher saponification value and lower iodine value. The peroxide value for mechanically extracted oil is too low to be determined because the oil was not in contact with heat whereas hexane-extracted oil showed the highest peroxide value probably due to overheating during extraction.

Table 2 Chemical properties of the oil

Method of Extraction	Solvent of Extraction	Acid Value (mg KOH /g oil)	Saponification Value (mg KOH/g oil)	Iodine Value (%)	Peroxide Value (meq/kg)
Soxhlet	Hexane	1.23	183.26	80.44	8.47
	Ethyl acetate	1.96	198.22	72.21	4.22
	Methanol	1.00	195.41	73.71	2.11
Mechanical press	-	1.68	205.7	66.4	N.D

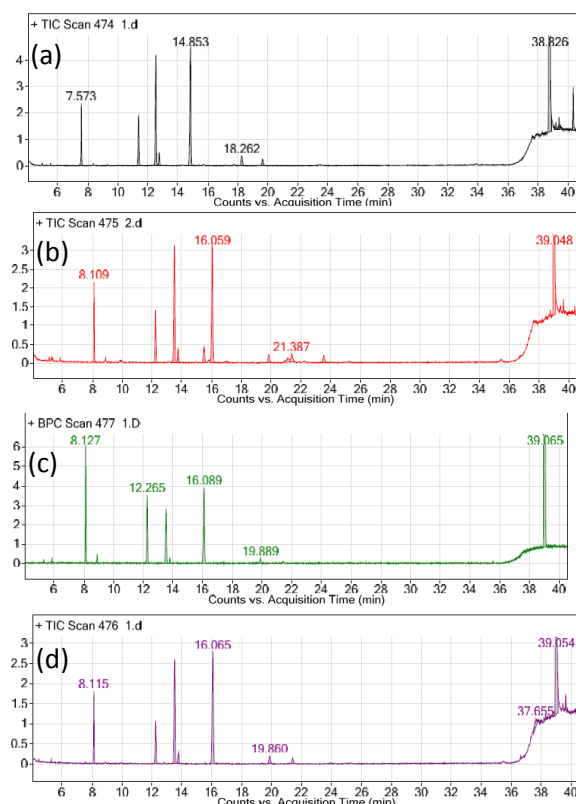


Fig. 3 a. mechanically-extracted oil chromatogram, b. hexane-extracted oil chromatogram, c. methanol-extracted oil chromatogram, d. ethyl acetate-extracted oil chromatogram

Table 3 Fatty acid composition of different oils

Method of Extraction	Soxhlet			Mechanical Press
Solvent of Extraction	Ethyl Acetate	Methanol	Hexane	-
% Palmitic Acid	1.121	1.084	1.252	1.11
% Stearic Acid	1.031	0.916	1.186	1.216
% Oleic Acid	3.180	2.930	3.551	3.6
% Linoleic Acid	3.815	3.485	4.493	4.26
% Linolenic Acid	0.321	0.316	0.345	0.416
% Eicosenoic Acid	0.246	0.2	0.741	-
% Ricinoleic Acid	90.284	91.068	88.432	90.315
Total	100	100	100	100

3.3.2 Fatty Acid Composition Result

The fatty acid composition of the oils extracted using solvent and mechanical press is shown in Table 3 and Fig. 3. The ricinoleic acid content of the castor oil is found to be an average of 90.5% for mechanically extracted and polar solvents whereas for hexane it is 88.43%. This shows a slightly less value, probably due to effect of polarity on extraction. In addition, it can be seen from Table 3 that the highest ricinoleic acid was found for extraction process using methanol, which verifies the effect of

polarity. However, the values of all other fatty acids were found to be in good agreement with those reported by Mubofu [6]. The only difference with the study of Mubofu [6] is that the oil studied in this article does not show any detectable dihydroxystearic acid in any of the oils. The mechanically extracted oil also does not show any peak for eicosenoic acid probably due to the very small content of this fatty acid. Finally, Ethiopian castor oil showed fatty acid content similar to that of other countries such as Brazil, India and Tanzania [15].

4. Conclusion

In this study, castor oil extraction and characterization were carried out using three solvents and a mechanical press. Three factors were studied in Soxhlet extraction: solid-to-solvent ratio, solvent type and time of extraction. The yield of the oil within the different factors were compared for the Soxhlet extraction method. In addition, both physical and chemical characteristics including GC-MS analysis of the oils were investigated and compared. Maximum extraction yields were obtained compared with any papers published till now, especially from the more polar solvents used for the extraction. A maximum yield of 72.3% was obtained for methanol, 68% for ethyl acetate and 61.6% for hexane. A ratio of 1 g seed/12 mL solvent was found to be optimum for the extraction process, and maximum yields were obtained at 7 h of extraction. All extracted oils were characterized for their physical and chemical properties. The fatty acid composition of the different oils was also determined via GC-MS to quantify the difference that comes with the extraction technique. The result showed that Ethiopian castor oil contains a high amount of ricinoleic acid. When using methanol as a solvent of extraction, the ricinoleic acid content reaches a maximum value of 91% due to polar interaction and also gave maximum yield. It was also noted that alcohol functional group solvents always gave higher yield than other solvents in the extraction of castor oil from castor seed. Hence, it can be concluded that for Ethiopian castor seed, a polar solvent should be used for extraction of castor oil to obtain both high yield and more pure substance suitable for further modification of the oil for industrial applications.

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