

EXTRACTION AND CHARACTERIZATION OF CASTOR SEED OIL FROM WILD *RICINUS COMMUNIS* LINN

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Abstract: Castor oil was extracted by mechanical cold pressing ($< 45^{\circ}\text{C}$) of the seeds of wild castor plant (*Ricinus communis* L.) found in wet marginal lands of Katsina, Nigeria. Oil yield was 39.43%. The virgin oil (obtained as clear, viscous, pale-yellow liquid) was further purified by filtration through sintered glass (fine mesh screen). This was to eliminate any dust, gum, or other particulates present. Physicochemical characterization of the purified oil revealed low acid value (2.07), low iodine value (84.18), low peroxide value (38.00), but relatively high specific gravity (0.959), hydroxyl (163.64) and saponification (175.31) values. These values compare favourably with ASTM standards and general specifications for industrial grade castor oil (WHC, 2012), suggesting that the oil has good industrial potential. Fourier transform infra-red (FTIR) spectroscopic analysis has confirmed the presence of major functional groups in the oil, notably hydroxyl (3414.12cm^{-1}), carbonyl (1739.85cm^{-1}), olefinic (1659.80cm^{-1}) and methylenic (2925.15cm^{-1}). Determined fatty acid composition of the oil by gas chromatography-mass spectrometry (GC-MS) shows ricinoleic acid (74.10%) as the major constituent fatty acid. Other fatty acids were linoleic (10.32%), oleic (7.55%), stearic (2.81%), palmitic (2.59%), erucic (1.70%) and eicosadienoic (0.93%). The appreciable oil yield (39.43%) from this castor seed variety, the quality, unique physicochemical characteristics of the extracted oil, and the renewable nature of castor oil as a natural resource justify the oil's commercialization and full exploitation in traditional, as well as novel areas (e.g. biopolymer and biofuel production) of industrial application.

Keywords: Castor oil; wild *Ricinus communis* Linn; variety minor; Katsina; physicochemical properties; fatty acid composition.

Introduction

The castor plant (*Ricinus communis* L.), belonging to the family Euphorbiaceae, is not only native to Africa (being of Ethiopian origin), but also widely distributed throughout the tropical, subtropical and even warm temperate regions of the world (Salunke and Desai, 1992; Ogunniyi, 2006; Salihu *et al.*, 2014). The plant is drought and pest resistant, and can be grown practically anywhere land is available. In Katsina (latitude 13°N of the Equator;

Longitude 7^o, 32'E of Greenwich Mean Time), Katsina State – Nigeria, it is commonly found in marginal lands around major drainages/water-ways notably in K/Kaura, K/Marusa, K/Durbi and K/Sauri areas, where it grows lavishly as weed. The wild *Ricinus communis* L. plants in these areas are annuals of the dwarf (1-2 metres tall) type (Weiss, 1983), while the seeds are of the small (8-10mm long; 5-6mm wide), oval-shaped and dark-chocolate coloured variety (Salihu *et al.*, 2014). These seeds come under the general classification of 'variety minor' believed to be the commonest variety in Northern Nigeria (Sule and Sani, 2008).

Castor oil, like other seed oils, is extracted from the ripe or mature seeds of the plant after sun drying, and following a sequence of seed processing operations that may include dehulling, pod or seed coat removal, winnowing, sorting, cleaning, grinding or milling, pre-heating etc. (Akpan *et al.*, 2006; Alirezalu *et al.*, 2011; CastorOil.in, 2015). Oil extraction is usually achieved by mechanical expression or solvent extraction, or both. Average oil content for all castor seed varieties is about 46-55% oil by weight (Ogunniyi, 2006) - actual yield depending on particular seed variety, geographical origin/climatic conditions, and on the oil extraction method(s) used. However, unlike castor seeds which have been widely reported as containing the toxic glycoprotein ricin, the toxic alkaloid ricinine and toxic castor bean allergen (CBA), castor oil is non-toxic (Dave, 2002; Ali *et al.*, 2005; ICOA, 2013), as these toxic ingredients are not carried along with the oil during extraction.

The versatility and tremendous utilitarian value of castor oil compared to other vegetable oils makes it invaluable. The oil owes its utilizability to its unique physicochemical nature. Chemically, castor oil consists of triglyceride molecules, each containing three esters of ricinoleic acid or ricinoleates (in place of three hydroxyl groups) on a glycerol backbone. Significantly, many researchers have reported that the oil contains up to 87-90% ricinoleic acid (Akpan *et al.*, 2006; Ogunniyi, 2006; Conceicao *et al.*, 2007), an 18-carbon hydroxylated monounsaturated fatty acid, with only about 4.2% linoleic, 3.0% oleic, 1.0% each stearic and palmitic, 0.7% dihydroxystearic, and 0.3% each linolenic and eicosanoic acids (Dave, 2002). Castor oil is the only commercial source of a hydroxylated fatty acid (ricinoleic acid). This high level of purity (by single fatty acid content) makes the oil unique among all naturally occurring fats and oils.

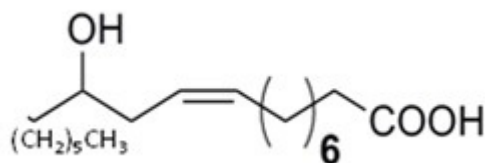


Fig. 1: Structure for ricinoleic acid

The presence of a hydroxyl group on C-12 of ricinoleic acid makes castor oil unusually polar, thus promoting hydrogen bonding. This accounts for the oil's unique combination of physical properties such as high viscosity, high specific gravity, high boiling point (313⁰C), excellent solubility in alcohols, and its ability to plasticize a wide variety of natural and synthetic resins, waxes, polymers and elastomers (Dave, 2002).

Generally, the ester linkages, the double bonds and hydroxyl groups in the oil provide reaction sites for the preparation of many useful castor oil derivatives. These characteristics, together with the oil's biodegradable and ecofriendly nature and its being a renewable resource, explain its limitless potentials. Hydroxyl functionality in the oil enables its use as biobased polyol in the polyurethane (PU) industry for the production of PU foams, elastomers, surface coating materials, adhesives and inter-penetrating polymer networks (IPNs) (Xie and Guo, 2002; Somani *et al.*, 2003). Similarly, castor oil's excellent solubility in methanol makes it ideal for biodiesel production (Noureddini *et al.*, 1998; Shrirame *et al.*, 2011; Bello and Mekanju, 2011). In the cosmetic industry, the oil's emollient and non-comedogenic nature makes it perfectly suitable in cosmetic formulations (ICOA, 2013). Turkey-red oil, a sulphonated castor oil derivative, has a marked ability to wet and disperse dyes, pigments and fillers, making it very relevant in paint, print and textile industries. Other major applications of the oil include soap manufacture, preparation of brake fluids, as a lubricant, in medicine as anti-flushing, purgative and laxative, and as food additive in the food industry (Ogunniyi, 2006; Alirezalu *et al.*, 2011).

These perspectives and prospects have made the physicochemical characterization of oil samples from different castor varieties and from different regions of the globe a topical subject of research interest. The need to obtain specific data for oil samples from particular castor varieties and particular regions cannot be overemphasized. The objective of this study was the extraction and physicochemical characterization of castor oil from wild *Ricinus communis* L. seeds found in Katsina, Nigeria, with the view of bringing out its industrial potentials.

Materials and Methods

Seed Collection and Preparation

Seeds were obtained from castor plants growing wild on wet marginal lands around Lambun Sarki, K/Marusa, Katsina. Harvested ripe castor fruits were manually cleaned and sun-dried for 4-5 days, until fruit capsules split open to discharge encased seeds. This was followed by seed pod removal and tray-winnowing to separate shells from beans (cotyledons). Castor beans were further dried (per 100g sample) at 80⁰C to constant weight for 9hrs in a hot air oven. The beans were then ground to a paste using mortar and pestle, prior to extraction.

Oil Extraction

Castor bean paste was wrapped in clean cloth and mechanically cold-pressed (below 45⁰C) using a manual machine extractor to obtain clear, viscous, pale-yellow virgin oil. 8.42kg of paste yielded 3.20 litres of the virgin oil. Percentage oil yield was evaluated using the expression (Muzenda *et al.*, 2012):

$$\% \text{ yield} = \frac{y_1 - y_2}{y_1} \times 100 \dots\dots\dots (1)$$

Where y_1 and y_2 are the weights of castor beans before and after extraction.

After settling for about 1 hr, good quality cold-pressed oil was further purified by filtration through sintered glass (fine mesh screen) in a Buchner funnel. This was to eliminate any dust, gum or other particulates present. The purified oil was kept in tightly stoppered dark bottles and stored in a refrigerator at 4⁰C.

Characterization of Oil

Physical analysis

pH of 2.30g dispersion of the oil in 15cm³ hot water was determined (after cooling to 30⁰C in a water bath) with the aid of a glass electrode pH meter (HANNA-209-209R).

Refractive index was measured at 30⁰C using a refractometer, DIGITAT REF960.4d.p. AOAC (1990) procedure was followed. Measurement of specific gravity (30⁰C) also followed AOAC (1990) procedure.

Relative viscosity of oil (in chloroform) was determined at 30⁰C from viscometric measurements using an ubbelohde dilution type viscometer with an efflux time of about 120 seconds for the solvent.

Chemical analysis

Hydroxyl (ASTM D 1957-86), acid (ISO 660:1996), saponification (ISO 3657:2002), iodine (ISO 3961:1996) and peroxide (AOCS, 2009) values of the oil were determined using indicated standard methods and procedures.

All physicochemical determinations were performed in triplicate, and an average evaluated in each case.

FTIR spectroscopic analysis

FTIR was used to characterize structural functionalities in the oil. FTIR spectrum of oil (in KBr pellets) was recorded with a Shimadzu 8400S FTIR spectrophotometer over the range 4500-350 cm^{-1} .

Fatty acid profile

The oil was also characterized in terms of its fatty acid profile. Fatty acid composition was determined after converting the fatty acids in the oil to fatty acid methyl esters (FAMES). The FAMES were prepared according to the modified ISO method (BS EN ISO 5508, 1995).

The separation of the FAMES was performed on a GC-MS QP2010 plus Shimadzu Japan instrument, comprising an AOC-20i liquid autosampler and a GC equipped with fused silica capillary column (30 x 0.25mm ID x 1 μm df) coated with 5% phenyl-, 95% dimethyl siloxane polymer. This is interfaced to a mass spectrometer at an interface temperature of 250 $^{\circ}\text{C}$.

Initial oven temperature was 70 $^{\circ}\text{C}$ (hold time 0 min), but this was increased at the rate of 10 $^{\circ}\text{C}/\text{min}$. to 280 $^{\circ}\text{C}$ (hold time 5min.). Injection temperature was 250 $^{\circ}\text{C}$, with a split ratio of 20.0. Ion-source temperature was 200 $^{\circ}\text{C}$. Helium was the carrier gas with flow rate of 1.8ml/min.

Peak identification was done by comparing peak retention times with reference standards for the individual FAMES. Retention time was defined as the time interval (min) between sample injection into the GC system and the recording of an individual peak in the chromatogram.

Results and Discussion

Determined physicochemical properties for the wild castor seed oil in this study are presented in Table 1. Percentage oil content (cold-press) for the seeds (39.43) is generally lower than yields reported for hot-press methods (Ayo *et al.*, 2012; Bhatol, 2013, Imasuen *et al.*, 2014). However, the yield of 39.43% is quite reasonable considering reported maximum yield of 45% for mechanically pressed castor oil in general (Ogunniyi, 2006). Apart from

preserving the natural properties of vegetable oils, the cold-press method is also known to yield castor oil of light colour, low impurity level and low light absorbance (Carr, 1976; Ogunniyi, 2006; Bhatol, 2013). These qualities ensure immediate end-use application of the oil even without refining.

Table 1: Physicochemical properties of wild castor seed oil

Property	Value	ASTM* Standard
pH	6.16	-
Specific gravity (30 ⁰ C)	0.959	0.957- 0.961
Refractive index (30 ⁰ C)	1.472	1.476-1.478
Relative viscosity (30 ⁰ C)	1.86	-
Acid value (mg KOH/g oil)	2.07	2 (max.)
Hydroxyl value (mg KOH/g oil)	163.64	160-168
Saponification value (mg KOH/g oil)	175.31	176 -184
Iodine value (Hanus) (gI ₂ /100g oil)	84.18	83-88
Peroxide value (ml/g oil)	38.00	-
% oil yield (cold press)	39.43	-

* American Society for Testing and Materials.

Physicochemical properties evaluated for the oil include low acid value (2.07), low iodine value (84.18), low peroxide value (38.00), but relatively high specific gravity (0.959), hydroxyl (163.64), and saponification (175.31) values. These values compare favourably with ASTM standards and general specifications for industrial grade castor oil (WHC, 2012). Low acid value and pH value of 6.16 suggest low free fatty acid (FFA) content in the oil.

Acid value is a measure of the FFA content in an oil. Iodine value on the other hand, is a measure of the oil's unsaturation level, while peroxide value is related to its oxidation level as a result of hydroperoxide formation at double bond positions. Double bond unsaturation in oils therefore is one of the most important parameters that influence lipid oxidation. In castor oil though, the double bond on C-9 of ricinoleic acid is believed to be protected against hydroperoxide formation by the hydroxyl group on C-12 (ICOA, 2013).

Low iodine value observed for the seed oil is suggestive of low unsaturation level typical of non-drying oils. This means that the oil may not be suitable as alkyd resin for paint formulation or used as vanishes (Kyari, 2007), though it could be used as lubricant or hydraulic brake fluid. Again, relatively high saponification value of 175.31 indicates that the oil has the potential to be used in the soap and cosmetics industries.

In effect, the combination of high hydroxyl value, low iodine and peroxide values observed for the seed oil largely accounts for the stability of castor oil against oxidative rancidity - the oil not turning rancid unless exposed to excessive heat.

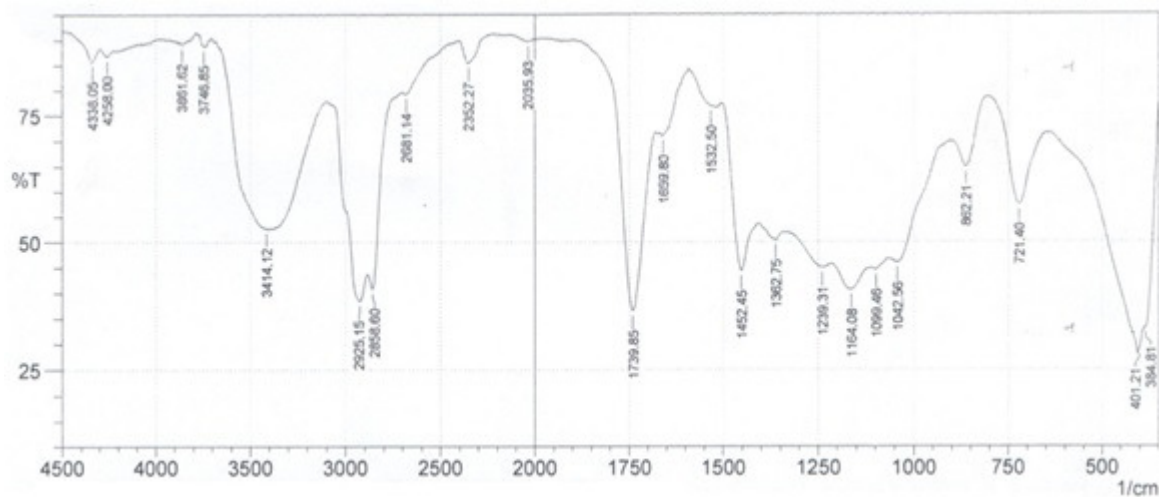


Fig. 2: FTIR spectrum for wild castor seed oil.

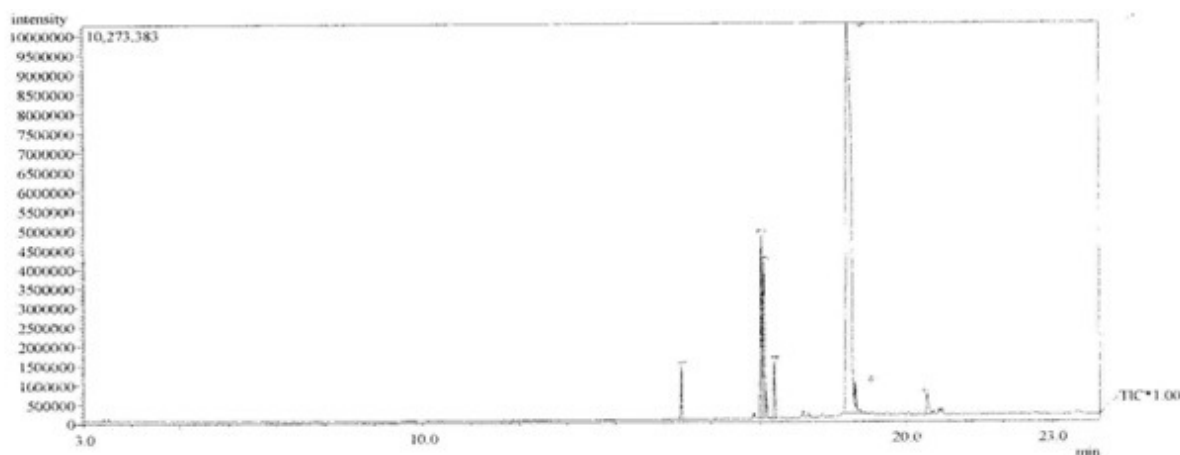


Fig. 3: GC-MS chromatogram for wild castor seed oil

FTIR spectroscopic analysis has been used to identify certain functional groups in most vegetable oils. These oils, irrespective of their origin, contain both saturated fatty acids (SFAs) and unsaturated fatty acids (UFAs). The UFAs may be monounsaturated FAs (MUFAs) or polyunsaturated FAs (PUFAs).

FTIR spectrum ($4500\text{-}350\text{cm}^{-1}$) of the wild castor seed oil (Fig.2) reveals major functional group absorption bands, notably for hydroxyl, carbonyl, olefinic, methylenic etc groups. The broad band peaking at 3414.12cm^{-1} represents O-H stretching vibration for hydrogen bonded OH groups. The large band area suggests high concentration of OH groups. This is consistent with the high hydroxyl value (163.64) determined for the oil by chemical analysis. Other well defined peaks are those for C=O stretching of triglyceride ester linkage

(1739.85 cm^{-1}), C-H stretching (CH_2) (2925.15 cm^{-1}), CH_3 bending (twisting) (1452.45 cm^{-1}), out-of-plane bending vibration of cis-disubstituted olefins (1164.08 cm^{-1}), overlapping of CH_2 rocking vibration and out-of-plane vibration of cis-disubstituted olefins (721.40 cm^{-1}), as well as C-O-C bending vibration (401.21 cm^{-1}). The shoulder at 1656.80 cm^{-1} is for C=C stretching vibration, one of two distinct absorption bands typically observed in oils containing UFAs. The other is at about 3010 cm^{-1} (Yousef *et al.*, 2001).

FA Composition

In Fig. 3, seven peaks representing seven major constituent FAMES in the oil's chemical composition are evident. Peak identification involved noting peak retention time and comparing with authentic reference standards for individual FAMES. Identified peaks were used to determine fatty acid composition, since peak area (recorded by data processing unit of the GC) is proportional to the concentration of the constituent FAME represented by the peak.

Peak 5 (retention time 18.817 min) represents ricinoleic acid (74.10%) as the major constituent fatty acid in the oil. Other fatty acids are linoleic acid (10.32%) (Peak 2), Oleic acid (7.55%) (Peak 3), stearic acid (2.81%) (Peak 4), palmitic acid (2.59%) (Peak 1), erucic acid (1.70%) (Peak 7) and eicosadienoic acid (0.93%) (Peak 6). The detailed fatty acid profile for the oil is given in Table 2, while Table 3 presents the SFA, MUFA and PUFA contents (% w/w) for the seed oil.

Table 2: Fatty acid composition of wild castor seed oil by GC-MS

Fatty Acid	Percentage
Palmitic ($\text{C}_{16:0}$)	2.59
Linoleic ($\text{C}_{18:2}$)	10.32
Oleic ($\text{C}_{18:1}$)	7.55
Stearic ($\text{C}_{18:0}$)	2.81
Ricinoleic ($\text{C}_{18:1}$)	74.10
Eicosadienoic ($\text{C}_{20:2}$)	0.93
Erucic ($\text{C}_{22:1}$)	1.70

Table 3: SFA, UFA and PUFA contents (% w/w) for wild castor seed oil.

Parameter	Value
SFA total	5.40
MUFA total	83.35
PUFA total	11.25
UFA total (MUFA + PUFA)	94.60

Ricinoleic acid content (74.10%) for the seed oil is substantially lower than values reported in literature (up to 84-95%) for most castor oil samples (Akpan *et al.*, 2006; Oggunniyi, 2006; Conceicao *et al.*, 2007), but falls within the range (70-90%) reported by Foglia *et al.* (2000) for castor oil samples in USA. Variations in percentage chemical content and composition among naturally occurring renewable resources are not uncommon, especially where there are differences in species or varieties, geographical origin, climatic conditions, soil types etc (Lochab *et al.*, 2012). Alirezalu *et al.* (2011) who studied the effect of climatic factors on the production and quality of castor oil among 10 different climatic regions in Iran, reported significant differences in physicochemical properties of the oil in the regions. Similarly, Salimon *et al.* (2010) reported differences in ricinoleic acid and UFA contents among Malaysian, Brazilian and Indian castor oil samples. In Nigeria, Bello and Anjorin (2012), and Nangbes *et al.* (2013) have reported ricinoleic acid contents of 89.50% and 83.97% for castor oil samples from Akure (Ondo state) and Pankshin (Plateau State) respectively.

SFA total for the oil sample in this study was 5.40%, while the UFA total, made up of MUFA (83.35%) and PUFA (11.25%), was 94.60%. UFA content in our sample is much higher than that for palm oil (Boyle and Anderson, 2007) and compares well with reported values for UFA-rich edible vegetable oils like sunflower, safflower, linseed and soybean oils (Chowdhury *et al.*, 2007; Kostik *et al.*, 2013).

Conclusion

This study has investigated the physicochemical properties and fatty acid composition of wild castor seed oil from Katsina, Nigeria. Among key physicochemical characteristics of this oil are high specific gravity, high saponification value, moderately high hydroxyl value, but low iodine and peroxide values. Fatty acids present in the oil as confirmed by this study

are ricinoleic, linoleic, oleic, stearic, palmitic, erucic and eicosadienoic acids. Oil composition and oil characteristics are definitely affected or influenced by oil seed variety and quality, as well as the interaction of environmental factors around the castor crop. Overall result from our study indicates good quality oil, good yield and high prospects for commercialization and industrial usage, especially in the areas of medicine, soap and cosmetics, lubricants, food additives, as well as in biopolymer and biofuel production. As non-edible oil, castor oil ought to be fully exploited to replace edible ones in this role. To this end, seed production needs to be boosted by mass cultivation of the castor crop.

Acknowledgement

The authors are grateful to the National Research Institute for Chemical Technology (NARICT) and the Department of Chemistry, Ahmadu Bello University, both in Zaria, Nigeria, for use of their research facilities.

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