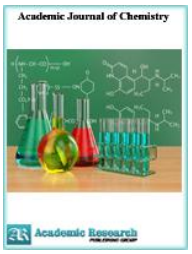

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Physico-Chemical Properties and Elements Composition of Fixed oil, Seed Extract, of *Jatropha curcus L.* [Blue Nile State (Aldamazien)]

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Abstract: *Jatropha curcus L* seed oil has been extracted by mechanical pressing**,** physicochemical parameters of the extracted oil such as moisture content, ash content, density, refractive index, kinematic viscosity, saponification value, Iodine value, peroxide value and acid value have been carried out on the seed oil using American Oil Chemist Society [1] and Association of Official Analytical Chemists [3] standard methods. The organic compounds of *Jatropha curcus L* oil have been evaluated by gas chromatography mass spectrometry technique (GC/MS), elements composition of extracted oil has been determined using inductively coupled plasma technique (ICP). The extracted oil is non-edible because all physicochemical characteristics have been found to be out of permissible limits assigned by World Health Organization [4] for edible oils. A total of 34 organic compounds of *Jatropha curcus L* seed oil have been detected, besides there have been some new compounds that have not been previously reported. The elements composition concentrations that have been found in extracted oil are Na, Mg, Ca, V, Fe, Ni, Cu, Al, As and Pb, these comply with WHO specification except the concentrations of Mg, Ni and As have been found to be 21.5, 0.1 and 2.872 ppm respectively.

Keywords: *Jatropha curcus L;* Mechanical pressing; GC/MS; ICP; Physicochemical properties of mechanical pressing.

1. Introduction

Jatropha curcas [2] belongs to the family of Euphorbiaceae [2] and has been widely known as biofuel crop [5]. Jatropha is originally native of tropical America, but now is widely common in many parts of the tropics and sub-tropics in Africa/Asia [6]. Jatropha grows in tropical and sub tropical regions, with cultivation limits at 30ºN and 35ºS. It is also grown in lower altitudes of 0-500 meters above sea level. Jatropha is not sensitive to day length (flowering is independent of latitude) and may flower at any time of the year [7]. The best soils for Jatropha are aerated sands and loams of at least 45 cm depth. Oil quality and consistency are important for producing biodiesel. The physical and chemical content of Jatropha oil can be extremely variable. Oil characteristics appear to be influenced by environment and genetic interaction, as are seed size, weight and oil content. The maturity of the fruits also can affect the fatty acid composition of the oil, and processing and storage further affect oil quality [8, 9].

However, the full potential of Jatropha has not been fully realized due to several reasons. Firstly, the growing and management of Jatropha is poorly documented. Secondly, there are fewer efforts put forward towards marketing its products. Therefore, actual or potential growers are generally reluctant to invest time and money in a crop that only has promise rather than concrete rewards [6].

Primarily, Jatropha has been well studied for its properties to serve as biodiesel in future [10]. It is well known that Jatropha oil can be used as fuel in diesel engines directly and by blending it with methanol [11]. Experimental testing of engines with Jatropha oil in Thailand has revealed satisfactory engine performance [12].

A technique to produce biodiesel with high free fatty acids contents (15% FFA) from Jatropha has been developed [13, 14]. This technique involved two-stage trans esterification process to improve methyl ester yield.

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The first stage involved the acid pretreatment process to reduce the FFA level of crude Jatropha seed oil to less than 1% , and second was the alkali base catalyzed trans esterification process which gave 90% methyl ester yield. Efforts have also been put forward by researchers to reduce cost of biofuel production from Jatropha.

Search for an alternative fuel, which should be not only sustainable, but also environmental friendly [15] Jatropha curcas is considered to be the best sustainable source.

In general, it is necessary to ensure low contamination of the oil, low acid value and low contents of phosphorus, ash and water. Crude Jatropha oil is relatively viscous, more so than rapeseed. It is characteristically low in free fatty acids, which improves its storability, though its high unsaturated oleic and linoleic acids make it prone to oxidation in storage [7]. The presence of unsaturated fatty acids (high iodine value) allows it to remain fluid at lower temperatures. Jatropha oil also has a high cetane (ignition quality) rating. The low sulfur content indicates less harmful sulfur dioxide (SO_2) exhaust emissions when the oil is used as a fuel [8].

These characteristics make the oil highly suitable for producing biodiesel. The objectives of this project are extraction of Jatropha oil and characterization by GC/MS and ICP, so as to determine its physicochemical characteristics and suitably to be used as bio-fuel.

2. Materials and methods

2.1. Materials

Hydrochloric acid (HCl), sodium hydroxide (NaOH), potassium hydroxide (KOH), iodobromine (IBr), sodium thiosulphate ($Na_2S_2O_3$), potassium iodide [16], and acetic acid (CH_3COOH). Other chemicals used are of analytical reagent grade (AR), they are obtained from Sigma Chemical Co. (St. Louis, MO).

2.2. Methods

2.2.1. Sample Collection

The *Jatropha curcas L.* seeds have been collected from Blue Nile State (Admazien area). Taxonomic authentication of the plant has been carried out in the National Center for Research (NCR) – Medicinal and Aromatic Plants Research Institute in Sudan.

2.2.2. Oil Extraction

The *Jatropha curcas L* seeds oil have been Extracted by mechanical pressing according to the method that already reported [3].

2.2.3. Physicochemical Properties of *Jatropha Curcas L* Oil

Different physicochemical parameters such as moisture content, ash content, density, refractive index, kinematic viscosity, saponification value, iodine value, peroxide value and acid value have been all carried out on the seed oil taking in consideration the methods reported [17, 18]:

2.2.3.1. Moisture and Ash Content Measurement

Jatropha oil (45.8g) has been taken in a crucible and the moisture content was determined by weight loss of the sample on drying at 105 °C for 3 h , then, the dry material was ashed in the muffle furnace at 550°C for 3 h until constant weight obtained and the ash content was calculated.

2.2.3.2. Density Measurement

Density of oil sample were measured by an R.D bottle with a capacity of 10 mL following the already reported mthod [19].

2.2.3.3. Refractive Index Measurement

The refractive index of the oil at room temperature has been determined using Carl Zeiss 110849. The oil drop has been placed on the slide and directed towards a source of light. It was then observed through the lens after adjustment to give a semi- circle on the glass prism in the refractometer, taking in consideration reported methods [20, 21]. The reading has been then taken.

2.2.3.4. Viscosity Measurement

The viscosity of oil sample has been measured by an Ostwald Viscometer techniconominal constant 0.05 Cs/c, ASTM, D 445 England. The flow time of oil samples have been recorded using a stop watch, as reported mthod [22].

2.2.3.5. Saponification Value Measurement

The saponification value has been determined, according to reported method [21], by taking 1.0 g of oil sample in a conical flask to which 15 mL 1 N KOH and 10 mL of distilled water has been added and heated under a reserved condenser for 30–40 min to ensure that the sample was fully dissolved. After this sample was cooled, phenolphthalein was added and titrated with 0.5 M of HCl until a pink endpoint was reached. A blank was determined with the same time conditions.

2.2.3.6. Iodine Value [17] Measurement

The oil sample (2g) was treated with an excess of iodobromine (IBr) in glacial acetic acid. Unreacted iodobromine was reacted with potassium iodide which converts it to iodine. The iodine concentration was then

$$\text{IV} \quad (b-v) \times N \times 126.9 \times 100/w \times 1000$$

determined by titration with standard sodium thiosulphate, as reported method [21].
where b is the quantity of sodium thiosulphate used for blank, v is the quantity of thiosulphate for sample, N is the normality of thiosulphate solution, w is the wt of the oil sample and 126.9 is the molecular weight of iodine [19].

2.2.3.7. Peroxide value (PV) Measurement

The oil sample (2g) was dissolved in acetic acid then chloroform and saturated KI mixture are added to the sample and the amount of iodine liberated from KI by the oxidative action of peroxides present in the oil is determined by titration with standard sodium thiosulphate using starch solution as an indicator. Titration was also performed for blanks, as reported method [4].

$$\text{PV(meq/kg oil)} \quad (S-B) \times W \times N$$

where B is the volume of sodium thiosulphate used for blank, W is the weight of sample, S is the volume of sodium thiosulphate consumed by the sample oil and N is the normality of standard sodium thiosulphate [18].

2.2.3.8. Acid Alue Measurement

Jatropha oil (0.5 mg) was accurately weighted and dissolved in 10 ml of 95 % ethanol and 2-3 drops of phenolphthalein indicator was added. The free acid was then titrated with standard 0.1 N aqueous sodium hydroxide solution by adding the alkali drop-wise at a uniform rate of about 30 drops per minute. The content of the flask was continuously agitated. The primary manifestation of the red coloration that did not fade within 10 seconds was considered the end point. Afterward, the acid value was determined using the following equation

$$\text{Acid value} = \frac{5.61 \text{ x (number of mL of 0.1 N NaOH))}}{\text{Weight of sample in gram}}$$

2.2.4. Gas Chromatography Mass Spectrometry (GC/MS) Analysis

The GC/MS analysis of Jatropha oil was performed on a GC-MS equipment (Thermo Scientific Co. Thermo GC-TRACE ultra ver.: 5.0, Thermo MS DSQ II. Experimental conditions of GC-MS system were as follows: TR 5-MS capillary standard non-polar column, dimension: 30Mts, ID: 0.25 mm, Film thickness: 0.25µm. Flow rate of mobile phase (carrier gas: He) was set at 1.0 ml/min. In the gas chromatography part, temperature program (oven temperature) was 75°C raised to 250°C at a rise of 5°C/min, and held for 30min. The injection volume was 1 µl and sample was injected in split less mode. Finally the sample was run fully at a range of 50–650 m/z and the results were compared by using Wiley Spectral library search program [16].

2.2.5. Identification of Elementary Composition of Jatropha Oil by Using ICP Technique

a. Instrumentation

The analytical determination of metals was carried out by ICP (Inductively Coupled Plasma): ELAN 9000 (Perkin Elmer Sciex Instrument, Concord, Ontario, Canada).

b. Calibration

The ICP calibration was carried out by external calibration with the blank solution and three working standard solutions (10, 20 and 30 µg/L) for all elements.

c. Sample preparation

A 0.5 mg of oil was weighed and transferred into a clean gosh crucible, then the peel was burned using muffle furnace at 550 ^{o}C for 2 hours. The ash peel was transferred by 10 ml concentrated hydrochloric acid into 100 mL volumetric flask, finally the volume was completed to the mark by deionized water [23].

3. Results and Discussion

The percentage yield of *Jatropha curcas L* seeds oil was found to be 28.9%($_{w/w}$%). Physicochemical properties of *Jatropha curcas L* seeds oil are shown in Table 1.

Table 1. Physicochemical characteristics of extracted *jatropha curcus L* oil

Test No	Characteristic	Unit	Value	WHO Specification for edible oils
1	Density	g/mL	0.91085 (±0.08)	Min 0.94
2	Kinematic viscosity	Millipoise	48.35 (±0.15)	83.12 – 120
3	Refractive Index	-	1.47 (±0.1)	1.4590 to 1.4670
4	Saponification value	-	230.49 (±1.2)	max125
5	Acid Value	-	28.1(±0.2)	Max10
7	Iodine Value	-	58.4(±1.5)	20 - 50
8	Peroxide value	-	2.0(±0.09)	Max 1.83
9	Moisture Content	% w/w	0.515 (±0.07)	Max 0.2
10	Ash Content	% w/w	0.09 (±0.01)	Max 0.05

Physicochemical properties of *Jatropha curcas L* have been found to be out of assigned permissible limits by world Health Organization [24] for edible oils. The use n-edible seed oils or the use of direct bioconversions from waste is now considered a major alternative for fossil petrodiesel [25]. In principle any oil rich plant seed can be used as a source of biodiesel.

The Gas chromatography chromatogram of *Jatropha curcas L* oil and organic compounds are shown in Fig 1 and Table 2 respectively.

Fig-1. Gas chromatography chromatogram of *Jatropha Curcas L* oil

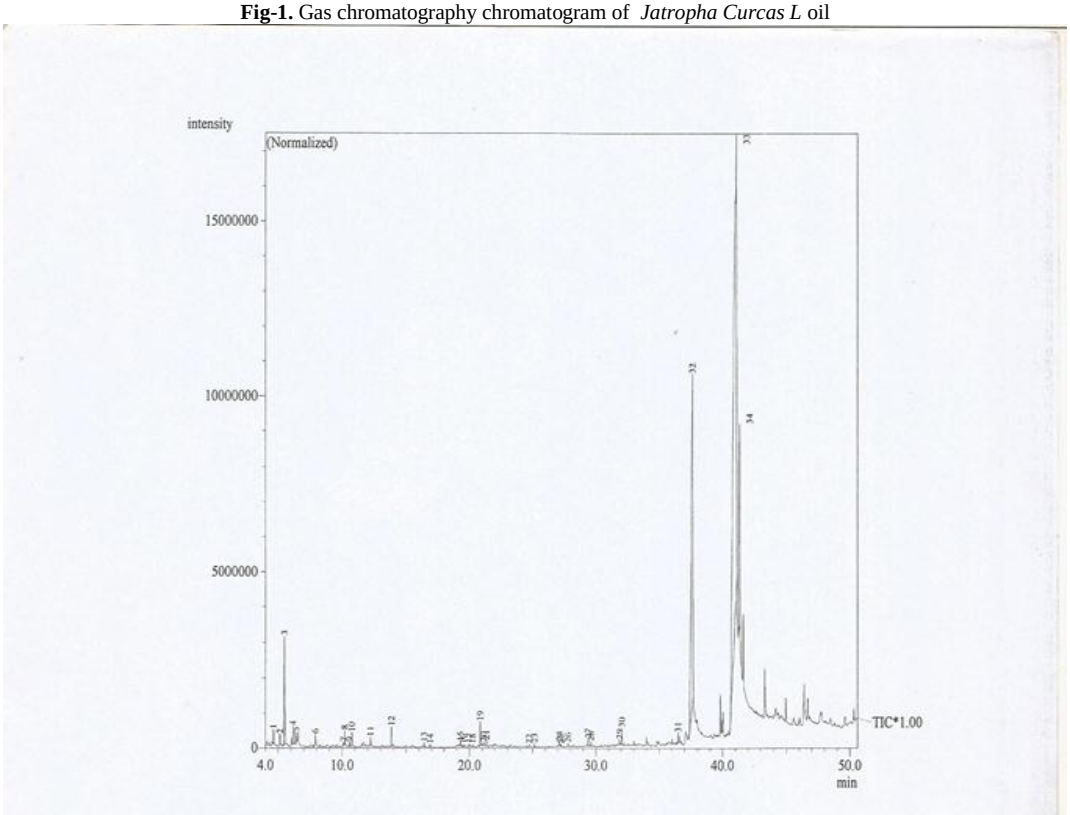

Table-2. Organic compounds of *Jatropha curcas L* oil

Peak No	Compound Name	M.W	M.formula	R.Time	Area%
1	2-Methyl,2-pentanol	102	$H_6C_{14}O$	4.633	0.33
2	3-Methyl,3-pentanol	102	$C_6H_{14}O$	5.136	0.22
3	Methylbenzene	92	C_7H_8	5.486	3.02
4	2-Hexanone	100	$C_6H_{12}O$	6.178	0.71
5	Chlorobenzene	112	C_6H_5CL	6.396	0.22
6	ChloroBenzene	112	C_6H_5CL	7.908	0.23
7	TetraHydro-2,2-Dimethyl Furan	100	$C_6H_{12}O$	10.134	0.05
8	2-methyl,2-pentanethiol	118	C_6H1_4S	10.216	0.26
9	Bicyclo{3.1.0}hex-2-ene,2-methyl-5-(1-methylethyl)	136	$C_{10}H_{16}$	10.578	0.07
10	2,2-Dimethyl-1-pentanol	116	$C_7H_{16}O$	10.768	0.31
11	3-Hexen-2-one	98	$C_6H_{10}O$	12.215	0.21
12	Methyl(1-methylethyl)Benzene	134	$C_{10}H_{14}$	13.867	0.40

13	Nonanal	142	$C_9H_{18}O$	16.426	0.10
14	p-methyl-6-en-2-ylmethyl ether	168	$C_{11}H_{20}O$	16.880	0.05
15	3-hexadecyloxycarbonyl-5-(2-hydroxy ether)-4-methylImidazolium ion	409	$C_{24}H_{25}N_2O_3$	19.289	0.07
16	2,2,4,4-tetramethylcyclobutan-1-ol	128	$C_8H_{16}O$	19.480	0.04
17	2,2-dimethyl 1-pentanol	116	$C_7H_{16}O$	19.967	0.05
18	2-ethyl-2-methylButanoic acid	130	$C_7H_{14}O_2$	20.255	0.04
19	2,5-Cyclohexadiene-1,4-dione,2-methyl-5-(1-methylethyl)	164	$C_{10}H_{12}O_2$	20.857	0.60
20	2-Decenal,(E)	154	$C_{10}H_{18}O$	21.110	0.05
21	5-ethyl-2,4-dimethyl-2-heptene	154	$C_{11}H_{22}$	21.325	0.10
22	Tetradecane	198	$C_{14}H_{30}$	24.729	0.03
23	1,4-methanoazulene,decahydro-4,8,8trimethyl-9-methylene,{1S(1.alpha.,3abeta.,4.alpha.,8a.beta.)}	204	$C_{15}H_{24}$	25.139	0.07
24	Eicosane	282	C_20H_{42}	27.128	0.08
25	Nonadecane	268	$C_{19}H_{40}$	27.235	0.03
26	2,4-bis(1,1-dimethylethyl)-phenol	206	$C_{14}H_{22}O$	27.789	0.07
27	2-iodo-3-methyl butane	198	$C_5H_{11}I$	29.390	0.21
28	Hexadecane	226	$C_{16}H_{34}$	29.612	0.09
29	Heptadecane	240	$C_{17}H_{36}$	31.867	0.12
30	Tetratriacontane	478	$C_{34}H_{70}$	32.495	0.40
31	Eicosane	282	$C_{20}H_{42}$	36.495	0.22
32	2,6-dihexadecanoate-1-(+)-ascorbic acid	652	$C_{38}H_{68}O_8$	37.626	22.42
33	9-Octadecenoic acid,(E)	282	$C_{18}H_{34}O_2$	41.073	63.43
34	6-Octadecenoic acid,(Z)	282	$C_{18}H_{34}O_2$	41.331	5.67

The data obtained from GC/MS (see Fig 1 and Table 2) has revealed the presence of 34 compounds of *Jatropha Curcas L* oil. Besides there have been some new compounds that have not been previously reported. The most abundant compounds (see Table 2) are Compound No 33 (E) 9-Octadecenoic acid, (32) 2,6-dihexadecanoate-1-(+)-ascorbic acid, (34) 6-Octadecenoic acid,(Z), and (3) Methylbenzene, a very toxic compound that confirms the non-edibility of Jatropha *curcas L*. A total of 10 free acids were detected that confirm using as biodiesel and justify its use [21]. The elements composition of Jatropha oil is shown in Table 3.

Table-3. The elements composition of extracted *jatropha curcus L* oil

Element	Concentration (ppm)	WHO Specification for edible oils (ppm)
Na	1.097 (±0.01)	11.48
Mg	21.594 (±0.02)	20.80
Ca	31.869 (±0.01)	1030
V	0.165 (±0.03)	0.9
Fe	3.515 (±0.02)	4.49
Ni	0.1 (±0.01)	0.07
Cu	0.1 (±0.02)	0.24
Al	3.895 (±0.05)	100
As	2.872 (±0.02)	1
Pb	0.1 (±0.01)	0.1

Table 3 has revealed that the highest metals concentration in *Jatropha curcus L* oil are Mg, Ca, Al, Na and Fe. The concentrations of heavy metals are very low except the concentration of Iron is 3.515 ppm. The high ash content of *Jatropha curcus L* oil is attributed to the concentrations of detected elements. Also it can be concluded that elements concentration of Mg, Ni and As are out of WHO specification.

4. Conclusion

In conclusion**,** the extracted essential oil from *Jatropha curcus L* seed has been found to be non-edible because all physicochemical characteristics do not comply with WHO specification for edible oils although the concentration of the elements has been found to be within the limits , besides some of the reported compounds by GC/MS have a high toxicity such as methylbenzene, chlorobenzene and 2,4-bis(1,1-dimethylethyl)-phenol. All physicochemical properties results confirm that the *Jatropha Curcus L* seed oil could be a source for the production of biodiesel because the oil content is very high (28.89%). Having found about 10 free fatty acids that confirm its use.

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