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## PHYSICOCHEMICAL PROPERTIES OF CASTOR SEED (*RICINUS COMMUNIS*) OIL EXTRACTED BY ETHER AND COLD PRESSING METHOD

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### ABSTRACT

*Ricinus communis* (Castor plant) grows abundantly in rural areas of Kano State of Nigeria and is highly underutilized, this work was carried out to determine the physicochemical properties of its extracted oil. The oil was extracted using ether and cold pressing by pneumatic press machine. The physicochemical properties of the extracted oil were carried out using standard methods. Results were statistically analyzed with MINITAB soft ware using one way ANOVA and Tukey method at  $p < 0.05$  and 95% confidence limit. The physical parameters of the ether extracted and cold pressed oil were as follows: Percentage Yield (33.67 and 28.80 respectively), Density (0.946 and 0.953  $g/cm^3$  respectively), Refractive index (1.4681 and 1.4621  $n_D$  respectively) Viscosity (120.82 and 137.823  $mpa*s$  respectively) Smoke point ( $169.000 \pm 1.00$  and  $176.333 \pm 0.577$  °C respectively), Flash point ( $228.333 \pm 0.577$  and  $235.667 \pm 1.55$  °C respectively) and fire point ( $238.667 \pm 0.577$  °C each.. The chemical parameters of the ether extracts and cold pressed oil were as follows: Moisture content ( $0.240 \pm 0.010$  % and  $2.50 \pm 0.100$  % respectively) pH ( $5.77 \pm 0.07$  and  $6.23 \pm 0.07$  respectively), acid value ( $1.009 \pm 0.056$  and  $1.253 \pm 0.032$  mg KOH/g respectively) The peroxide value, saponification value and iodine were  $3.493 \pm 0.023$ ,  $181.333 \pm 0.577$  mE (mg KOH/g, and  $83.667 \pm 1.155$  wj's g/100g for cold pressed oil and  $3.120 \pm 0.040$ ,  $176.000 \pm 1.000$  and  $81.333 \pm 0.577$  ether extracts oil respectively.

### INTRODUCTION

Castor plant, *Ricinus communis*, is a species of flowering plant in the spurge family, *Euphorbiaceae*. It is commonly known in Hausa as *Zurman*. Its seed is the castor bean which, despite its name, is not a true bean. Castor plant is indigenous to the southeastern Mediterranean Basin, Eastern Africa, and India, but is widespread throughout tropical regions (Phillips and Martyn, 1999). The seeds are oblong and light brown, mottled with dark brown spots. Global castor seed production is around one million tons per year. Leading producing areas are India (with over 60 percent of the global yield), China, and Brazil

(Sims and Frey, 2005). The seeds are extremely poisonous to both humans and animals and should be kept away from young children and livestock. The seeds contain the deadly toxin ricin. Castor Bean, along with Rosary Pea (*Abrus precatorius*), are two of the most poisonous plants in the World (Sims and Frey, 2005). Castor oil is pale amber viscous liquid derived from the seeds of the plant *Ricinus Communis*, sometime known as *ricinus oil*. Castor oil is one of the few naturally occurring glycerides that approach being a pure compound, since the fatty acid portion is nearly nine-tenths *ricinoleic* (Marter, 1981).

A crude Castor oil is a pale straw colour but turns colourless or slightly yellowish after refining and bleaching. The crude oil has distinct odour, but it can easily be deodorized in the refining process. Like any other vegetable oils and animal fats, it is a triglyceride, which chemically is a glycerol molecule with each of its three hydroxyl group esterified with a long chain fatty acid. (Sims and Frey, 2004).

Various techniques such as mechanical extraction, solvent extraction, traditional extraction and super critical fluid extraction are used to obtain the oil from the seeds. The solvent extraction has become the most popular method of extraction of oil because of its high percentage of oil recovery from seeds. Solvent extraction bridges the gap between mechanical extraction which produces oil with high turbidity metal and water content and supercritical fluid extraction which is very expensive to build and maintain its facilities. (Weiss, 2000).

Alizeralu *et al.*, (2011) reported that castor oil obtained by pressing method has low acid value, low iodine value, lighter color and slightly higher saponification value compared to the solvent-extracted oil. The purpose of this research was to compare the phytochemical content and antibacterial properties of castor seed oil extracted by ether and cold pressing methods.

## 1. MATERIALS AND METHODS

### 2.1 Sample Collection

The castor plant seeds used in this study was the white variety of the *Ricinus communis*, verified by an agronomist from Faculty of Agriculture and Agricultural Technology, Kano University of Science and Technology, Wudil. The sample was collected from a farm at Gaya town, Gaya Local Government area of Kano State, Nigeria. The sample was grinded into powdered form using pestle and mortar and then transported to Food analysis/Processing Laboratories of the Department of Food Science and Technology, Kano University of Science and Technology, Wudil, where the analysis was carried out.

### 2.2 Extraction of castor seed oil

Ether extraction was carried out using Soxhlet extractor as described by the Official Methods of Analysis (AOAC, 1990). Exactly 300 g of dry

ground seeds was oven-dried at 105°C for 1 hour. The oil was extracted using petroleum ether (Bp 40-60 °C) as solvent. The sample was refluxed for 6 hours in mild temperature (40-60°C) in a Soxhlet extractor. The solvent was evaporated using water bath at 80 °C. The extracted oil was kept in an oven at 100°C for 30 min before accurately weighing to determine oil yield. The extraction was repeated many times until reasonable amount was gathered for the research. The extracted seed oil was kept in a closed container and stored in a refrigerator at 4°C for subsequent physicochemical (AOAC,1990).

Cold pressing was determined using a modified method of Edgar Cayce's Association for Research and Enlightenment, Inc (A.R.E, 2004). The seed was dehulled and boiled for 30 minutes to detoxify it. It was then sun dried and fried so as to soften it and prepare it for easy extraction the oil. The seed was pressed in a large powerful screw press, and the oily matter which flows out was mixed with an equal proportion of water, and boiled to purify it from mucilaginous and albuminous matter. After boiling for about an hour, it was allowed to cool, the water layer was drawn off in a separating funnel and the oil was transferred to a clean container. The oil was allowed to stand for about eight hours, bleaching in the sun, after which it was weighed to determine percentage yield and stored in a refrigerator at 4°C for subsequent physicochemical, phytochemicals and antimicrobial analysis.

### 2.3 Physical Analysis

#### 2.3.1 Smoke point

Exactly 10 ml of the castor oil was transferred into a metal container and heated at a controlled rate in an oven. The temperature (°C) at which a thin continuous stream of bluish smoke is first observed was recorded as the smoke point (Onwuka, 2005).

#### 2.3.2 The flash point

Exactly 10 ml of the castor oil was transferred into a metal container and heated at a controlled rate, with a flame being passed over the surface of the sample at regular intervals. The temperature (°C) at which a flash appears at any point on the surface of the sample due to the ignition of volatile gaseous products was recorded and is called "flash point" (Onwuka, 2005).

### 2.3.3 The fire point

Exactly 10 ml of the castor oil was transferred into a metal container and heated at a controlled rate, with a flame being passed over the surface of the sample at regular intervals. The temperature ( $^{\circ}\text{C}$ ) at which evolution of volatiles due to the thermal decomposition of the lipids proceeds so quickly that continuous combustion occurs (a fire), is called "fire point". The result was tabulated (Onwuka, 2005).

### 2.3.4 Refractive index

This was determined according to the method described by Onwuka, (2005) using Hanna refractometer.

### 2.3.5 Specific gravity

This was determined according to the method described by Onwuka, (2005) using pycnometer bottle.

### 2.3.6 Determination of viscosity.

This was determined according to the method described by Onwuka, (2005). The viscosity of the extracted oil was determined using viscometer model NDJ-8S Digital LCD at  $30 \pm 0.1^{\circ}\text{C}$  using spindle number 2 at speed of 12rpm, reading was taken after 2 minutes of rotation.

## 2.4 Chemical Analysis

The chemical properties which includes Moisture content, iodine value (IV), acid value (AV), free fatty acid (FFA), peroxide value (PV), saponification value (SV) and unsaponifiable matter was determined according to Official Methods of Analysis, (AOAC,1990)

### 2.4.1 Moisture content

A clean flat dish made of platinum was dried in an oven and cooled in a dessicator and weighed as ( $W_1$ ). Exactly 5 ml of the oil sample was weighed into the dish ( $W_2$ ). The dish and its content was transferred into an air oven at  $105^{\circ}\text{C}$  to dry for about 3 hours and weighed. The dish was returned to the oven for half an hour and again cooled in the dessicator and weighed. The process was repeated until constant weight was obtained and recorded as ( $W_3$ ), (AOAC,1990)

**Calculation:** % Moisture =  $(W_2 - W_3) / (W_2 - W_1) \times 100$

### 2.4.2 pH determination

Exactly 100ml of each oil sample was placed in 250ml glass beaker. The pH was determined using a standardized (with buffer of pH 4.0 and 7.0) pH meter Model PHS-3C Onwuka, (2005).

### 2.4.3 Acid value and % free fatty acid

Exactly 25 ml diethyl ether was mixed with 25 ml alcohol and 1 ml of 1% phenolphthalein. This was neutralized with 0.1 M NaOH. Then 5g of the oil was dissolved in the neutralized solvent and then titrated with 0.1M NaOH solution shaking constantly until a pink colour, which persists for 15 seconds, was obtained.

**Calculation:** Acid value =  $X \text{ mls} \times 5.61/w$

Where: X = Volume of 0.1 M NaOH used in titration

W = Weight of sample

taken

The FFA was calculated as oleic acid; % FFA (as Oleic)  $\times 1.99 =$  Acid value, in which case the FFA = acid value/2

### 2.4.4 Saponification value

Exactly 2g oil was weighed into a conical flask and 25ml alcoholic KOH solution was added into the flask. A reflux condenser was attached to the flask. The mixture was heated in boiling water for one hour with frequent shaking. There after 1ml of 1% phenolphthalein was added and titrated hot with standard 0.5 N HCl (a ml). End point was colourless. A blank was determined (b mill)

**Calculation:**  $SV = (b - a) \times 28.05 / Wt$  (g) of sample.

### 2.4.5 Iodine value

Exactly 0.5g of the oil was accurately weighed into a glass stoppered bottle (250ml), and 10ml  $\text{CCl}_4$  was added to dissolve the oil. 20mls  $\text{I}_2$  solution was added and stoppered and then allow to stand in subdued light for 30 minutes. Exactly 15ml KI solution (10%) and 100ml water was added and titrated with 0.1M  $\text{Na}_2\text{S}_2\text{O}_3$  using starch indicator. A blank determination was carried out commencing with 10ml of  $\text{CCl}_4$ .

**Calculation:** Iodine Value =  $(b - a) \times N \times 12.96 / Wt$  (g) of sample.

Where: b = Blank titration and a = Test titration

### 2.4.6 Peroxide value

One gram of the oil was weighed into a clean dry boiling tube. One gram powdered potassium iodide and 20ml solvent mixture were added and the solution was placed in a boiling water bath for 60 seconds. The content was poured into a titration flask containing 20ml 10% potassium iodide solution. The Tube was washed twice with 25ml portions of water and added the washings to the titration flask. The mixture was titrated with 0.002M thiosulphate using starch as indicator.

Peroxide value =  $2 \text{ vmEq/kg} = (\text{Titre} - \text{blank}) \times (\text{molarity of thiosulphate} \times 100) / \text{weight of sample}$ .

### 3.0 RESULTS

#### 3.1 Physical Properties and Percentage Yield of the Extracted Oils from the Castor Seed

According to this research finding, the percentage oil yield of ether extracts of the seed oil was observed to be 33.7 % which is highly significant ( $p < 0.05$ ) compared to the value obtained by cold press method (28.08%).

Several parameters such as particle size, type and volume of solvent, operating temperature, and extraction time were found to affect the percentage yield of oil from the seed. The physical properties of the castor seed oil obtained by ether extraction method also showed that it has lower viscosity with significant difference ( $p < 0.05$ ) and refractive index, very clear with no trace of suspended particles. The cold pressed oil is more viscous with slight presence of suspended particle and has higher smoke and flash points. The ether extracted oil with low viscosity value indicated that it is light and so probably highly unsaturated; the high viscosity value in cold pressed oil might be as a result of the suspended particles still present in the crude oil sample. However both the oils from the two extraction methods possess the same fire point. Both ether extracted oil and the cold pressed oil were pale yellow in colour, and liquid at room temperature (27°C) and showed no solid fat content at 0°C.

**Table 3.1:** Physical Properties and Percentage Yield of the Extracted Oils From the Castor Seed

Sample	% Yield	Density (g/cm <sup>3</sup> )	Refractive Index (nD)	Viscosity (mpa*s)	Smoke Point (°C)	Flash Point (°C)	Fire Point (°C)
EEO	33.67 ± 0.67 <sup>a</sup>	0.946 ± 0.002 <sup>a</sup>	1.468 ± 0.002 <sup>a</sup>	120.83 ± 1.01 <sup>a</sup>	169.00 ± 1.00 <sup>a</sup>	228.33 ± 0.58 <sup>a</sup>	233.67 ± 0.58 <sup>a</sup>
CPO	28.08 ± 0.33 <sup>b</sup>	0.953 ± 0.000 <sup>b</sup>	1.462 ± 0.006 <sup>a</sup>	137.82 ± 1.19 <sup>b</sup>	176.33 ± 0.58 <sup>b</sup>	235.67 ± 1.55 <sup>b</sup>	233.67 ± 0.58 <sup>a</sup>

Values on the Same Column, with different Superscript are Significantly Different ( $P \leq 0.0005$ ).

KEY: (EEO) = Ether extracted oil, (CPO) = Cold pressed oil

#### 3.2 Chemical Properties of the Extracted Oils from the Castor Seed

The chemical analysis of the ether extracted castor seed oil showed that it has lower moisture content, lower acid value, Free Fatty acid and lower pH with significant difference

( $p < 0.05$ ) compared to the cold pressed castor seed oil. The Saponification value, Peroxide and Iodine values were also found to be lower in the ether extracted castor seed oil with significant difference ( $p < 0.05$ ) compared to the cold pressed castor seed oil.

**Table 3.2 Chemical Properties of the Extracted Oils From the Castor Seed**

Sample	% Moisture	pH (at 20°C)	Acid Value (mg/g oleic)	% Free Fatty Acid	Peroxide Value (meq/g)	Saponification Value (mg KOH/g)	Iodine Value wij's g/100g
EEO	0.240 ± 0.01 <sup>a</sup>	5.77 ± 0.07 <sup>a</sup>	1.01 ± 0.06 <sup>a</sup>	0.507 ± 0.023 <sup>a</sup>	3.120 ± 0.040 <sup>a</sup>	181.00 ± 1.00 <sup>a</sup>	83.333 ± 0.577 <sup>a</sup>
CPO	2.50 ± 0.10 <sup>b</sup>	6.23 ± 0.07 <sup>b</sup>	1.25 ± 0.03 <sup>b</sup>	0.629 ± 1.016 <sup>b</sup>	3.493 ± 0.023 <sup>b</sup>	176.33 ± 0.58 <sup>b</sup>	81.667 ± 1.155 <sup>b</sup>

Values on the Same Column, with different Superscript are Significantly Different ( $P \leq 0.0005$ ).

KEY: (EEO) = Ether extracted oil, (CPO) = Cold pressed oil

#### 4.0 DISCUSSION

According to this research, the percentage yield of ether extracts produced higher yield of 33.67% which significantly difference at  $p < 0.05$  compared to the yield of 28.08% obtained by cold press method (Table 4.1). The variation may be attributed to the effect of temperature and water activity involved in the processing methods. A study conducted by Sefah (2006) indicated that high moisture content reduces oil flow possibly because the structures of the finely milled particles have been altered (high aggregation) and also moisture lubricates the pulp during pressing.

Another study by Sefah (2006) revealed that temperature plays an active role in the seed treatment for mechanical extraction and ensures an effective solvent process by heating the solvent which hastens the extraction process. At the right temperature and moisture content, the individual oil droplets unite to form a continuous phase and flow out maximizing oil yield. Sefah, (2006) also indicated that petroleum-derived product such as petroleum ether has been extensively used as solvent for the oil extraction from oilseeds because of its low vapourisation temperature (boiling point 63 – 69°C), high stability, low corrosiveness, low greasy residual effect, and better aroma and flavour productivity for the milled products.

The result showed that the two extracted oils are less dense than water and therefore can comfortably be used as antibiotic, for easy flow and spread easily on the skin. However, the cold pressed oil was found to be denser ( $0.953 \text{ g/cm}^3$ ) compared to the ether extracted oil ( $0.946 \text{ g/cm}^3$ ). The difference in the density of the extracted oils may be as a result of suspended particles still present in cold pressed oil. Both the values fall slightly below the specified range of  $0.957\text{-}0.968 \text{ g/cm}^3$  by ASTM (2016).

The result for the refractive index and viscosity shows that the two oils differ significantly at  $p < 0.005$  and 95% confidence limit. The ether extracted oil was found to be more viscous and higher refractive index (Table 4.1) with values of  $1.4681 (n_D)$  and  $137.823 (\text{mpa}\cdot\text{s})$  compared to the values of  $1.4621 n_D$  and  $120.834(\text{mpa}\cdot\text{s})$ . According to Codex Alimentarius (1999), castor seed oil mostly have refractive index values within the

acceptable range of  $1.4677$  to  $1.4707 n_D$  (Nourrechni et al. 1992) stated that viscosity increased with the molecular weight and decreased with increasing unsaturated level and high temperature therefore the repeated boiling processes might have contributed to the low viscosity of the cold pressed oil.

Smoke point and flash point of the ether extracted oil were found to be  $169.000 \pm 1.00^\circ\text{C}$  and  $228.333 \pm 0.577^\circ\text{C}$  and was found to be lower than the values  $176.333 \pm 0.577^\circ\text{C}$  and  $235.667 \pm 1.55^\circ\text{C}$  observed in the cold pressed oil, however both the oils possess the same fire point at  $238.667 \pm 0.577^\circ\text{C}$ . These values contradict the findings of Aremu et al. (2006) where he reported the values of  $335^\circ\text{C}$  and  $298^\circ\text{C}$  for fire point and flash point respectively.

The chemical analysis of the ether extracted castor seed oil showed that a higher significant difference existed at  $p < 0.05$  between the moisture content of the ether extracted oil which was  $0.240 \pm 0.010 \%$  compared to that of cold pressed oil which was found to be  $2.50 \pm 0.100 \%$  (Table 2). The exclusion of added water during the processing and extraction of the ether extracted oil might be the cause of the low moisture content in the oil. Oil and water can each wet the solid components of oilseeds, though the two differ in affinity for the hydrophilic surfaces of particles. Sefah, (2006) stated that moisture lubricates the pulp during pressing of castor seed and causes a slower pressure increase and reduces oil yields

The pH values of the two oils was found to be significantly different ( $p < 0.05$ ). The pH values of  $5.77 \pm 0.07$  and  $6.23 \pm 0.07$  were observed for the ether extract and cold pressed oil respectively. The addition of water during the processing and extraction of the cold pressed oil might be the cause of the higher pH content in the oil Aremu et al., (2006) stated that Low acid value and pH in oil indicates that the oil will be stable over a long period of time and protect against rancidity and peroxidation.

The acid value and free fatty acid content of the ether extracted oil were found to be  $1.009 \pm 0.056$  and  $0.507 \pm 0.023$  (Table 4.2), which are significantly different compared to the values of  $1.253 \pm 0.032$  and  $0.629 \pm 0.016$  obtained in the cold pressed oil.

The quantity of free fatty acid in oil is an indicator of its overall quality. They may be formed through hydrolysis or in the advanced stages of oxidation. An excessive amount of free fatty acids lowers the smoke point of oil (Aremu *et al.*, 2006).

The peroxide value, saponification value and iodine value showed higher significant difference ( $p < 0.05$ ). Values recorded for the cold pressed oil were  $3.493 \pm 0.023$ ,  $181.333 \pm 0.577$  mE (mg KOH/g, and  $83.667 \pm 1.155$  wj's g/100g respectively, compared to values of  $3.120 \pm 0.040$ ,  $176.000 \pm 1.000$  and  $81.333 \pm 0.577$  found in ether extracted oil. Values obtained fall within ASTM specification for Quality Castor Oil in the range of 175-187 mE (mg KOH/g for saponification value and 82-88 wj's g/100g for Iodine value. Nangbes *et al.*, 2013 reported values of 158.64 mE (mg KOH/g, and 53.64 wj's g/100g respectively for castor seed oil.

The iodine value is a measure of the degree of unsaturation and it is an identity characteristic of seed oils, making it an excellent raw materials for soaps cosmetics industries. Saponification value is a measure of oxidation during storage, and also indicates deterioration of the oils. An increase in

saponification value in oil increases the volatility of the oils. Peroxide value (PV) is the most common indicator of lipid oxidation. The unrefined vegetable oils are characterized by greater PV, compared to refined oils (Aremu *et al.*, 2006).

A research conducted by Alizeralu (2011) on chemical analysis of castor oil revealed that castor oil that ass obtained from the cold pressing method had low acid value, low iodine value, lighter color and slightly higher saponification value compared to the solvent-extracted oil.

#### 4.1 Conclusion

The solvent extraction has shown advantages over the cold pressed method because of its high percentage of oil recovery from the castor seeds. The result for the physicochemical properties of the ether extracted oil and cold pressed oil shows that the two oils differ significantly at  $p < 0.05$  and 95% confidence limit. Physical parameters of the ether extracted oil revealed higher quality oil compared to cold pressed oil. Further study should be carried out to extract the oil using pneumatic press with other solvents in place of water.

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