

## **Physicochemical Analysis and Potential Uses of Oil Extracted from Terminalia catappa Seeds**

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**Abstract:** Terminalia catappa is a widely planted ornamental tree in the Samaru campus of Ahmadu Bello University, Zaria. Although its seeds are known to be edible, there is limited research on their potential uses in the food and industrial sectors. This study aimed to extract oil from Terminalia catappa seeds using n-Hexane, Petroleum ether, and Ethanol solvents and analyze its physicochemical properties. The extraction was carried out using the Soxhlet apparatus, and the physicochemical properties determination was done using standard methods of analysis. The percentage oil yields were 51.80 %, 49.77 %, and 43.08 % for n-Hexane, Petroleum ether, and Ethanol extracts, respectively. The acid values (mg KOH/g), saponification values (mg KOH/g), iodine values (gI<sub>2</sub>/100g), and peroxide values (meq/kg) were determined to be 4.488, 3.366, and 15.147; 211.78, 166.898, and 123.42; 6.091, 6.345, and 6.345; and 8.75, 9.00, and 10.00 for n-Hexane, Petroleum ether, and Ethanol extracts, respectively. The highest oil yield was achieved using n-Hexane, and petroleum ether extract was most suitable for edible purposes. All three extracts could be used for soap making, but the n-Hexane extract was the most suitable. However, the three extracts were considered non-drying oil based on iodine values and not suitable for ink and paint production. Based on peroxide values, they were stable to oxidative rancidity.

**Keywords:** Terminalia catappa, oil extraction, physicochemical analysis, n-Hexane, petroleum ether, ethanol solvent



## التحليل الفيزيائي والكيميائي والاستخدامات المحتملة للزيت المستخرج من بذور الكاباس

**الملخص:** تعتبر *Terminalia catappa* من أشجار الزينة المزروعة على نطاق واسع في حرم جامعة Samaru بجامعة Ahmadu Bello ، Zaria. على الرغم من أن بذورها معروفة بأنها صالحة للأكل ، إلا أن هناك أبحاثاً محدودة حول استخداماتها المحتملة في قطاعي الأغذية والصناعة. هدفت هذه الدراسة إلى استخلاص الزيت من بذور نباتات كاتابا باستخدام *n-Hexane* و *Petroleum ether* و *Ethanol Solvents* وتحليل خواصه الفيزيائية والكيميائية. تم الاستخراج باستخدام جهاز Soxhlet ، وتم تحديد الخصائص الفيزيائية والكيميائية باستخدام طرق التحليل القياسية. كانت النسبة المئوية لإنتاج الزيت 51.80٪ و 49.77٪ و 43.08٪ لمستخلصات *n-Hexane* و *Petroleum ether* و *Ethanol* على التوالي. تم تحديد قيم الحمض (mg KOH / g) وقيم التصبن (mg KOH / g) وقيم اليود (gI<sub>2</sub> / 100g) وقيم البيروكسيد (meq / kg) لتكون 4.488 و 3.366 و 15.147 ؛ 211.78 و 166.898 و 123.42 ؛ 6.091 و 6.345 و 6.345 ؛ و 8.75 و 9.00 و 10.00 لمستخلصات *n-Hexane* و *Petroleum ether* و *Ethanol* على التوالي. تم تحقيق أعلى إنتاجية للزيت باستخدام *n-Hexane* ، وكان مستخلص الإيثر البترولي هو الأنسب للأغراض الصالحة للأكل. يمكن استخدام المقتطفات الثلاثة لصنع الصابون ، لكن مستخلص *n-Hexane* كان الأنسب. ومع ذلك ، اعتبرت المستخلصات الثلاثة زيتاً غير جاف بناءً على قيم اليود وغير مناسبة لإنتاج الحبر والطلاء. بناءً على قيم البيروكسيد ، كانت مستقرة للنتانة التأكسدية.

## 1. Introduction

Vegetable oils, as an essential component of the human diet, provide energy, fat-soluble vitamins, and essential fatty acids. They play a vital role in maintaining healthy bones, protecting the liver, boosting the immune system, and providing structural integrity to cells (FAO, 2009). Vegetable oils are obtained by extracting oil from seeds, kernels, and nuts of various plants using mechanical pressure or solvents. These oils are primarily composed of triacylglycerol (Gunstone, 2011). The global demand for vegetable and seed oils is increasing due to their various applications in domestic, industrial, pharmaceutical, and cosmetic industries. Therefore, there is a need to search for high-quality vegetable and seed oils to meet the growing demand.

However, obtaining large quantities of oil is only possible through the extraction of available nuts and oil seeds (Okene & Evbuomwan, 2014). Almost 80% of the global vegetable oil production is consumed by humans, while the remaining 20% is used for chemicals and animal industries. The availability, ease of processing technology, production rate, and utilization potential define the potential of an oil seed as a feedstock for industries (Aremu *et al.*, 2015). While soybean, groundnut, castor, coconut, palm, and jatropha are the sources of most oils, there are many underutilized potential sources of oil (Ogala *et al.*, 2018). Terminalia catappa seeds oil is one such source of nutritional oil (Agunbiade & Olanlokun, 2006).

Terminalia catappa, also known as the umbrella tree or tropical almond, is a member of a group of nuts that possess hard-shelled seeds which enclosed a single edible kernel. However, the crop is underutilized. The small size of the seeds and their difficulty in extraction may have contributed to their limited usage in many areas (Adu *et al.*, 2013). The Terminalia catappa tree is mostly planted in the tropics, particularly along sandy seashores, as a source of edible fruits, for shade, and ornamental purposes. It tolerates salt spray, relatively high salinity in the root zone, and strong winds (Lex & Barry, 2006).

The oil extracted from the dried nuts of Terminalia catappa is edible and used for cooking in parts of South America (Adu *et al.*, 2013). However, there is a lack of in-depth information on the nutritional potential of Terminalia catappa, resulting in the underutilization of the seeds.

Therefore, this research aims to extract oil from Terminalia catappa seeds using the solvent extraction method with different solvents and to characterize the oil extracted from the seeds using physical and chemical analysis. Furthermore, this study compares the effects of different solvents for Terminalia catappa oil extraction.

## 2.0 Materials and Methods

### 2.1 Collection, identification, and drying of the seeds

The Terminalia catappa seeds were sourced under some of the Terminalia catappa trees in the Samaru campus of Ahmadu Bello University Zaria. Moreover, the seeds were deshelled and dried for five days. In preparation for extraction, a pestle and mortar were used to ground the deshelled seeds.

### 2.2 Extraction procedure

Three samples each of 65g of the grounded Terminalia catappa seeds were wrapped in a filter paper and subjected to the extraction process using n-Hexane, Petroleum

Ether, and Ethanol respectively for 3 hours in a Soxhlet extractor. To recover some of the solvents in the extracted oil, a rotary evaporator was employed, and the remaining oil-solvent mixture was heated to the boiling points of the solvents to remove the residual solvent.

### 2.3 Determination of physicochemical properties of the oil

#### 2.3.1 Color

The oil's colors were determined by visual inspection

#### 2.3.2 Oil yield (%)

The % yield of the oil samples was determined using equation 2.1.

$$\text{Oil yield (\%)} = \frac{\text{Weight of the oil}}{\text{Weight of the seed}} \times 100 \quad (2.1)$$

#### 2.3.3 Oil recovery (%)

The oil recoveries were determined using equation 2.2

$$\text{Oil recovery (\%)} = \frac{\text{Weight of oil}}{X \times \text{weight of the seed}} \times 100 \quad (2.2)$$

Where X is the oil content of the seed = 60%

#### 2.3.4 Specific gravity

The specific gravities of the oils were determined using a 2 ml syringe. A 2ml capacity dry and clean syringe was weighed and recorded as  $W_0$ , it was then filled with each oil sample and reweighed and recorded as  $W_1$ . The syringe was then washed and dried and then filled with distilled water to give  $W_2$ .

The specific gravities were calculated using equation 2.3;

$$\text{Specific gravity} = \frac{W_1 - W_0}{W_2 - W_0} \quad (2.3)$$

Where,

$W_0$  = empty dry syringe weight;

$W_1$  = syringe + oil weight;

$W_2$  = syringe + distilled water weight

#### 2.3.5 Acid value

25 ml alcohol (propanol) and 2 drops of phenolphthalein indicator were mixed and were then neutralized using 0.1M KOH. 1g of the oil sample was added to the neutralized solvent and titrated with aqueous 0.1M KOH and vigorously agitated to a pink color, which marked the endpoint. The volume of the 0.1M KOH consumed was recorded. Equation (2.4) was used to calculate the acid value:

$$\text{Acid value} = \frac{56.1 \times V \times M}{w} \quad (2.4)$$

Where,

V = KOH used Volume;

M = KOH molarity and

w = weight of the sample.

#### 2.3.6 Iodine value

1g of the oil sample was placed in a 250 ml conical flask, and 20 ml of Wiji's solution was added. The flask was stoppered, then shaken. The mixture was allowed for 30 minutes in the dark. This was followed by adding 20 mL of 10 % potassium iodide solution and then shaking. The resulting mixture was then titrated against 0.1 M Sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) with 3 drops of starch solution as an indicator. The volume of  $\text{Na}_2\text{S}_2\text{O}_3$  was recorded at the endpoint. Further, a blank titration was carried out. Equation (2.5) was used to calculate the Iodine value (I.V).

$$I.V. = \frac{(b-a) \times N \times 12.69}{\text{Weight of the sample}} \quad (2.5)$$

Where;

b = Blank titre

a = Sample titre

N = Normality of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> used

### 2.3.7 Determination of Saponification Value

25 ml of 4% ethanolic potassium hydroxide solution was placed into a 250 ml conical flask and 2.0g of sample oil was added to it. The mixture was heated in a water bath for 30min. After it has cooled, 2 drops of phenolphthalein were dissolved in it and then titrated against 0.5M Hydrochloric acid solution with vigorous agitation to the disappearance of the purple color. Further, as a control, a blank titration was carried out. Equation (2.6) was used to calculate the saponification value.

$$SV = \frac{(b-a) \times M \times 56.1}{\text{Weight of the sample}} \quad (2.6)$$

Where;

b = Blank titre

a = Sample titre

M = HCl Molarity

### 2.3.8 Peroxide value

1 g of the oil sample was placed into a 250 ml capacity conical flask. 20 ml of solvent mixture (glacial acetic acid/chloroform, 3/2 by volume) and 1.0 g of potassium iodide were added and the mixture was boiled for one minute. Further, the hot solution was transferred into a flask containing 20 ml of 5% KI. Consequently, three drops of the starch solution were added to the mixture and titrated with 0.025 N standardized Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Equation (.27) was used to calculate the peroxide value.

$$P.V = \frac{V \times N \times 100}{\text{Weight of the sample}} \quad (2.7)$$

Where;

V = ] Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> Volume;

N = Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> Normality

### 2.3.9 Ester value

The ester value was calculated using equation (2.8) by the procedure described by Pearson (1991).

$$\text{Ester value} = \text{Saponification value} - \text{Acid value} \quad (2.8)$$

## 3.0 Results and Discussion

Table 3.1: Percentage of oil yield and oil recovery assuming 60 % oil content in the seed

S/no	Parameters/Solvents	n-Hexane	Petroleum Ether	Ethanol
1	Oil yield (%)	51.80	49.77	43.08
2	Oil recovery (%)	86.4	82.95	71.80

Table 3.2: Summary of physical properties of the extracted oil with the three different solvents

S/no	Properties/Solvents	n-Hexane	Petroleum Ether	Ethanol
1	Color	Pale brown	Medium brown	Dark Brown
2	Specific gravity	0.892	0.892	0.921
3	Odor	Smell of groundnut	Smell of groundnut	Smell of groundnut
4	State at room temperature	Liquid	Liquid	Liquid

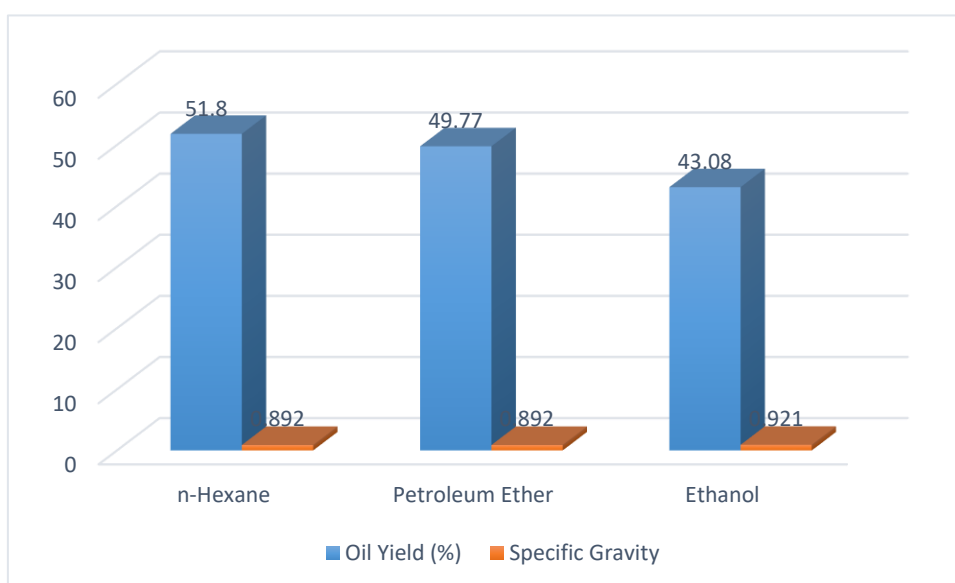


Figure 3.1: Oil yield and specific gravities of the three oil samples

Table 3.3: Summary of chemical properties of the extracted oil with the three different solvents

S/no	Properties/Solvents	n-Hexane	Petroleum Ether	Ethanol
1	Acid Value (mg KOH/g)	4.488	3.366	15.147
2	Saponification Value (mg KOH/g)	211.780	166.898	123.420
3	Iodine Value (gI <sub>2</sub> /100g)	6.091	6.345	6.345
4	Peroxide Value (meq/kg)	8.750	9.000	10.000
5	Ester Value (mg KOH/g)	207.292	163.532	108.273

### 3.2 Discussion of Results

#### 3.2.1 Oil yield

The percentage oil yield using n-Hexane, Petroleum ether, and Ethanol solvents were 51.80%, 49.77%, and 43.08% respectively as shown in Table 4.1. These values are greater than 38.00% for Terminalia mentalis using Petroleum ether as the extraction solvent (Kayode, 2015) and 10.34% for Zobo seeds (Theodora & Cosmas, 2017). However, the values are closer to 47.08% and 49.34% for Sesame and Cashew seeds (Saeed & Shola, 2015). The study shows that the highest amount of oil extraction was achieved with n-Hexane, followed by Petroleum ether, then Ethanol. Moreover, these oil yields can be considered economical for commercial production since they are within the standard range of  $\geq 32\%$  (AOAC, 1990)

#### 3.2.2 Specific gravity

The oils obtained using n-Hexane, Petroleum ether, and Ethanol recorded specific gravities of 0.892, 0.892, and 0.921 respectively as shown in Table 4.2 and Figure 4.1. The values obtained for n-Hexane and Petroleum ether are comparable to 0.85 for ackeed seed oil (Omosuli, 2013), and that of the Ethanol is comparable to 0.90 for Zobo seed oil (Theodora & Cosmas, 2017). However, the values are all greater than 0.500 and 0.512 for Cashew nut and Cashew shell liquid oil (Idah *et al.*, 2014). The result obtained in this study shows that water is denser than the three oil samples. Moreover, the extracted oil using Ethanol solvent is denser than those extracted using n-Hexane and Petroleum Ether. Perhaps, Ethanol extracts not just oil but also some pigments in the seed.

#### 3.2.3 Chemical properties

##### 3.2.3.1 Acid value

The free fatty acid present or the degree of hydrolysis of oil determines the acid value of oil. The Acid value for an oil that is suitable for edible purposes should be less than or equal to 4 mg/g (Janporn *et al.*, 2014). Acid value depends on the degree of rancidity which is used as a measure of oil freshness (Ochigbo & Paiko, 2011). The acid values obtained using n-Hexane, Petroleum Ether and Ethanol were 4.488, 3.366, and 15.147 mg KOH/g respectively as shown in Table 4.3. Kayode (Kayode, 2015), reported a lesser value of 0.052 mg KOH/g for Terminalia mentalis using Petroleum ether as the extraction solvent. Also, Olatidoye *et al.* (Olatidoye *et al.*, 2011), reported a lesser value of 1.3 mgKOH/g for Terminalia catappa using Petroleum ether as the extraction solvent. However, the values obtained in this study for n-Hexane and Petroleum ether are similar to the reported values of 4.77 mg KOH/g for white cultivars of melon seed and 5.99 mg KOH/g for ground nut oil by Olaofe *et al.* (Olaofe *et al.*, 2012). The acid value obtained in this study using Petroleum ether as the extraction solvent is within the recommended limit. Hence, it is suitable for edible purposes. However, those obtained using n-Hexane and Ethanol



need to be refined properly before consumption because they exceeded the required value for edible oils. The results also suggested that the obtained oil using Petroleum ether is most stable for a long period and resistant to rancidity and peroxidation.

### 3.2.3.2 Saponification value

The measure of oils' oxidation on storage and their deterioration is defined by their saponification value. The presence of a higher number of carbon atoms fatty acids indicates a high saponification value (Ardabili *et al.*, 2011). The saponification value recorded in this study for n-Hexane, Petroleum ether, and Ethanol were 211.78, 166.898, and 123.42 mg KOH/g respectively as shown in Table 4.3. The value obtained for the oil extracted using Petroleum ether is greater than 140.275 mg KOH/g obtained for *Terminalia mentalis* (Kayode, 2015), and 128.0 mg KOH/g recorded for *Terminalia catappa* (Olatidoye *et al.*, 2011). However, the saponification values obtained for the extracted oils using n-Hexane and Petroleum ether are quite higher than 159.33 mg KOH/g for *Dennettia tripatala* fruit oil (pepper fruit) reported by Nwinuka *et al.* (Nwinuka *et al.*, 2009) and 143.76 for African pear oil (Ikhuoria & Maliki, 2007).

The saponification values of the oils fall within the range of these oils, therefore they could also be used for soap making. Higher saponification value determines the usage of fat or oil for soap making, therefore, the oil extracted using n-Hexane would be most suitable for soap making among the three extracts. Moreover, the oil extracted using Ethanol is higher than 93.0 mg KOH/g of bese wax (Mabrouk, 2015). Hence, it can also be used for soap making but finds the least application among the three oils.

### 3.2.3.3 Iodine value

The degree of unsaturation in oil is determined by its iodine value, which determines the number of double bonds present in the oil which defines its proneness to oxidation (Bello *et al.*, 2011). Oils with less than 100 gI<sub>2</sub>/100g iodine value are classified as non-drying oils, more than 100 gI<sub>2</sub>/100g but less than 130 gI<sub>2</sub>/100g are semi-drying, while oils with iodine value above 130 gI<sub>2</sub>/100g are classified as drying oils (Aremu *et al.*, 2006). The iodine values obtained are 6.091, 6.345, and 6.345 gI<sub>2</sub>/100g using n-Hexane, Petroleum ether, and Ethanol respectively as shown in Table 4.3. These values are less than 54.567 gI<sub>2</sub>/100g for *Terminalia mentalis* oil extracted using Petroleum ether (Kayode, 2015), and 65.0 gI<sub>2</sub>/100g for *Terminalia catappa* extracted using Petroleum ether (Olatidoye *et al.*, 2011). However, the values are closer to the 9.4 gI<sub>2</sub>/100g for coconut seed oil extracted using Isopropyl alcohol solvent and 9.3 gI<sub>2</sub>/100g for the same oil extracted using Petroleum ether (Okene & Evbuomwan, 2014). In this study, the oils obtained using the three solvents can be classified as non-drying oils. Hence, not applicable for paint and ink production, owing to their non-drying properties. However, could be used in soap manufacturing.

### 3.2.3.4 Peroxide value

The rate of lipid oxidation, which causes rancidity is determined by peroxide value. Normally, oils are said to be rancid when their peroxide value is between 20.0 meq/kg to 40.0 meq/kg (Babalola, 2011). The maximum limit for nuts and seed oils as marked by the Codex Alimentarius Commission is 10 meq/kg (SON, 2000). The values obtained in this study for n-Hexane, Petroleum ether, and Ethanol are 8.75 meq/kg, 9.00 meq/kg, and 10.00 meq/kg respectively as presented in Table 4.3. These values are greater than 2.600 meq/kg for *Terminalia mentalis* extracted using Petroleum ether as the extraction solvent (Kayode, 2015) and 2.8 meq/kg for

Terminalia catappa oil extracted using Petroleum ether (Olatidoye *et al.*, 2011). However, these values are less than 13.80 meq/kg and 10.80 meq/kg for Moringa oil and Ground nut oil respectively (Afolayan *et al.*, 2014).

The peroxide values obtained using Petroleum ether and n-Hexane as the extraction solvents were less than the maximum limit while that obtained using Ethanol was equal to the maximum. Therefore the oils are not prone to oxidative rancidity.

#### **4. Conclusion**

This study successfully extracted oil from the seeds of Terminalia catappa using three distinct solvents: n-Hexane, Petroleum ether, and Ethanol via the Soxhlet apparatus. The results showed that n-Hexane achieved the highest oil yield of 51.80%, followed by Petroleum ether at 49.77% and Ethanol at 43.08%. Physicochemical analysis revealed that the Petroleum ether extract is suitable for edible purposes without undergoing refining, while the other two extracts require refining to be considered edible. Additionally, all three extracts can be used in soap making, with n-Hexane being the most suitable based on their saponification values. However, none of the extracts are suitable for ink and paint production since they were classified as non-drying oils. The peroxide values of the three samples were less than the maximum allowable limit of 10 meq/kg, indicating that they will be stable to oxidative rancidity. Terminalia catappa seed is a potential source of oil that can meet the ever-growing demand for quality oil that could be used in industries and other domestic purposes.

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