

Physicochemical Characteristics Study of Oil Extracted from Almond Seed

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Abstract

The physicochemical properties of the extracted oil from almond seed were investigated. Physical properties such as cloud point, pour point, fire point, smoke point, specific gravity, refractive index, and viscosity; and the chemical properties such as acid value, free fatty acid, saponification value, iodine value, ester value, and peroxide value were investigated for both raw almond oil and transesterified almond oil. The various properties were investigated using ASTM standard methods and calculations. Results obtained for physical properties: cloud point, pour point, fire point, smoke point, specific gravity, refractive index, and kinematic viscosity were $-3^{\circ}C$, $-9^{\circ}C$, $220^{\circ}C$, $130^{\circ}C$, 0.928, 1.462, $21.84\text{ mm}^2/\text{sec}$ respectively for the transesterified almond oil. Result obtained for the chemical properties of the transesterified almond oil: acid value, free fatty acid, saponification value, iodine value mgI_2/g , and ester value, values were determined to be: 2.05, 1.03, 79.71, 34.33, and 77.66. It was concluded that transesterified Almond oil have a great potential for use as fuel alternative and substitute for internal combustion engine with its high purity, low corrosion ability and ease of ignition.

Keywords: Almond seed, physicochemical properties, saponification, transesterified, corrosion.

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INTRODUCTION

Renewable energy is the energy collected from renewable resources, which are naturally replenished on a human timescale, such as sunlight, wind, rain, tides, waves, biomass, and geothermal heat. Renewable energy often provides energy in four important areas: electricity generation, air and water heating or cooling, transportation, and rural (off-grid) energy services [1]. In the case of renewable fuels for compression ignition (diesel) engines, the majority of efforts to date have focused on biodiesel, which consists of alkyl esters of fatty acids. Biodiesel has been shown to give engine performance that is generally comparable to that of conventional diesel fuel while reducing engine emissions of particulates, hydrocarbons and carbon monoxide [2]. In recent years, the fluctuating price of crude oil, depleting fossil fuel resources and the growing concerns over environmental pollution has stimulated renewed interest in seed oils as a substitute for petroleum crude. Edible oils are much more valuable as a cooking fuel and as such, in this research concentration is on the development of biodiesel from non-edible oils (Almond). To avoid some of the problems which vegetable oil gives when used straight on the CI engines, they are converted via a chemical

process (transesterification) to produce biodiesel, which is a biodegradable and non-toxic renewable fuel. Furthermore, biodiesels have reduced molecular weights (in relation to triglycerides), reduced viscosity, and improved volatility when compared to ordinary vegetable oils. Biodiesel from soya, rapeseed, and oil palm dominate the current market for biofuels, but some companies are going aggressively to develop and market a number of advanced second-generation biofuels made from non-food feedstocks, such as municipal waste, algae, perennial grasses, and wood chips. These fuels include cellulosic ethanol, bio-butanol, methanol and a number of synthetic gasoline/diesel equivalents. Until we are able to produce a significant number of electric vehicles that run on renewably-produced electricity, biofuels remain the only widely available source of clean, renewable transportation energy [3].

Almond seeds are in the family *Rosaceae* in addition to *Pomoideae* (apples, pears), *Prunoideae* (apricot, cherry, peach, and plum) and *Rosoideae* (blackberry, strawberry) fruits. There are two major varieties of almonds, the bitter almond (*Prunus amygdalus* “*amara*”) and the sweet almond (*Prunus*

amygdalus "dulcis") used mainly for culinary purposes, and making of oils and flavourings, respectively [4]. Almond (*Prunus amygdalus "dulcis")* is one of the species of *Prunus* belonging to the subfamily *Prunoideae* of the family *Rosaceae*. Almond seeds contain approximately 51% lipid, 21% protein, 20% carbohydrate and 12% fiber [5].

Muhammed *et al.*, [6] carried out a research work on the tropical almond seed oil using CaO derived from snail shell as catalyst which is also heterogenous catalyst. The extract was analysed to examine the physicochemical characteristics. It was concluded that tropical almond seeds oil is a good feedstock for biodiesel production with little modification in its properties.

A research work on the sweet almond (*Prunus amygdalus "dulcis")* seed oil (SASO) as a non-conventional feedstock for the preparation of biodiesel in Nigeria, rather than the traditional oils of palm, groundnut and palm kernel was carried out by [5]. It was extracted via the solvent method, pretreated to reduce the acid value, and transesterified using methanol (solvent) and sodium hydroxide (homogenous catalyst). The oil content and acid value of SASO were $51.45 \pm 3.92\%$ and 1.07 mg KOH/g, respectively. The fatty acid composition of the oil shows the predominance of oleic acid (69.7%), linoleic acid (18.2%) and palmitic [acid (9.3%). Specific fuel properties of sweet almond oil methyl esters (SAOME) were determined using standard test methods and were found to satisfy both EN 14214 and ASTM D6751 biodiesel standards; the cold flow properties were particularly outstanding (cloud point; -3°C and pour point; -9°C). They come into conclusion that the seed oil appears to offer great promise as a potential feedstock for biodiesel production in Nigeria.

Therefore, this work aims to perform a physicochemical characteristics study of oil extracted from Almond oil seeds that includes free fatty acid, iodine value, acid value, saponification value, specific gravity, viscosity, ester value, heat of combustion, cloud point, pour point, flash point, refractive index and estimation of Cetane number for energy applications.

MATERIALS AND METHODS

Materials and Equipment

The materials and equipment used for the study include Almond seed, Solvent (n-hexane), Alcohol (Methanol), Catalyst (Calcium oxide, CaO), Phenolphthalein, Beakers, Conical Flasks, Measuring Cylinder, Tripod Stands, Magnetic Striver, Thermometer, equipped with a reflux condenser and a thermometer, Abbe's refractometer, Pycnometer bottle, 1L Electro thermal Soxhlet (Heating Mantle).

METHODS

Sample Collection and Preparation

Almond seeds were collected domestically at the premises of the Federal University of Technology Akure, Nigeria. These seeds were allowed to dry for seven days before breaking them manually to get the kernel.

Other chemicals and reagents (methanol, n-hexane, and Calcium Oxide) were purchased at Pascal Chemical Store, Akure, Nigeria.

Oil Extraction

Dried Almond seeds of total weight 375 g was allowed to dry for two days in the oven at (70 to 100) $^{\circ}\text{C}$, after which they were pulverized into powder form (Fig. 1) with the use of an electric blender, to allow for easy solvent contact and better extraction. The pulverized seed was weighed using an electronic scale shown in Fig. 2, and 194.45 g of the powdered seeds was poured into a white piece of muslin cloth which was tied at the mouth, and this was inserted into a 1000 ml capacity soxhlet extractor. The extraction was done for two samples. For the extraction, n-hexane was used as the solvent, and the 1 litre round bottom flask was filled to three-quarter of its volume. The powdered form of the seeds was fed to Soxhlet extractor shown in Fig. 3; the system was connected to a 1 litre round bottom flask and a reflux condenser. The extraction was carried out for about 3 hours per sample. After this, the acid value of crude almond seed oil was determined, which indicated that there will be a need for acid pretreatment (esterification) because of its high acid value before transesterification.



Fig-1: Pulverized Seed



Fig-2: Weighing the Pulverized



Fig-3: Soxhlet Extractor

Transesterification

In order to produce oil that is similar to the conventional diesel fuel, a portion of the extracted oil undergoes catalyzed transesterification processes. A two-step acid catalyst esterification and alkali catalyst transesterification, proposed by [7], were employed because the free fatty acid (FFA) content of the extracted oil was greater than 1%.

Physical and Chemical Characteristics of Oil

Refractive Index: The refractive index was determined with refractometer. The prism of the refractometer was wiped with tissue paper moistened with acetone to remove dirt, oil or grease. A drop of the oil sample was placed on the surface and clamped. The lens was viewed and the control knob adjusted until a faint blue line was seen inform of a cross across the meter. The reading was then taken and recorded.

Specific Gravity: This is the ratio of the mass of oil in gram weight to that of equal volume of water.

A 50 ml pycnometer bottle was washed, then dried and weighed (W_1). The bottle was filled with water and weighed (W_2). The bottle was emptied and dried. Then, the bottle was filled with the oil sample and was weighed (W_3). It was calculated using equation (1).

$$\text{Specific Gravity} = \frac{W_3 - W_1}{W_2 - W_1} \quad (1)$$

W_3 is the Weight of Oil

W_2 is the Weight of volume of water

W_1 is the Weight of empty bottle

Kinematic Viscosity: A clean and dry viscometer with a flow time above 200 seconds was selected for the oil sample. The viscometer was charged with the sample by inverting the tube's thinner arm into the liquid sample and suction force was drawn up to the time mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into holder and inserted to a constant temperature water bath set at 40°C. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time was recorded by timing the flow of the sample as it flowed freely from the upper timing mark to the timing mark to the lower timing mark. This was obtained using equation (2).

$$\text{Viscosity} = \frac{\text{Flow time} \times \text{Specific gravity} \times 1.002}{\text{Flow time of water}} \quad (2)$$

Smoke Point: A petri dish was filled with 20 ml of the sample oil and this was heated continuously on a hot plate until the sample gave off a thin fume with continuous streams of bluish flame. A thermometer was clamped on the retort-stand, the sensitive part of thermometer was dipped into the petri dish to record the temperature at this point.

Flash Point: The same oil sample was used and with the aid of a clamp holder, a thermometer was hung and dipped inside the oil, ensuring that the thermometer does not touch the bottom of the petri dish which was placed on a hot plate. The sample was heated until a sufficient vapour is produced when flame was applied, it causes burning for more than one minute. The temperature is then recorded.

Fire Point: The sample that was used for the flash point was used with the heating continued until a sufficient vapour was produced and when the flame was applied, it caused burning for a period of more than one minute. The temperature was then recorded.

Cloud Point: 50 ml of sample was measured into a glass bottle containing a thermometer and immersed together in a water bath. The water bath and the content were cooled in a refrigerator with the stirring of the oil. The temperature at which the

thermometer was no longer visible was taken as the cloud point

Pour Point: 5 ml of oil was drawn into a capillary tube tied to a thermometer, placed in a 250 ml beaker containing distilled water immersed together in a water bath for controlled heating. The temperature at which the oil just begins to move downward due to its weight is called the pour point.

Almond Oil Yield: The powdered seeds before oil extraction were weighed and also the powdered seeds after oil extraction. The percentage oil yield was obtained using equation (3).

$$\% \text{ Oil Yield} = \frac{\text{Initial weight of Sample} - \text{Final weight of Sample}}{\text{Initial weight of Sample}} \times 100\% \quad (3)$$

Mean molecular mass: The mean molecular mass of the oil is calculated using equation (4):

$$\text{Mean molecular mass} = 100 \times \left[\frac{56}{\text{SV}} \right] \quad (4)$$

Where; S.V is the Saponification value.

Cetane Number: Cetane number was calculated as reported by Mohibbe *et al.*, 2005 using equation (5).

$$\text{Cetane No} = 46.3 + \frac{5458}{\text{SV}} - 0.225(\text{IV}) \quad (5)$$

Where;

SV is the saponification value, and

IV is the iodine value.

High Heating Value: The heating value was calculated as reported by [8] using the equation (6):

$$\text{High Heating Value} = 49.43 - 0.041(\text{SV}) - 0.015(\text{IV}) \quad (6)$$

Where;

SV is the saponification value, and

IV is the iodine value.

Ester Value: This is the number of milligrams of potassium hydroxide required to saponify the ester present in 1g of the oil. It is obtained as the difference between the saponification value and the acid value.

RESULTS

Tables 1 and 2 show the details of the results of physical and chemical properties of the raw extracted oil and the transesterified oil of Almond oil as compared with the standard limits and diesel oil using standard test methods of America System of Test Materials (ASTM) and European Standard (EN).

Table-1: Physical Characteristics of Raw and Transesterified Oil of Almond Seed

PARAMETER	UNITS	EXTRACTED OIL	TRANSESTERIFIED OIL	ASTM LIMITS	EN LIMITS	DIESEL
Refractive Index @ 32°C	°C	1.464	1.462	-	-	-
Specific Gravity	-	0.981	0.928	-	-	-
Kinematic Viscosity @ 40°C	mm ² /s	33.62	21.85	1.9-6.0	3.5-5.0	3.384
Pour Point	(°C)	-	-9	-15 to 10	-	-20
Cloud Point	(°C)	-	-3	-3 to 12	-	-19
Smoke Point	(°C)	-	130.000	-	-	-
Flash Point	(°C)	-	172.000	>130	>120	85
Fire Point	(°C)	-	220.00	-	-	82
Free Fatty Acid	(mg/g)	3.04	1.03	-	-	-
Peroxide Value	(mg _{KOH} /g)	2.15	-	-	-	-
Saponification Value	(mg _{KOH} /g)	169.76	79.71	-	-	159.89
Density	(g/cm ³)	0.97	0.91	0.87 – 0.90	0.86-0.90	0.848
Moisture Content	(%)			-	-	-
Cetane Number		65.18	107.04	> 47	>51	46
Mean Molecular Mass	%	32.9	70.3	-	-	-
Higher Heating Value	(MJ/kg)	41.49	45.65	-	-	-
Heat of Combustion	(cal/g)			-	-	-
pH Value		4.80	6.70	-	-	-

Table-2: Chemical Characteristics of Raw and Transesterified Oil of Almond Seed

PARAMETER	UNITS	EXTRACTED OIL	TRANSESTERIFIED OIL	ASTM LIMITS	EN LIMITS	DIESEL
Acid Value	(mg _{KOH} /g)	6.08	2.05	< 0.80	< 0.50	-
Free Fatty Acid	(mg/g)	3.04	1.03	-	-	-
Peroxide Value	(mg _{KOH} /g)	2.15	-	-	-	-
Saponification Value	(mg _{KOH} /g)	169.76	79.71	-	-	159.89
Iodine Value	(gI ₂ /g)	58.98	34.33	-	< 120	-
Ester Value	(mg _{KOH} /g)	163.68	77.66	-	-	-

DISCUSSIONS

Refractive Index: The almond oil and the transesterified oil had refractive indices of 1.464 and 1.462 at 32°C respectively. The result agrees with most of the seed oils reviewed that have their refractive index values within the range of 1.468 to 1.471 for virgin and refined oils according to Codex Standards for fats and oils for vegetable/plant sources and this indicates that there is high purity in the oil [9].

Kinematic Viscosity: Viscosity is a measure of a fuel's flow characteristics and its tendency to deform under stress. Kinematic viscosity is an important parameter with regard to fuel atomization as well as fuel distribution and the Kinematic Viscosity of biodiesel is significantly influenced by the feedstock used [5]. The Kinematic Viscosity of the oil and its biodiesel under study at 40 °C were determined to be 33.62 mm²/s and 21.85 mm²/s, which are above the ranges specified by ASTM D445 and EN standards, and 9.1 mm²/s for Algae oil biodiesel reported by [10], and 5.20 mm²/s reported by [11]. The attained values agree with the work of [6] that reported 31.90 mm²/s and 19.10 mm²/s for extracted and transesterified oils respectively. The high values would result in lower fuel consumption and less wear, but starting will be poor.

Cetane Number: This is the measure of the tendency of fuel to self-ignite at the temperature and pressure in the cylinder when the fuel is injected. It is one of the key parameters considered in the use of biodiesel as fuel since a satisfactory cetane number is required for good engine performance. The calculated cetane number for the transesterified oil is 107.4. The Cetane number determined for the transesterified oil satisfies both ASTM and EN quality standards that set minimum limits of 47 and 51, respectively. The higher value would result in higher combustion efficiency and smoother combustion [12].

Pour Point: The pour point of the transesterified oil was determined to be -9°C which was within the range of the ASTM limits for standard specification of -15 to 10°C. Pour point is the lowest temperature at which frozen oil can flow and used to specify the cold temperature instability of fuel oil [13]. This shows that the biodiesel from almond oil would perform very well in very cold and temperate regions.

Cloud Point: The cloud point of the transesterified oil was determined to be -3°C. Cloud point and Pour point are the key flow properties for the winter fuel specification. Cloud point is the temperature at which wax form a cloudy appearance, and it is measured as the temperature of first formation of the wax as the fuel is cooled.

Flash Point: The flash point of the transesterified oil was determined to be 172°C. The

flash point is the measure of flammability of fuels. The flash point of this biodiesel is higher than the standard limit specified by ASTM and EN, which makes it much safer in handling than diesel. That is, the fear of fire outbreaks would be eliminated.

Smoke Point: The smoke point determined for the transesterified oil was 130°C. This is the temperature at which oil when heated gives off the first smoke. The smoke point of oil is dependent on the level of its purity and the time duration between when the oil was produced and smoke tested. Since the flash point for the transesterified oil is favourable therefore the smoke point will be favourable likewise.

Acid Value: The acid value for both the extracted oil and the transesterified oil were determined to be 6.08 mg_{KOH}/g and 2.05 mg_{KOH}/g respectively. Acid Value measures the extent to which glycerides in the oil have been decomposed by liquid. This acid value is higher than 0.78 mg_{KOH}/g reported by [14] for Tropical Almond oil but the acid value of the oil is lower than 15.37 mg_{KOH}/g reported by Bello and Agge, (2012) for groundnut oil. The acid value of the biodiesel under study is slightly higher than 0.8 mg_{KOH}/g specified by ASTM but less than 3.366 mg_{KOH}/g reported by [15] for groundnut oil biodiesel. Acid value and Free Fatty Acid (FFA) can be used to check the level of oxidation deterioration of the oil by enzymatic or chemical oxidation [16]. Increase in FFA of oil lower the chances of conversion to biodiesel with different types of side reactions. It also serves as one of the key factors that determined the suitability of oil for use in biodiesel production [17]. If the acid value is low, it indicates that ethyl ester biodiesel produced may not cause severe corrosion in internal combustion engine and fuel system [18].

pH Value: This is the measure of the degree of acidity or alkalinity of a solution. The pH value of the extracted oil and the transesterified oil were measured as 4.80 and 6.70, which signifies that the extracted oil is slightly acidic in nature and may possess some corroding capabilities while that of the transesterified oil would be a viable fuel substitute as its corrosion ability is low. The normal pH for diesel fuel is between 5.5 and 8.0, so a reading of below 5.5 is an indicator there could be a problem. The industry recognizes that the lower the pH, the higher the potential for serious corrosive damage to metal components in the system.

Iodine Value: The Iodine value of the extracted almond oil was 58.98 g_{I₂}/100g. This value is higher than 33.88 g_{I₂}/100g obtained by [6], and lower than 121.19 g_{I₂}/100g reported by [14]; both for tropical almond. For the transesterified oil, 34.33 g_{I₂}/100g was obtained. This value agrees with that of the biodiesel of 36.67 g_{I₂}/100g reported by [6]. For both the extracted oil and the transesterified oil, the values were lower than 100.

This shows that oil from almond seed contained saturated fatty acid, which places the oil in non-drying groups and does not pose any danger of inflammability [19].

Ester Value: The ester value obtained for the extracted oil and the transesterified oil of the almond seed were 163.68 mgKOH/g and 77.66 mgKOH/g respectively. These were comparable to 312.14 mgKOH/g recorded by [19] for Raffia fruit, also was comparable to 174.09 mgKOH/g for Castor oil and 191 mgKOH/g for rubber seed oil recorded by [20] which was also viable sources of biofuel.

Saponification Value: The Saponification value for the extracted oil and the transesterified oil were determined to be 169.76 mg_{KOH}/g and 79.71 mg_{KOH}/g. Saponification Value is used in checking adulteration. Though, this result is lower than 330.99 mg_{KOH}/g and 179.52 mg_{KOH}/g reported by [19] for Raffia regalis but the value agrees with the result presented by [6] that gave 78.5 mg_{KOH}/g as saponification value for the biodiesel, but is lower than 182.4 mg_{KOH}/g reported by [11] for tropical Almond biodiesel.

Density: The Density determines the specific gravity of both the oil and the diesel. The density of the extracted oil is 0.97 g/cm³ and that of the transesterified oil is 0.91 g/cm³ which is within the range of the ASTM standard, while that of diesel is 0.848 g/cm³. The density of the biodiesel is lower than 0.96 g/cm³ reported by [11] for Tropical Almond biodiesel. The density of the biodiesel affects the performance of the pump and atomizers.

Higher Heating Value: This is one important parameter of biodiesel fuel which represents the amount of heat transferred to the chamber during combustion and indicates the available energy in a fuel [21]. The higher heating value result obtained for the extracted oil and the transesterified oil was, 41.76 MJ/kg and 45.65MJ/kg which the transesterified oil is higher than 29.88 MJ/kg reported for *Hevea brasiliensis* seed oil biodiesel [22]. This indicates that the biodiesel produced contain appreciably high amount of energy. Fuel having higher heating value gives higher power output and its small quantity will cover long distance drive [23].

Peroxide Value: This value helps in determining which oil could be easily susceptible to oxidative rancidity, as higher peroxide value is indicative of higher susceptibility to oxidation [24]. The peroxide value for the extracted oil of almond oil is 2.15meq/kg. These were quite higher than the values of 1.8meq/kg reported for Raffia Regalis by [19] and quite low when compared to the value of 6.40meq/kg reported for *Aphelia Africana* by [25]. This showed that

the oil would be stable and this stability is further confirmed by the low level of free fatty acids.

CONCLUSION

The physicochemical characteristics of extracted and transesterified almond seed oil were studied. Results indicated that the oil has high potential for industrial applications. The physicochemical composition of the transesterified almond oil showed that it will be very good fuel substitute for internal combustion engine with its high purity, low corrosion ability and ease of ignition.

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