



### EXTRACTION, CHARACTERIZATION, AND BIODIESEL POTENTIAL OF PUMPKIN SEED OIL AS A SUSTAINABLE ENERGY RESOURCE

Omale, P. A., John, O. A. and Joseph, O. S.

Department of Agricultural and Biosystems Engineering, Joseph Sarwuan Tarka University, Makurdi-Benue State Corresponding Author's Email: omale.paul@uam.edu.ng

#### **ABSTRACT**

The study focused on the extraction and characterization of pumpkin seed oil as a feedstock for biodiesel production, exploring its potential as a sustainable energy resource. Oil was isolated from pumpkin seeds using the solvent extraction method, with hexane selected as the primary solvent due to its high efficiency in extracting oil. The extracted oil was characterized for its physicochemical properties to determine its suitability as a biodiesel feedstock. The result of Tin Layer Chromatography (TLC) for the determination of Fatty Acid Methyl Ester (FAME) and biodiesel purity showed that, the biodiesel produced was pure when compared with ASTM D6751 and EN 14214 standards. The transesterification process, a key step in biodiesel production was conducted using methanol and a base catalyst (potassium hydroxide) which convert pumpkin seed oil into fatty acid methyl esters (FAMEs). The reaction parameters, including molar ratio of methanol to oil, catalyst concentration, temperature, and reaction time was considered to maximize biodiesel yield and purity. The results of the density, API gravity, FFA, pH, flash point, cloud point, pour point and fire points for the pumpkin seed oil and its biodiesel were 0.93 and 0.84 (g/cm<sup>3</sup>), 3.62 and 3.98 (g/cm<sup>3</sup>), 32.27 and 28.54 (%), 5.81 and 7.84, 130 and 150 (°C), 20 and -7 (°C), 17 and -8 (°C), 140 and 170 (°C), respectively. The results indicated that, pumpkin seed oil exhibits favorable characteristics for biodiesel production with promising fuel properties, meeting most of the required standards for use in diesel engines. The utilization of pumpkin seeds, often considered agricultural waste when used as biodiesel, adds value to pumpkin and promotes its sustainable practice.

Keywords: Extraction, Characterization, Biodiesel, Pumpkin, Sustainable, Energy

#### INTRODUCTION

The escalating global demand for energy, coupled with pressing environmental concerns stemming from the combustion of fossil fuels, has intensified the search for renewable, sustainable, and eco-friendly energy alternatives (Demirbas, 2021; Nanda *et al.*, 2023). Among these, biodiesel has emerged as a highly promising substitute for conventional petroleum diesel (Nyishember *et al.*, (2024). Derived from biological sources like vegetable oils and animal fats, biodiesel offers significant advantages, including reduced greenhouse gas emissions, inherent biodegradability, and compatibility with existing diesel engines (Knothe, 2022; U.S. Department of Energy, 2024). While various plant-based oils are widely favored for biodiesel production due to their abundance and renewable nature, the focus on sustainable and cost-effective feedstocks is increasingly shifting towards underutilized, non-edible sources to avoid competition with food supply (Atabani *et al.*, 2022; Nizami *et al.*, 2021).

Pumpkin seed oil, derived from the seeds of various *Cucurbita* species (e.g., *Cucurbita pepo*), has gained increasing attention in recent years for its versatile applications and rich bioactive compound profile, including unsaturated fatty acids, tocopherols, phytosterols, and phenolic compounds (Stevenson *et al.*, 2020; Montesano *et al.*, 2021). Pumpkin seeds are often a byproduct of the food industry, making them a potential and largely underutilized feedstock for biodiesel production. Traditionally, pumpkin seed oil has been valued for its nutritional and medicinal properties; however, its potential as a biofuel has garnered growing interest (Rashid *et al.*, 2023). The effective extraction and subsequent characterization of oil from pumpkin seeds are crucial for determining its suitability for biodiesel production, as key properties such as fatty acid composition,





viscosity, and oxidative stability directly influence the quality and performance of the resulting biodiesel (Bikash *et al.*, 2018; Samani *et al.*, 2022).

Despite the recognized environmental and energy security benefits of biodiesel, a significant challenge lies in identifying and effectively utilizing sustainable and economically viable feedstocks that do not compete with food resources (Atabani *et al.*, 2022). While numerous studies have explored various non-edible oils for biodiesel production, the potential of pumpkin seed oil as a viable, non-edible feedstock remains underexplored in terms of its comprehensive characterization specifically for biofuel applications. Existing research often focuses on its nutritional or cosmetic properties, with limited dedicated studies addressing its specific suitability and performance metrics for biodiesel production. This knowledge gap hinders the full realization of pumpkin seed oil's potential as a sustainable and abundant alternative in the biofuel sector. The study aims to extract and characterize physiochemical properties of pumpkin seed (*Cucurbita pepo*) oil and evaluate the biodiesel produced from the seed against international standards (ASTM D6751, EN 14214) as a sustainable energy resource.

#### **MATERIALS AND METHODS**

The oil used for this work was extracted from pumpkin seeds purchased from Jos, Plateau State. The extraction of the oil was done using solvent extraction method. The extraction was carried out at the Food and Technology Department Laboratory, of the Joseph Sarwuan Tarka University Makurdi, Benue State.

#### **Materials**

The following materials were used for this study; Pumpkin seeds, sample container, n-hexene, electric blender, plastic bucket, sieving cloth for separating the oil from mixture. A refrigerator for monitoring pour point and cloud point. Thermometer for noticing flash point and fire point. A U-tube viscometer for determining kinematics viscosity. A test tube for pour point and cloud point determination. Electric stove and frying pan for heating of oil and automatic weighing balance, ethanol, diethyl ether, phenolphthalein, solution of NaOH and density bottle for determining specific gravity. Materials used for the Biodiesel Production includes; A measuring cylinder, A bowl or settling vessel after reaction, Syringe for removing biodiesel from the top layer of the separated mixture, Power stirrer or electric stirrer, Water bath and reaction vessel, Mixer or standard agitator, Potassium Hydroxide (KOH) as catalyst (2.2 kg), 0.1 litre of ethanol, 0.5 litre of pumpkin seed oil, Stop watch to note duration of reaction, A distiller for removing alcohol. Warm water to remove residual catalyst,

#### Methodology

#### **Gravimetric Analysis of Oil Seeds**

Pumpkin seeds were weighed using the weighing scale and the average weight of the seeds was recorded. Seeds were grinded to remove the oil seeds. The weights of the seeds and oil seed were noted and their respective average weights were recorded.

Oil to seed ratio =  $\frac{\text{weight of oil}}{\text{weight of seed}}$ 

#### Oil Extraction by Solvent Method

Pumpkin seeds were purchased from Jos, Plateau State. The seeds were dried and crushed into paste using an electric blender. The seed paste of 547.74 g was then taken to the laboratory where 1 litre of solvent (n-hexene) was added and mixed together for 48 hours to properly extract the oil from the seed paste. Sieving cloth was used to separate the n-hexene and seed oil mixture from the seed paste and then the n-hexene and seed oil mixture was put in a conical flask and placed on an electric stove for 10 minutes, where the n-hexene was evaporated leaving the seed oil.

#### **Oil Generation**

The raw pumpkin seeds were sun dried for one week, and then 547.74 g of pumpkin seeds were obtained. The seeds were dried to agitate oil before grinding. Adequate care was taken in controlling source of heat to avoid burning of seeds which may alter the Chemo-physical properties of the seeds. The dried seeds were crushed into paste using an electric blender. 547.74 g of the e was





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#### **Biodiesel Generation**

Potassium hydroxide (KOH) 0.02g was weighed and dissolved in 2 ml of ethanol and stirred properly using a standard/power mixer. The ethanol and KOH mixture was charged into a closed vessel and oil added. The system from here on was totally closed to the atmosphere to prevent the loss of ethanol. The reaction mixture was kept just above the boiling point of ethanol (around 71°C) to speed up the reaction and room temperature of 50°C for safety reasons, this was done for about 1 hour 15 minutes. Excess ethanol was used to ensure total conversion of the oil to esters. When the reaction was completed, the whole content was poured in a conical flask for 3 days. Two major products were separated, glycerol which is darker, much denser, and smaller in quantity and esters (biodiesel) which is amber – yellow in colour, less viscous and larger in quantity and settles on top while the glycerol settle below. The products were separated using syringe and stored.

### Analysis of Chemo-Physical Properties of Oil and Biodiesel

A small quantity of the specimen was taken in a beaker and perceived. The specimen odour is smoky or musky.

#### **Colour Test**

The sample was taken in transparent beakers and examined visually against sunlight. The colour of sample was described with reference to the standard description of colour.

#### **Free Fatty Acid Profile Determination**

2-2.5 (g) of the specimens was taken and put in a conical flask. 25 ml of diethyl ether mixed with 25 ml ethanol and 1ml phenolphthalein solution was prepared and dissolved in the specimen. 0.1 m Sodium Hydroxide (NaOH) solution was titrated against the mixture, shaking the mixture constantly until a pink colour that persisted for 15 seconds was obtained. The acid valued was then determined using equation below;

Acid value =  $\frac{\text{Titre. Value}(ml) \times 5.61}{\text{Weigh to f sample}}$ 

#### pH Determination

pH of oil sample was determined using the Labtech digital pH meter. The meter was calibrated with a natural solution, 25 ml of the specimen was taken in a beaker and the meter electrode inserted in them, one at a time. The pH of the specimen was read off the digital display screen of the pH meter.

#### **Specific Gravity**

Specific Gravity bottle was weighed on the electric weighing scale and the weight was recorded. The bottle was filled with sample and weighed. An equal volume of water was put in another specific gravity and its weight recorded. The API was also gotten.

#### **Determination of Specific Heat Capacity**

About 25 ml of specimen was warmed in a beaker until it was some degrees above room temperature (28°C). A copper calorimeter was weighed and its weight recorded. It was then filled to about one-third its capacity with the warmed oil and then reweighed, and the weight recorded.

The temperature of the oil was taken when it was about 6°C above the room temperature (34°C). A small dry piece of the ice was added until the temperature was 6°C below the room temperature. The calorimeter on its content was then weighed and the weight was recorded. The specific heat capacity of the specimen was determined using;

 $(M_2C + M_1C) (t_1-t_2) = MCw (t_2-0)$ 

 $M_1$  = Mass of calorimeter,  $M_2$  = Mass of oil (kg), M = Mass of ice (kg), Mw = Mass of water (kg) C = Specific heat capacity oil (Jkg-1k-1), C1 = Specific heat capacity of Copper (Jkg-1), L= Specific latent heat of fusion of ice (Jkg-1),  $t_1$ = Initial oil temperature (K),  $t_2$  = Final oil temperature (K)





#### **Flash Point**

The oil sample was kept in an aluminium container and heated over a Bunsen burner flame at the rate of 10°C per minute. A thermometer was placed inside the oil and at each 5°C, increase in temperature a small flame was passed over the oil surface until a flash appeared. The temperature this flash occurred was recorded.

#### Fire Point

The sample undergoing flash point determination was subjected to further heating after the flash point was obtained until the small flame passed over the oil surface at 5°C interval produced continuous burning flame for a minimum of 5 seconds. The fire point for the sample was then read on the thermometer and then recorded.

#### **Cloud Point**

The oil sample was kept in a test tube with a cork and thermometer; the test tube was put in a refrigerator so as to cool the oil sample. At interval, the test tube was checked until it turned cloudy. Temperature that cloudiness occurred was recorded for the oil sample.

#### **Pour Point**

The oil sample undergoing cloud point determination was further cooled. The test tube was tilted at interval to check for oil movement. When no oil movement was observed when the test tube was placed horizontally for 5 seconds, the temperature was noted and recorded as the pour point of the sample.

#### **Moisture Content**

Preliminary moisture test was carried out by heating a little quantity of oil sample in a container up to 110°C over a hot plate. This temperature was maintained until a crackling noise indicated the presence of water; a final test was carried out by percentage weight loss. The sample is taken in a container and weighed before drying in the electric oven between 105°C and 110°C the container was weighed continually until constant weight was obtained. The percentage water loss was determined for the sample.

#### RESULTS AND DISCUSSION

The gravimetric analysis of pumpkin seeds (Table 1) showed an average seed weight of 547.74 g, yielding 44.17 g of oil, which corresponds to an oil yield of 8% and a biodiesel production rate of 63%. Although the oil yield is relatively low, the high biodiesel yield indicates that pumpkin seed oil is a viable feedstock for biodiesel production. This yield is comparable to other oilseed crops commonly used in biodiesel manufacturing, such as rapeseed and soybean (Rezig *et al.*, 2012).

Table 1. Gravimetric Analysis of Pumpkin Seed Oil

Parameters	Values
Average weight of seed (kg)	547.74
Average weight of oil produced per batch (kg)	44.17
Percentage oil yield (%)	0.08
Percentage of biodiesel production per batch (%)	63

The physicochemical properties of pumpkin seed oil and its biodiesel (Tables 2-4) revealed significant differences (p < 0.001). Biodiesel exhibited lower density (0.84  $\pm$  0.01 g/cm³) and free fatty acid content (28.54  $\pm$  0.12%) compared to oil (0.93  $\pm$  0.01 g/cm³ and 32.27  $\pm$  0.08%, respectively). In contrast, biodiesel had a significantly higher pH (7.84  $\pm$  0.01) than oil (5.81  $\pm$  0.02). These results confirm the successful reduction of free fatty acids during transesterification, improving fuel quality.

Cold flow properties were also enhanced in biodiesel, with lower cloud point ( $-7.00 \pm 0.00^{\circ}$ C) and pour point ( $-8.00 \pm 1.00^{\circ}$ C) compared to oil ( $20.00 \pm 1.00^{\circ}$ C and  $17.00 \pm 1.00^{\circ}$ C, respectively). These improvements, which were highly significant (p < 0.001), suggest that pumpkin seed biodiesel would perform better in cold conditions than the raw oil. Furthermore, while biodiesel exhibited a higher flash point ( $150.00 \pm 20.00^{\circ}$ C) than oil ( $130.00 \pm 1.00^{\circ}$ C), the difference was not statistically





significant (p > 0.05). However, the fire point of biodiesel (170  $\pm$  1.00°C) was significantly higher than that of oil (140.00  $\pm$  1.00°C, p < 0.001), demonstrating improved safety in handling and storage.

Table 2. Results of Flash Point, Fire Point, Pour Point of Oil and Biodiesel.

Oil Samples	Flash point (° C)	Fire point (° C)	Cloud point (° C)	Pour point (° C)
Seed Oil	130	140	20	17
Biodiesel	150	170	-7	-8

**Table 3**. Density and API Gravity Determination for Oil Sample and Biodiesel.

Parameter	Sample	Replicate 1 g/cm <sup>3</sup>	Replicate 2g/cm <sup>3</sup>	Replicate 3g/cm <sup>3</sup>	Mean
Density	Seed Oil	0.92	0.93	0.93	0.92
	Biodiesel	0.84	0.83	0.84	0.83
API gravity	Seed Oil	3.63	3.62	3.62	3.62
	Biodiesel	3.98	3.99	3.98	3.98

Table 4. Free Fatty Acid Content and PH Test Values of Seed Oil and Biodiesel

Parameters	Sample	Replicate 1	Replicate 2	Replicate 3	Mean
Free Fatty Acid (%)	Seed Oil	32.26	32.25	32.2	32.2
	Biodiesel	28. 61	28.62	28.40	28.54
Ph	Seed Oil	5.81	5.82	5.79	5.81
	Biodiesel	7.83	7.84	7.85	7.84

The Thin Layer Chromatography (TLC) test confirmed the production of fatty acid methyl esters (FAMEs), with the biodiesel showing a pure single spot that aligned with reference standards (Table 5). The obtained retention factor (Rf) values of 0.12 and 0.50 for biodiesel and glycerol, respectively, validate the successful transesterification reaction.

**Table 5**. TLC for determination of FAME and Biodiesel Purity

Sample	Distance Travelled by Solvent	Distance Travelled by Solute 1	RF Values 1	Distance Travelled by Solute 2	RF Values 2
Oil	7.8	1.01	0.12	4.0	0.5
Biodiesel	7.8	1.00	0.12	-	-
Glycerol	7.8	-	-	4.0	0.5

Key: TLC = Tin Layer Chromatography, FAME = Fatty Acid Methyl Ester, RF = Retention Factor = Distance travelled by sample

Distance travelled by solvent

When compared with ASTM D6751 and EN 14214 standards (Table 6), the biodiesel properties; density (0.83 g/cm³), flash point (150°C), cloud point (-7°C), and pour point (-8°C) all fell within acceptable limits, underscoring its suitability as an alternative fuel. Additional analysis (Table 7) confirmed that the biodiesel possessed desirable characteristics, including amber yellow colour, smoky odour, pH of 7.84, and very low moisture content (0.02%).

The comparison of the Physicochemical Properties between the Oil and Biodiesel Extracted from Pumpkin Seeds (Table 8), the density (g/cm³) was 0.93 and 0.84, pH of 5.81 and 7.84, flash point (°C) of 130 and 150, cloud point (°C) of 20 and -7, pour point (°C) of 17 and -8, and fire point (°C) of 140 and 170 for oil and biodiesel, respectively. Analysis of variance revealed that the oil and biodiesel physicochemical properties are significantly different at 95% confidence level. This is different from the report on pour point (4°C), flash point (169°C), cloud point (8°C) and fire point (180°C) by Awulu *et al.*, (2018) for tigernut oil extracted using chemical method. Fatty acid profile of pumpkin seed oil, dominated by linoleic acid (36.2–60.8%) and oleic acid (21.0–46.9%), as



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reported by Procida *et al.* (2013), further supports its potential as a biofuel feedstock. Other studies similarly report palmitic and stearic acids present in smaller amounts, with total unsaturated fatty acids often above 70-80% (Stevenson *et al.*, 2020).

**Table 6.** Comparison of produced biodiesel with Standards

Parameters	Units	Biodiesel	ASTM Standard	EN Standard
Density	g/cm <sup>3</sup>	0.83	0.85 - 0.9	0.85 - 0.9
Flash Point	° C	150	130 - 191	120 min.
<b>Cloud Point</b>	° C	-7	-3 – 12	
<b>Pour Point</b>	° C	-8	-15 - 10	
<b>API Gravity</b>	-	3.98	-	-

**Key:** ASTM = American Society for Testing and Materials, EN = European Standard

**Table 7**. Summary of Physiochemical Analysis of Bio-diesel from pumpkin seed oil

S/N	Property	X
1	Colour	Amber Yellow
2	Odour	Mosky or Smoky
3	Density g/cm <sup>3</sup>	0.83
4	API Gravity	3.98
5	PH Value	7.84
6	Free Fatty Acid (FFA %)	28.54
7	Flash Point (°C)	150
8	Fire Point (°C)	170
9	Cloud Point (°C)	-7
10	Pour point (°C)	-8
11	Moisture content (%)	0.02

**Table 8**: Comparison of Physicochemical Properties between Oil and Biodiesel Extracted from Pumpkin Seeds (Mean ± SD)

Parameter	Oil	Biodiesel	t-value	P value
Density (g/cm <sup>3</sup> )	$0.93\pm0.01^a$	$0.84\pm0.01^{b}$	-19.09	< 0.001
Free fatty acid (%)	$32.27\pm0.08^a$	$28.54 \pm 0.12^{b}$	-43.39	< 0.001
pН	$5.81 \pm 0.02^{b}$	$7.84 \pm 0.01^a$	192.90	< 0.001
Flash point (°C)	$130.00 \pm 1.00$	$150.00 \pm 20.00$	1.55	> 0.05
Cloud point (°C)	$20.00\pm1.00^a$	$-7.00 \pm 0.00^{b}$	-46.77	< 0.001
Pour point (°C)	$17.00 \pm 1.00^{a}$	$-8.00 \pm 1.00^{b}$	-30.62	< 0.001
Fire point (°C)	$140.00 \pm 1.00^{b}$	$170\pm1.00^{\rm a}$	36.74	< 0.001

Mean  $\pm$  SD with different superscripts in a row are significantly different

#### CONCLUSION AND RECOMMENDATION

The study revealed the following conclusion:

- 1. Oil was successfully extracted from pumpkin seeds using solvent extraction method and 44.17 g oil yield was gotten from 547.74 g of pumpkin seeds which represents 8% oil yield from 100% pumpkin seeds which shows a low oil yield.
- 2. The physicochemical properties was analyzed and had Flash point (130°C), Fire point (140°C), Cloud point (20°C), Pour point (17°C), Density (0.92 g/cm³), API Gravity (3.62), Fatty Acid (32.2%) and pH (5.81).
- 3. Biodiesel was produced from the extracted pumpkin seeds oil and it was revealed to be pure when compared with ASTM D6751 and EN 14214 standards.

Based on the experimental results obtained, the following recommendations can enhance the viability of pumpkin seed oil biodiesel production;





- 1. Further research should explore alternative extraction methods and agronomic improvements to enhance pumpkin seed oil yield.
- 2. Engine performance tests and life cycle assessments are recommended to validate its sustainability and practical application as a biodiesel feedstock.

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