

EXTRACTION, CHARACTERIZATION AND TRANSESTERIFICATION OF GMELINA ARBOREA SEED OIL USING CLINKER AS CATALYST

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ABSTRACT

Gmelina arborea seed oils were extracted, characterized and transesterified using methanol in presence of a heterogeneous catalyst (clinker). Effects of particle size, temperature, extraction time and volume of solvent on the extraction yield were studied. Results showed that the optimum oil yields were 52.60% for gmelina seed oil (GSO)) at 65°C and 0.150 mm particle size. Physicochemical characterization of the purified oil revealed relatively low acid values, low iodine values, low peroxide values, low ash contents but relatively high specific gravity, kinematic viscosities and saponification values. The viscosity, flash point, density, pour point and cloud point of the produced biodiesel were measured and compared to the properties of automotive gas oil. The biodiesel produced from gmelina oil has properties within the acceptable range for automotive gas oil while some Neem biodiesel properties were not within the range. The gmelina biodiesel properties were viscosity (5.45 mm²/s), flash point (146 °C), density (820 kg/m^3), pour point (4°C) and cloud point (5 °C). Fourier transform infra-red (FTIR) spectroscopic analysis confirmed the presence of major functional groups in the oil and fatty acid methyl ester (FAME), notably hydroxyl, carbonyl, olefinic and methylenic. The constituents of the fatty acid composition of the oil and its biodiesel were determined by gas chromatographymass spectrometry (GC-MS). The oil sample extracted is a good feedstock for biodiesel production and other industries. The properties of the Gmelina oil methyl ester (GOME) are significant when compared with standards of ASTM D6751 and EN 14214.

Keywords: biodiesel, characterization, clinker, gmelina arborea, non-edible oil, transesterification

INTRODUCTION

The fast growing demand for energy and industrial raw materials from crude oil and the resultant depletion of crude oil with adverse environmental problems and very fluctuating nature of its

prices make necessary the need to explore alternative sources of fuel and industrial raw materials. Concerted effort has been made in producing alternative renewable energy like biodiesel which is also known as Fatty Acid Methyl Ester. Fatty Acid Methyl Ester is an alternative biofuel produced through transesterification of triglycerides (TGs) or the esterification of Free Fatty Acids (FFAs) with methanol [1]. Biodiesel possesses all the favourable characteristics of diesel as it is renewable, abundant, biodegradable, non-toxic and carbon neutral since no net amount of carbon is released to the atmosphere. One important feature is that they can be blended with any fuel obtained from petroleum whose properties conform to the end use. For example, biodiesel with diesel fuel, bioethanol with gasoline can all be blended.

Biodiesel has octane number which allows cold start and less idle noise [2]. According to Ramadhas et al [3], biodiesel has no sulphur content, no storage difficulties and have good lubricating properties.

Vegetable oils and fats are the main feedstock for biodiesel and an economical supply that is sustainable is an important factor [4]. Many researchers used edible oils for example melon and palm oil to produce biodiesel but the fluctuating prices of edible oils and increasing demands for nutritional needs have made Gmelina seed oil to be the raw material of choice. In order to overcome these issues, there is a dire need to produce biodiesel using second generation vegetable oil and the use of catalyst that reduces energy requirement in transesterification as well as cost effective.

Gmelina arborea is a fast growing tree which grows on different localities and prefers moist fertile valleys. They attain moderate to large height up to 40 m and 140 cm in diameter. It is occurring naturally throughout greater parts of India at altitudes up to 1500m. It also occurs naturally in Thailand, Cambodia, Nigeria, Malaysia and Vietnam [5].

This work aims to study the extraction, characterization and transesterification of oil from Gmelina seeds using clinker as catalyst.

MATERIALS AND METHOD

The raw materials used to carry out this research are the gmelina seeds. The seeds were collected from Auchi polytechnic Edo State, Nigeria and subsequently sorted into good and bad ones.

Cement clinker was collected from Dangote Cement Plant, Obajana, Kogi State, Nigeria. Chemicals used in this study were analar grade and obtained from Sigma-Aldrich, Germany.

Preliminary Procedure for the Gmelina Seed Oil Extraction

The good seeds were washed and sun dried for three days. Later, they were deshelled, sundried for two days. The mechanically crushed kernels were stored in a polythene container. Oil was extracted from known weights of crushed seed samples using soxhlet extractor and hexane as solvent, after which the oil was filtered and oil yield calculated.

Oil Extraction Procedure and Factors That Affect Oil Yield

The oil extraction, transesterification and characterizations were carried out at Auchi Polytechnic, Springboard Research Laboratories, Awka; and at Ahmadu Bello University, Zaria. The crushed seeds were extracted using soxhlet extractors with n – hexane as the solvent. The extraction was carried out considering four factors that affect the yield of oil extracted. These factors are particle size, temperature, extraction time and volume of solvent used. The extracted oil for each factor was then collected and weighed. Further extractions were carried out at different extraction process variables and the oils obtained were subjected to pre-treatment like degumming, neutralization and dehydration.

Determination of physico-chemical properties of the oil extracted

The properties determined were acid value, saponification value, kinematic viscosity, peroxide value, unsaponifiable matter, refractive index, flash plant, phenolic content, color, P^H value, ester value, Iodine value, Moisture content, specific gravity, odour were determined using standard methods [6].

Preparation of Limestone based (Clinker) Catalyst

The cement clinker was washed with 1% aqueous solution of sulphuric acid to remove dirt and stains on the surface of the shell. The cement clinker was crushed and ground using grinding machine to reduce the particle size to around 150 µm to ensure a large surface area per unit mass. Chemical composition of the clinker was obtained from X-ray florescence with in-built XRD (ARL 8660S) and this indicates a significant CaO content useful for transesterification process. The catalyst activation was performed by soaking with methanol followed by calcination at 700 °C for 6 hours in the furnace.

FTIR Test for the Oil and FAMEs (FTIR spectroscopic analysis)

FTIR was used to characterize the structural functionalities in the oil and FAME. FTIR spectrum of oil (in KBr pellets) was recorded with a Shimadzu 8400S FTIR spectrophotometer over the range 4500-350 cm⁻¹.

GC-MS Test for the Oils and FAMEs (Fatty Acid Profile Analysis)

The oil was also characterized in terms of its fatty acid profile. Fatty acid composition was determined after converting the fatty acids in the oil to fatty acid methyl esters (FAMEs). The FAMEs were prepared according to the modified ISO method (BS EN ISO 5508, 1995). The separation of the FAMEs was performed on a GC-MS QP2010.

Transesterification (Biodiesel Production)

About 1 Litre of the oil sample was measured out into a three mouth flask having a cooling system to circulate the methanol. The oil samples were heated up to 65 °C while undergoing stirring by a combination of hot plate and magnetic stirrer. Previously prepared alcohol-catalyst solution was added to the oil in the flask at the same temperature, while stirring the mixture. The alcohol catalyst solution was added while the oil was being stirred. When the reaction was completed, the catalyst was filtered out and the end product was poured into a separating funnel and was left for 12hours to ensure total separation. The product mixture was separated into two liquid layers after settling: crude ester layer (FAME) on top and glycerol at the bottom. The lower layer which is glycerol was drawn out and the excessive amount of methanol was evaporated. The top layer (FAME) was washed three times with distilled warm water to remove methanol and impurities. The transesterification reaction was performed at 65°C and stirred for 2 hours, optimum catalyst concentration of 5% (w/w) [7].

During the transeterification of the oil for methyl ester (biodiesel) production, effect of temperature, contact time, catalyst particle sizes and oil to methanol ratio were investigated. The standard procedures as reported by Meda et al [7] were employed.

Determination of Physico-Chemical Properties of Gmelina Seed Oil Biodiesel

The properties determined using standard methods [6] were acid value, saponification value, kinematic viscosity, peroxide value, unsaponifiable matter, refractive index, flash plant, phenolic content, color, ph value, ester value, iodine value, moisture content, specific gravity and odour.

RESULTS AND DISCUSSION

The Effect of Extraction Temperature on the Extraction Yield

Figure 1 shows linear relationships between temperature and the oil yield, the higher the temperature the higher the oil yield. It is observed that oil yield increased initially with increasing temperature and then subsequently decreased with further increase in temperature. The optimum oil yield in gmelina seed sample was obtained at 65 °C. The reason for the increase in the yield is that increase in temperature is believed to facilitate the rupturing of oil cell walls, creating a void which serves as migratory space for the contents of the oil bearing cells. Increase in temperature also lowers the viscosity of the oil and coagulates protein, thus facilitating the release of the oil out of the cells into the inter-kernel void. It was observed that at 90 °C the colour of the oil turned dark brown.





The Effects of Particle sizes on the Extraction Yield

Figure 2 shows the variation of gmelina particle sizes respectively on the oil yield. From the figure, it could be seen that decrease in particle sizes brought about increase in oil yield and this made the optimum size of 0.150 mm to give the maximum oil yield of 52.60%. This agrees with

the fact that size reduction increases the surface area and increases number of ruptured cells resulting in a high oil concentration at the particle surface which in turns increases the yield of the seeds. Sayyar et al. [8] while investigating the extraction of oil from *Jatropha* seed posited also that larger particles present smaller contact surface areas and are more resistant to solvent entrance and oil diffusion. However, linear relationship exists between the two variables. Increase in the particle size leads to decrease in yield and vice - versa. The regression analysis of the result of gmelina seed, Table 1 gives the equation y = 52.65-15.14x, and the variables are highly correlated ($R^2 = 0.910$).



Figure 2: Effect of particle sizes on extraction yield of gmelina seed oil (GSO) when temperature $= 68^{\circ}$ C, Time = 60min and volume of solvent = 250 ml.

The Effects of Volume of Solvent on the Extraction Yield

Figure 3 shows the effect of volume of solvent on oil yield of gmelina samples. Figure 3 shows that the optimum volume of solvent to give the maximum oil yield for 100 g of the ground sample is 350 ml. The oil yield at this point for gmelina seed sample is 44.20% but as the volume of solvent reduces below this point, the oil yield decreases and when the volume increases beyond this point, the yield remains unchanged. It was also observed that when the ratio of weight of sample to volume of solvent increased beyond 1:5, the yield began to decrease. This is due to higher energy and longer time requirements to heat the solvent to boiling point.



Figure 3: Effect of volume of solvent on extraction yield of gmelina seed oil (GSO) when temperature = 68° C, Time = 60min and Particle sizes = 0.355mm

The Effects of Time on the Extraction Yield

Figure 4 shows that at extraction time of 70 minutes, the maximum oil yield of 45.42% was obtained at optimum time of 60minutes. It is important to note that below and above this time the oil yield reduces.

Consequently, the regression analysis carried out show significant relationships between the variables and the R^2 values obtained in each case is good enough to describe the degree of variability between the variables, high correlation also exist between the variables in each case.



Figure 4: Effect Time on extraction yield of gmelina seed oil(GSO) when temperature = 68° C, volume of solvent= 250ml and Particle sizes = 0.355mm

The regression equations developed are good enough to predict the values of Y when the value of X is known (see Table 1).

0.010	D		D ²
S/NO	Parameters	Regression Equation	R ²
1.	Particle Size Versus Yield	y=52.65-15.41x	0.910
2.	Temperature Versus Yield	y=35.78+0.160x	0.844
3.	Time Versus Yield	y=31.44+0.216x	0.929
4.	Volume of Solvent Versus Yield	y=37.64_0.0184x	0.656

Table 1: Regression equation for Gmelina seed oil

Characterization of calcined clinker

The elemental composition analysis of the calcined clinker sample with XRF spectroscopy was tabulated as shown in Table 2 below.

XRF analysis showed that the sample was composed mainly of calcium oxide with 66.81wt.%. These results indicated that all calcium carbonate (CaCO₃) in the samples was completely transformed to CaO. So, large quantity of Calcium (Ca) and Oxygen (O₂) were observed in the sample.

Table 2: Clinker analysis with XRF-XRD.

Element CaO	SiO ₂ Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃ K ₂ O	Na ₂ O	P_2O_5	TiO ₂
Wt. % 66.14 2	22.02 5.96	3.50	0.68	0.44 0.79	0.12	0.03	0.32

Characterization of Gmelina seed oil, and Biodiesel

From Table 3, the acid value of gmelina oil is 5.04. The acid value gives an indication of the amount of FFA present in the oil at the time of the test. Low acid value is an indication of good non-degraded state of the oil and is within limits for industrial useful oils. Low FFA also suggests low levels of hydrolytic and lipolytic activities in the oil. The acid value also gives a measure of the extent to which the constituent glyceride has been decomposed by lipase action. The maximum limit of acid value specified in the biodiesel fuel standards such as ASTM D6751 [9] and EN 14214 [9] is 0.50 mg of KOH/g. The acid value of biodiesel obtained is 0.13 mg of KOH/g which is far below the specified maximum limit and the free fatty acid values are 0.065 mg of KOH/g.

The saponification value provides information on the amount of KOH that will saponify the oil. Table 3 also shows that the saponification value of gmelina oil is 108.99. The results obtained

compared favorably with the saponification value of palm oil (196-205), olive oil(185-196), soy oil (193) [10]. Also, the biodiesel standards such as ASTM D6751 and EN 14214 have no specification for the saponification number. The biodiesels obtained from soybean, sunflower, palm, canola, and Jatropha curcas oils have saponification numbers 201, 200, 207, 182 and 202 mg KOH/g respectively [11]. In the present study, saponification numbers of biodiesels prepared from gmelina seed oil was calculated and found to be 182.3 mg KOH/g which is comparable to the values of biodiesels obtained from soybean, sunflower, palm, canola and Jatropha curcas oils. The pH value of 10.6 obtained for GSO and compare favourably with those obtained for castor seed oil (6.8) and luffa cylindrical (3.93). This value is an indication of the presence of reasonable amounts of free fatty acid and the advantageous utilization of the oils in soap making. The iodine value provides information on the measure of unsaturation of oils. In general, the greater the iodine value, the higher the degree of unsaturation and the higher the tendency of the oil to undergo oxidative rancidity Table 3 shows the iodine value for GSO to be 41.05. The low iodine value of the sample also indicates that the oils are non-drying types of oil. Although the iodine values are low, the oil have proven to be good source of raw materials for biodiesel production even against that, the higher the iodine value the more the number of unsaturated double bonds present in the molecular structure and the less the viscosity of the oil. Also an iodine value of GOME is 38.63 this is within the accepted standard [8]. The peroxide value of 9.75 meq/g was obtained for the GSO. Peroxide value depends on a number of factors like oxidation by oxygen, extraction methods and storage. The low peroxide values suggest that gmelina oils are stable to oxidative degradation caused by over exposure to oxygen, heating and improper storage.

From Table 3, the pour point of GSO is 4°C. Also the pour point of GOME is 4°C (Table 3). This indicates the temperatures at which these oil seizes to flow, it therefore shows that, in a temperate climate below the pour points of these oil, they are not suitable for use as biodiesel as it can lead to incomplete combustion in diesel engines.

The moisture contents of GSO (8.2%) was very high, far exceeding the stipulated ASTM standard (0.05%) for biodiesel production, 0.02% was detected in GOME. This high moisture content creates problem in transesterification, reduces shelf life and may cause corrosion in internal combustion engine

The cetane numbers of GOME (48.0) is within ASTM D6751 standard while GSO (45.0) fall outside the range(Table 3). Cetane number (CN) is a dimensionless indicator that characterizes ignition quality of fuels for compression ignition engines (CIE). Since in the CIE burning of the fuel air mixture is initiated by compression ignition of the fuel; the number is a primary indicator of fuel quality as it describes the ease of self-ignition. Table 3 shows the flash point of the GOME to be 146°C, which is within the acceptable range of the flash point of ASTM D6751 [9]. The yield of biodiesel from Gmelina arborea seed oil was 92.18 wt.% at 60°C within 2 hours.

S/N	PARAMETERS	UNIT	BIODIESEL ASTM	GSO	GOME
			D6751		
	Acid Values	mgkOH/g	< 0.50	5.04	0.13
	рН	-		10.61	6.50
	Iodine Value	-		41.05	38.63
	Saponification Value	mgkoH/g		108.99	182.3
	Refractive Index at 28 ⁰ C	-		1.45	1.472
	Free Fatty Acid	mgkoH/g		2.52	0.065
	Colour	-		Golden	Greenish
				yellow	yellow
	Specific Gravity	-	0.88	0.89	0.82
	Kinematic Viscosity (a 40 ⁰ C)	mm ² /s	1.9-6.0	36.63	5.45
	Cetane Index	-	48-65	45.0	48.0
	Ash Content	%		3.88	1.39
	Flash Point	°C	100-170	324.60	146
	Peroxide Value	Meq/kg		9.75	-
	Moisture Content	%	0.05	8.20	0.03
	Fire Point	^{0}C		383.06	
	Yield	%		52.60	92.5
	Physical State at room temp.	-	Liquid	Liquid	Liquid
	Pour Point (⁰ C)	^{0}C	-15 to 10	4.0	4.0
	Cloud Point (⁰ C)	⁰ C	-3 to 12	7.0	5.0

Table 3: Physiochemical properties of GSO, GOME, and Biodiesels, ASTM D6751

Fourier-transform infrared spectroscopy analysis of Gmelina Seed oil

FT-IR spectra (4000-400cm⁻¹) of the gmelina seed oil (GSO) and its biodiesel (GOME) in Figures 5 and 6, reveal major functional group absorption bands, notably for hydroxyl, carbonyl, olefinic, methylenicetc groups. The GSO shows two small peaks at 3483.21 cm⁻¹ and 1625.94cm⁻¹ corresponding to the stretching and bending vibration of O-H bonds of water

molecule of the unsaturated fatty acid in the oil. The FT-IR spectra of the GSO and its biodiesel are very similar to each other as a result of the similarities in the chemical natures of the triglycerides and methyl esters. But, very small differences were observed where the peaks appeared at 846.47, 1391.93, 1916.09 and2130.46cm⁻¹ in the GSO were shifted to 833.63, 1377.48, 1935.14, and 2119.43 cm⁻¹ in the biodiesel respectively. Whereas, the disappearance of the peaks at 1625, 2458 and 2977 cm⁻¹ from the spectrum of the GSO and appearance of new peaks in the biodiesel sample at 1219cm⁻¹,2490cm⁻¹ and2863cm⁻¹ clearly show the conversion of GSO into biodiesel. Also, the absence of a broad peak in the 3199- 3404cm⁻¹ region of GOME, reveals that OH groups is negligible, and this suggests that the synthesized biodiesel possesses very low water contents as observed in other methods stated earlier.



Figure 5: FTIR spectral of Gmelina Seed Oil



Fig.6: FTIR Spectral of Gmelina Seed Biodiesel

Fatty acid profile of GSO, and GOME

Figure 7 shows seven peaks representing seven major constituents of fatty acid in GSO's chemical composition. Peak identification involved noting peak retention time and comparing with authentic reference standards for individual FAMEs. Identified peaks were used to determine fatty acid composition, since peak area (recorded by data

processing unit of the GC) is proportional to the concentration of the constituent FAME represented by the peak. Peak 3 (retention time 9.680 min) represents myristic acid (55.021%) as the major constituent fatty acid in the oil. Other fatty acids are (peak6) palmitoleic acid (23.501%), (peak5) Oleic acid (13.971%), (Peak 2) Lauric acid (3.180%), (peak4) Margaric acid (3.012%), (peak) Arachidic acid (0.859%) and (peak1)Palmitoleate (0.455%). The detailed fatty acid profile for the GSO is given in Table 4. Saturated fatty acid (SFA) for the oil sample in this study was 62.072%, while the unsaturated fatty acid (UFA) was 37.928% of the oil sample.

On the other hand, fatty acid profile of the biodiesel prepared from Gmelina seed oil (GSO) was determined by GC-MS analysis with eight peaks (Figure 8). The Biodiesel consists of 6.728 % of methyl palmitoleate (C16:2), 25.687% of methyl Laurate (C12:0), 0.761% of methyl myristate(C14:0), 15.038% of methyl linoleate (C18:2),26.027% of methyl palmitate (C16:0), 7.632 % of methyl oleate (C18:1), 17.961.% of methyl palmitoleate (16:1), and 0.166.% of methyl arachidate (C20:0). The Gmelina oil methyl ester(GOME) contains about 52.641 % of unsaturated and 47.359 % of saturated fatty acids (Table 5).



Wavenumber cm⁻¹





Wavenumber (cm⁻¹)

Figure 8: Gmelina Biodiesel fatty acid profile

Component	Name	Concentration PPM	% Concentration
C16.2	Palmitoleate	0.1873	0.455
C12	Lauric Acid	1.3087	3.180
C14	Myristic Acid	22.6451	55.021
C18:2	Linoleic Acid	1.2398	3.012
C16	Palmitic Acid	5.7504	13.971
C18:1	Oleic Acid	4.6724	23.501
C20	Arachidic Acid	0.3534	0.8599
	Saturated fatty acid		62.070
	Unsaturated fatty acid		37.930
Total		148.5451	

Table 4: Gmelina seed oil fatty acid profile

Table 5: Fatty acid profile for gmelina seed biodiesel					
Component	Name	Concentration PPM	% Concentration		
C16.2	Methyl Palmitoleate	9.9948	6.728		
C12	Methyl Laurate	38.1572	25.687		
C14	Methyl Myristate	1.1297	0.761		
C18:2	Methyl Linoleate	22.3381	15.038		
C16	Methyl Palmitate	38.6612	26.027		
C18:1	Methyl Oleate	11.3370	7.632		
C16:1	Methyl Palmitoleate	26.6808	17.961		
C20	Methyl Arachidate	0.2463	0.166		
	Saturated fatty acid		52.640		
	Unsaturated fatty acid		47.360		
Total		148.5451			

Table 5. Party actu profile for ginefina secu biouleser	Table 5:	Fatty	acid	profile	for	gmelina	seed	biodiesel
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CONCLUSION

The result of the analysis of the factors that affected oil yield of the gmelina seeds shows that the smaller the particle size, the higher the oil yield. Also increase in temperature, time volume of solvent all increases the yields of the oils. The optimum yield of gmelina oil is 52.60wt% while that of neem is 38.5wt%. From the oil yield of the seeds, it can be concluded that gmelina seed oil has high oil yield. The heterogeneous catalyst, clinker showed a good conversion (92.18%) FFA gmelina seed oil to biodiesel. The results revealed that the catalyst is not negatively affected by moisture and free fatty acids and can be recycled very easily without a significant loss in its activity. The catalyst can be recovered by filtering out the catalyst and washing with acetone for reuse. The characterization results of the oil seed and biodiesel produced compare favourable to ASTM D6751 standard; hence the fatty acid methyl ester (FAME) of Gmelina seed oil can be used in diesel engine.

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