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Review Article

Novel Soybean soapstock Biodiesel Synthesis through Esterification/Transesterification Reactions using Multiple Solvents

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KEYWORDS

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ABSTRACT

Soybean soapstock (SS) a by-product from the soybean processing food chain can serve as a viable feedstock for biodiesel production. This viability can be attributed to its low costs and high conversion rates. Biodiesel from this feedstock requires both esterification and transesterification reactions, using methanol and n-hexane as solvents. Determination of the component fatty acids was carried out using gas chromatography while functional groups were obtained using the FTIR (Fourier transform infrared spectroscopy) for both feedstock and biodiesel. The produced biodiesel had a higher percentage of unsaturated fatty acids when compared to its feedstock (soybean soapstock) with oleic acid (26.045%) and linolenic acid (22.344%) constituting the bulk of the unsaturated fatty acids. This conversion of saturated fatty acids into unsaturated fatty acids as confirmed in the GC analysis highlights the effect of transesterification in biodiesel production. The effect of transesterification was further confirmed by the presence of cyclic ester compounds and carbonyl groups as observed in the FTIR analysis. Desirable fuel properties for the produced biodiesel were confirmed by comparing its physico-chemical properties with standard fuel properties. A high biodiesel yield of 96.8wt% and positive economic indices (38% ROI) obtained further validates soybean soapstock as a viable feedstock for biodiesel production.

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1. Introduction

High energy costs and environmental concerns arising from fossil fuel use have necessitated the need for more research into alternative energy sources such as solar, wind, hydro-electricity, geothermal and biofuels [12]. Though most of these renewable energy sources have received significant attention over the years, biofuels rank as one of the best in terms of sustainability based on statistics by the International Energy Agency [7]. Biodiesel, a fuel derived from oils in plants and fats of animals has gained significant attention in recent years because of its renewability and environment-friendliness compared to fossil diesel. Biodiesel has been mainly produced from edible vegetable oils including soybean, palm, and rapeseed oil. Transesterification has over the years been established as the most viable method of producing biodiesel, and mostly through the use of acid or base catalysts [21].

With the high costs of vegetable oils, the use of non-edible and cheaper oils such as waste cooking oils and soapstocks have come to the fore as feedstocks for biodiesel production [10,3]. Soybean soapstock, a non-edible oil has been investigated to have favorable qualities as feedstock for biodiesel production because it is relatively centralized, readily available and reasonably cheap [13]. Soybean soapstock (SS) generated is about 6% of the volume of the crude oil produced with its price only one-tenth of the refined oil [23].

Nakyung, C.(2014) produced biodiesel from soybean soapstock (SS) by carrying out esterification and transesterification reactions using a basic catalyst for esterification while conventional acidulation was applied to separate high-acid acid oil (HAAO) having over 96% free fatty acid from water and other substances. [16] also produced biodiesel from soybean soapstock using a solid acid catalyst. Hydrolysis using potassium hydroxide (KOH) and acidulation by sulfuric acid were used to obtain biodiesel. The high cost of acidulation which involves high temperatures and pressure and the high cost of the catalysts involved in the works by [15,16] however necessitated the need for an alternative production route. This work will however seek to produce biodiesel through the transesterification of soyabean soapstock using 2 solvents (methanol and n-hexane) to boost biodiesel yield and quality and thus by-passing the high cost of acidulation carried out in previous works.

2. Materials

2.1. Reagents

Methanol (primary solvent), sodium hydroxide (NaOH), , sulfhuric acid, magnesium trisilicate, sodium sulfate, phenolphthalein and n-hexane (secondary solvent).

2.2. Equipments

Centrifuge, Weighing balance, Oven, Heating mantle (98-I-B Series), HH-S thermostatic water bath, Gas chromatography coupled with FID/ECD and Buck scientific infra-red spectrophotometer.

2.3. Collection of Sample

Contaminated soybean soapstock was sourced from Phinomar Nigeria Limited, an integrated poultry and feed processing plant in Ngwo, Enugu State, Nigeria. The soybean soapstock was collected from the soybean mill in a clean container. Contaminants and debris were first removed through sedimentation and decantation and then centrifuged for 7 minutes, at 3,000 rpm and 25 oC. After separation, the top layer which is the acid oil (AO), commonly regarded as soapstock was collected for use. The bottom layer which is mostly undesirable oils and contaminants are discarded.

2.4. Characterization of Feedstock and Soybean Soapstock Biodiesel

Gas chromatography (GC) was used to determine the fatty acid profile using AOAC's official method [1]. The GC was calibrated by injecting methyl ester standards. Good separations were achieved by diluting the samples (n-hexane collected) in a small amount of ethyl acetate with hydrogen serving as the carrier gas. The oven temperature was increased from 80 oC to 340 oC at a rate of 6 oC/min. The identification of peaks was done by comparing their retention time and mass spectra with the mass spectra library (NIST05s LIB) [5]. The gas chromatography analysis was carried out for both the soybean soapstock and its biodiesel.

2.5. FTIR Analysis

FTIR analysis was performed to monitor the functional groups in the soybean soapstock and the biodiesel produced using an FTIR spectrometer (Shimadzu, model No: 3116465).

2.6. Biodiesel Production

Soybean soapstock and methanol were mixed in equal proportion. Sulfuric acid was then added in the ratio of 1:10 to the solution. The solution was heated to 60 oC for 80 mins to achieve esterification. The esterified oil was then separated using a separating funnel. The oil was reacted with methanol and n-hexane in the ratio of 1:3:3 respectively. 2% Sodium hydroxide (NaOH) catalyst (2%) was added and the mixture stirred at 300 rpm on a heating mantle for 50 mins at 55oC. The optimized reaction parameters used (reaction temperature, catalyst concentration, reaction time, solvents ratios and agitation speed) were obtained using response surface methodology (RSM). Sedimentation and decantation was used to separate the top layer (biodiesel) from the bottom layer (glycerol) with the aid of a separating funnel.

2.7. Physico-Chemical Analysis

Standard methods were used to determine the physico-chemical properties of the feedstock and biodiesel. Kinematic viscosity (ASTM D-445), density (ASTM D-1298), flash point (ASTM D-93), cloud point (ASTMD-2500), acid value (ASTM D-664), pour point (ASTM D-97), sulfur content (ASTM D-4294) and calorific value (ASTM D-246) were determined. Other properties such as refractive index, specific gravity and iodine value were determined using appropriate AOAC methods (AOAC,2000).

3. Results and Discussion

3.1. Fatty Acid Profile

The fatty acid composition of the biodiesel obtained from the transesterification of soybean soapstock comprises mainly saturated fatty acid with oleic acid having the highest percentage composition of 26% and linolenic acid slightly lower at 22% as seen in Table 1 below. The characteristics of the biodiesel would tend towards the characteristics exhibited by oleic and linolenic compounds. [8,9] proposed a viable fatty acid composition mix of C16:1, C18:1 and C14:0 in the ratio 5:4:1 for biodiesel production. This signifies that the unsaturated fatty acids should ideally have a higher ratio to the saturated ones with one of the reasons being that oxidative stability is influenced by unsaturation (Ramos et al., 2009). This was observed in the soybean soapstock biodiesel as oleic acid (C18:1) with a % concentration of 26.045% had a similar ratio (3:1) with Myristic acid (C14:0) with a percentage concentration of 10.15% when compared with the ratio (4:1) suggested by [20].

It was however observed that the ratio of unsaturated to saturated fatty acids in the feedstock was different from the fatty acid profile of the biodiesel with the saturated fatty acid higher than the unsaturated fatty acid. The oleic acid to Myristic acid ratio of 1:2 however does not conform with that suggested by [20] (4:1) for good biodiesels. This further proves the ability of transesterification to produce biodiesels through the conversion of saturated compounds into unsaturated fatty acids. It was observed that the predominant unsaturated compound in soybean soapstock was linolenic acid while the predominant saturated compounds were stearic acid, myristic acid and palmitic acid as seen in Table 1. The characteristics of the predominant fatty acid in the feedstock are usually reflected in the fatty acid composition of the biodiesel and consequently in the biodiesel physiochemical properties. Unsaturated fatty acids (Oleic and linolenic) recorded the highest composition after transesterification (26.045% and 22.344% respectively). This change in fatty acid composition in soybean soapstock and its biodiesel further confirms that transesterification transforms compounds from saturation to unsaturation.

Table 1. Comparison of Soyabeansoapstock feedstock and biodiesel fatty acid compositions

Component	Name	Soybean soapstock biodiesel concentration(%)	Soybean soapstock concentration (%)
C12	Lauric acid	6.326	5.993
C16	Palmitic acid	17.409	14.898
C18	Stearic acid	7.839	15.542
C18:2	Linoleic acid	9.882	6.148
C18:1	Oleic acid	26.045	9.462
C18:3	Linolenic acid	22.344	32.261
C14	Myristic acid	10.15	15.696

3.2. FTIR Analysis

The presence of both groups (OH and OR) in the soybean soapstock and biodiesel as seen in Table 2 are complemented by the presence of cyclic ester compounds (RCOO) seen at 1839.703cm-1 for the oil and 1833.90cm -1for the biodiesel which are the parent compounds needed for esterification and transesterification. The conversion of the OH group to the OR group is further explained by the absence of the carboxylic acid group (RCOOH) in the biodiesel produced which is noticed at a peak of 2156.41cm-1 in Table 2. Biodiesel is also confirmed by the presence of an intense band of C=O (carbonyl group) stretching of methyl ester. This can be seen at peaks of 2160.35cm-1 and 2278.519cm-1 in the FTIR spectra of the biodiesel. The presence of more cyclic ester compounds and carbonyl groups is an indication of the conversion into biodiesel through esterification/transesterification reactions.

Table 2. Table of FTIR spectra of soya bean soap stock oil and biodiesel.

S/n	Wavenumber (cm^{-1})		Functional group	Compounds
	biodiesel	Feedstock		
1	755.56	758.72	C-CI	Chloro(Cl symmetric stretch)
2		896.23	C-CI	Chloro(Cl symmetric stretch)
3	1232.69	1091.59	R-O-R	Ether (C-O symmetric stretch)
4	1309.29	1391.49	$H_2C=CH_2$	Ethene (C=C symmetric stretch)

5		1405.13	$H_2C=CH_2$	Ethene (C=C symmetric stretch)
6	1627.61	1619.94	RNH_3	Primary amine (N-H stretch)
7	1833.90	1839.70	RCOO	Cyclic ester (C-O symmetric stretch)
8	1999.50	1949.92	R-S-C≡N	Thiocyanate (S-C-N antisymmetric)
9		2156.41	RCOOH	Carboxylic acid C-O stretch
10		2160.35	RC=O	Carbonyl (C-O stretching vibration)
11		2278.51	RC=O	Carbonyl (C-O stretching vibration)
12	2451.62	2642.52	R-C≡N	Nitriles (C-N antisymmetric stretch)
13	2657.43	2877.74	CH_2	Methylene (C-H stretch)
14		2867.88	CH_2	Methylene (C-H stretch)
15	3035.53	3064.81	RCHOH	Primary alcohol (O-H stretch)
16	3175.34	3177.10	RCHOH	Primary alcohol (O-H stretch)
17	3300.53	3318.33	R_2CHOH	Secondary alcohol (O-H stretch)
18	3417.74	3431.59	R_2N	Secondary amine (N-H stretch)
19	3680.02	3649.07	R_3CH0H	Tertiary alcohol (O-H stretch)

3.3. Physico-chemical Properties of Soybean soapstock Biodiesel

The physico-chemical properties of soybean soapstock biodiesel are presented in Table 3.The acid value of soybean soapstock biodiesel was high (5.5 mgKOH/g) when compared to the acid value of other biodiesels. This however is a feature of feedstocks of plant origin when compared to those of animal origin. The high FFA content in these cases could be attributed to the age, geographical location and storage condition of the seeds [4]. The soybean soapstock having a high acid value of 48.62 mgKOH/g, necessitated a two-step transesterification required to boost biodiesel yield from the oil. A multi-step transesterification or blending of the soybean methyl ester with other methyl esters might be required to further bring down the acid value closer to the required standard (0.8 mgKOH/g) as seen in Table 3 below. This will improve its suitability for application in diesel engines to avoid corrosion of the fuel system.

Viscosity is a crucial property in fuels for the determination of optimum handling, storage and operational conditions. The high viscosity in soybean soapstock methyl ester though not desirable, represents a feature of biodiesels from plant sources. It is however important to note that the viscosity of unsaturated fatty acid depends on the number and nature of double bonds but is less affected by position [17]. The viscosity in the biodiesel however fell within the standard limits as seen in Table 3. Blending with other fuels could however go a long way in solving the high viscosity and density problems associated with biodiesel. The calorific value of the biodiesel (30, 739 kJ/kg) which is usually a reflection of its feedstock (soybean soapstock) falls just short of the standard limit (35,000 kJ/kg) and could also be compensated by blending and proper feedstock refining. Cold flow properties (cloud point and pour point) are important low- temperature fuel parameters that prevent blockage of fuel lines and filters which could lead to engine breakdown due to poor lubrication. The pour point temperature becomes higher as chain length increases in saturated FAMEs and becomes lower for unsaturated FAMEs [8]. This was noticed as biodiesel from soybean soapstock having pour point 0oC just falls within the upper standard limit. [8] however stated that cloud point decreases with increasing double bonds which was highlighted with the low cloud point of soybean soapstock biodiesel realized (3 oC) when compared to the upper standard limit of 12oC owing to the high percentage of branched- chain double bonds present in the soybean soapstock biodiesel value which is usually a measure of the degree of unsaturation should be less than 130 I2/100g of oil to be suitable as biodiesels because oils with high degree of unsaturation when heated are prone to polymerization of the glycerides causing the formation of deposits

[9]. The iodine value of soybean soapstock biodiesel (63.2 mgI2/100g) was well within the standard limits as seen in Table 3.

Table 3. Fuel properties of soybean soapstock biodiesel compared with standards

Physico-chemical properties	Soybean soap stock biodiesel	Standard Limit		Test
		min	max	
Acid Value (mgKOH/g)	5.5	_	0.8	ASTM D664
Specific gravity	0.87	_	0.88	ASTM D6751
Ash content (%)	0.09	_	0.02	ISO 3987
Moisture content (%)	8.08			
Viscosity (Pa.s)	4.1	1.9	6	ASTM D445
Calorific value (kJ/Kg)	30,739.14	35,000		DIN51900
Refractive index	1.4573		1.4	B100
Cloud point (^oC)	3	-3	12	ASTM D6751
Pour point (^oC)	0	-15	0	ISO 3016
Flash point (^oC)	70	100	170	ASTM D93
Saponification value (mgKOH/g)	1.4	_	0.5	ASTM D664
Iodine value (mgI_2/100 g)	63.2	_	130	EN 14111
Peroxide value (Meq/kg)	2.8			
Smoke point (^oC)	62			
Titre (^oC)	-12			
Conductivity (µs/cm)	0		0.87	ASTM D6751
Sulphur (%)	0.03		0.05	ASTM D6751

The effect of saturation on the kinematic viscosity of soybean soapstock methyl ester (SSSME) was highlighted when compared with rapeseed oil methyl ester (RME), coconut oil methyl ester (CME), soybean oil methyl ester (SME) and palm kernel oil methyl ester, (PKME) as seen in Table 4. It was observed that higher saturation reduces kinematic viscosity with PKME and CME having high percentages of saturated fatty acids recording lower kinematic viscosities of 2.9 mm2/sec and 2.7 mm2/sec respectively while RME and SME with lower percentages of saturated fatty acids having higher viscosities of 4.5 mm2/sec and 6.63 mm2/sec respectively when compared to SSSME with kinematic viscosity of 4.1 mm2/sec. Saturation was also seen to favor cold flow properties with highly saturated methyl esters such as CME and PKME having lower pour points (-5oC) when compared to the pour point of SSSME (0oC). Other cold flow parameters such as cold filter plugging point, cloud point (CP) and low-temperature filterability test which also determines the cold flow behavior of diesel fuel are also affected by the compositional changes in fatty acids [14].Unsaturation on the other hand favors higher densities with RME and SME which had the highest percentage of unsaturated fatty acids recording the highest densities as seen in Table 4. The net calorific values of methyl ester fuels are about 15% lower than those of diesel fuel as seen in Table 4. Comparing the net calorific values in methyl ester fuels, soybean methyl ester had the highest calorific value due to the high calorie content of the feedstock (soybeans). CTME had a low calorific value of 34.2 MJ partly due to its animal origin whose calorie level is naturally lower compared to biodiesels of plant origin. Since less net calorific value makes more fuel consumption for combustion, SSSME thus proved to be the better fuel as regards calorific value. The calorific value of the methyl esters could however be brought to par with the diesel fuels through more efficient separation and purification of the feedstock. The densities of methyl ester fuels were generally higher than those of diesel fuel. The density of SSSME at 870 kg/m3 falls within the range of the ASTM D6751 standard limits as seen in Table 3.

Table 4. Comparison of Physico-chemical properties of methyl esters with fossil fuel and standards.

Physico-chemical properties	SSSME	CTME	CME	PME	PKME	RME	SME	Fossil diesel	Standard min	Standard max
Calorific value (MJ)	30.7	34.2	35.22	36.85	35.61	36.55	38.22	43.12	35	
Density (kg/m^3)	870	842	874	879	877	886	883	826	-	880
Kinematic viscosity (Pas)	4.1	1.52	2.7	4.5	2.9	4.5	4.1	2.5	1.9	6
Pour point (^{o}C)	0	-9	-5	12.5	-5	-7.5	0	-12.5	-15	0

3.4. The Use of n-hexane in Biodiesel Production

3.4.1. Effect of n-Hexane on Biodiesel Production

The transesterification of esterified soybean soapstock with methanol as the only solvent proved to be a problem as poor FAME yield was recorded without n-hexane (co-solvent). This however led to a need to use a co-solvent in the transesterification reaction. This led to an increased FAME yield of up to 96.8%. N-hexane enhanced miscibility thus improving FAME yield [19].

3.4.2. Effect of Use of n-Hexane on Man/Environment and Mitigation

N-Hexane, a colourless but flammable liquid is a mixture of hexanes and small amounts of other chemicals. They have adverse effects on humans when inhaled or through skin contact. They can cause irritation of eyes, skin, nose, throat and lungs, with extreme exposures leading to headache, nausea, vomiting and sometimes death. To forestall these hazards, preventive and control measures will need to be put in place which includes proper labeling of containers, provision of eye wash/emergency showers, wearing of appropriate personal protective equipment and self consciousness of high n-hexane concentrations. There is thus a need to store n-hexane in tightly closed containers and in cool, dry environments away from ignition sources to avoid contaminations and in extreme cases explosions [22].

3.5. Profitability Comparison with Biodiesel Produced from Alternative Feedstock (Cow Tallow)

Economic analysis of biodiesel produced from soybean soapstock and cow tallow were carried out to ascertain the profitability of using soybean soapstock as feedstock for biodiesel production. The feasibility studies involved scaling up of laboratory experiments, calculation of cash flows and determination of profitability/ breakeven points.

To achieve these objectives, several assumptions were made as seen in Table 5 below.

Table 5. Summary of assumptions for cow tallow and soybean soapstock biodiesel production.

Parameter	CTB	SBS
Production capacity (litres /year)	66,000	80,000
Product price (litres)	₦1000	₦1000 ($2.30)

Parameter	CTB	SBS
Plant life (years)	5	5
Base year	2022	2022

CTB: cow tallow biodiesel, SBS: Soybean soapstock biodiesel

The biodiesel price was obtained from the international price of biodiesel at an average cost of $ 2.40/litre (MDA 2022). At the official exchange rate of N435 to 1 US dollar (as at September, 2022), the approximate cost comes down to N1000 ($2.30)/litre. A higher conversion ratio was obtained when using soybean soapstock due to the high conversion ensuring higher output (capacity) as seen in Table 5 above.

Profitability indicators such as Net present value (NPV), annual gross profit (GP), annual net profit (NP), payback period, return on investment (ROI) and interest rate of return (IRR) were used to determine and compare profitability using both feedstocks.

3.5.1. Net Present Value (NPV), Profit and Return on Investment (ROI).

The equations for the calculation of net present value (NPV), gross profit (GP), payback period (PP), return on investment (ROI) and internal rate of return (IRR) are shown in Equations 1 – 5 presented as follows:

$$NPV = \text{sum}\ (Ct / (1+r)^{t}) - Co \quad (1)$$

Annual gross profit (GP) = Total annual sales - Total annual costs (2)

Payback period (PP) = Total capital investment/Annual average net cashflow (3)

Return on investment (ROI) = Annual profit / Total investment costs * 100. (4)

Solving for internal rate of return is an iterative process and was estimated using equation 5 below:

$$N0 = \text{sum}\ NCF/ (1+IRR)*t \quad (5)$$

Where Ct is the net cash inflow during the period t, Co is the total initial investment costs,

r is the discount rate and t is the number of time periods.

NCF is the net cash flow, and IRR is internal rate of return

Economic analysis at 10% depreciation gave NPV as ₦87,460,690 and ₦109,482,304 as seen in Table 6, which shows the current value of the CT and SBS plants respectively. A pay-back time of 10 months and 9 months for cow tallow biodiesel and soybean soapstock biodiesel respectively shows a faster pay-back period for SBS. The faster payback period from the soybean soapstock feedstock was as a result of the higher conversion rates leading to higher profits and return on investment (ROI) and internal rate of return (IRR). ROI shows total investment growth over a number of years while IRR shows the annual growth rate. These are also in agreement with other profitability indicies such as the net present value (NPV) and pay-back period as elaborated in table 6 below.

Table 6. Profitability indicators for cow tallow and soybean soapstock biodiesel.

Probability Indicator	Cow tallow biodiesel	Soybean soapstock biodiesel

Net present value,NPV (₦)	87,460,690	109,482,384
interest rate of return, IRR (%)	80	88
Gross profit, GP (₦)	45,800,000	66,080,000
Net profit, NP (₦)	36,640,000	52,864,000
Payback period (years)	0.84	0.75
Return on investment, ROI (%)	30.28	38.47

3.6. Review of Related Works

Guo, F.,et al.(2012) produced biodiesel from acidified soybean soapstocks with the aid of a solid catalyst. An 80% yield was however a far cry from the yield obtained from earlier works. [11] studied the feasibility of biodiesel production from soybean soapstock using an ion exchange resin catalyst. A FAME yield of 98.1% realized in this work was an upgrade on the 91.7 % yield obtained without the use of the catalyst. However, the availability and cost of Amberlyst-15 (the resin catalyst) posed a problem in this process. In earlier work, [23] had earlier produced biodiesel from soybean soapstock using sulfuric acid as a catalyst with a biodiesel yield of 97.6% obtained. The high energy demand needed for this process due to the high temperature and pressure requirements however posed a limitation to the commercialization of this process. These cost and feasibility challenges encountered in these alternative routes however make the use of n-hexane in transesterification of soybean soapstock an attractive prospect for biodiesel production.

4. Conclusion and Recommendation

Findings from this work show that soybean soapstock could be used to produce biodiesel using both methanol and n-hexane as solvents. The biodiesel produced was economically viable and compared favorably with methyl esters from other feedstocks and fossil fuels. Using n-hexane as an additional solvent both improved the yield (96.8 wt%) and at a cheaper cost when compared to conventional biodiesel production methods. The fuel properties of the biodiesel produced were however significantly enhanced by transesterification. Though this production route involved an additional solvent (n-hexane), the ability to achieve higher conversion and by extension better economic value justified this process. It is thus recommended that soybean soapstock like other vegetable oil effluents be investigated further to be used as feedstocks for biodiesel production.

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The authors declare no conflicts of interest

Data Availability

Data supporting reported results can be found in the links to publicly archived datasets analyzed.

Conflicts of Interest

The authors declare no conflicts of interest.

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