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Synthesis and Characterization of Soybean Oil Based Biodiesel under Optimal Sonication Power

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Abstract

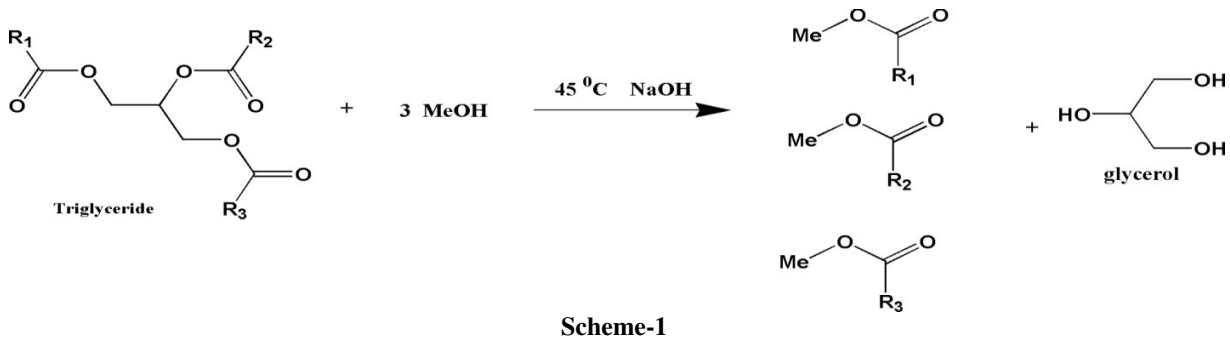
Biodiesel is gaining more and more importance due to depletion and uncontrollable rates of fuel resources. Biodiesel is a renewable, biodegradable and nontoxic fuel. The use of vegetable oils and their derivatives as alternative for diesel fuel is the best solution in the current scenario. Nowadays economical processes to save the energy and to reduce experimental cost plays very important role in biodiesel production. The present study employed simultaneous application of sonic and mechanical energy to assist transesterification of soybean oil. The objective was to optimize the sonication power to achieve the highest possible yield of methyl ester of soybean oil. Experimental results showed that the optimized reaction conditions involved oil to methanol molar ratio 1:6, sonication power 210 W, catalyst concentration 0.2 wt% of oil and mechanical agitation speed 200-300 rpm at 45 ^{0}C to yield the soybean oil methyl ester (SOME) up to ~95% yield in 60 minutes. Further, the synthesized SOME was characterized for density, kinematic viscosity, acid number, cetane number, calorific value, flash point, distillation temperature, sulfated ash content, carbon residue and fatty acid composition according to ASTM standards to ensure the quality of SOME to act as biodiesel.

Keywords: Base catalyst, transesterification, soybean oil, sonication power, biodiesel.

Introduction

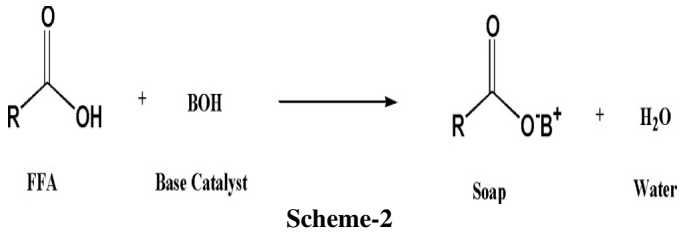
Diesel fuel, a petroleum based product, is largely utilized in number of sectors for mechanical power generation, some of them being transport, agriculture, commercial, domestic and industrial sectors. Though being efficiently used, the exponentially rising prices and high depletion rate of petroleum based products have made the human beings to search for economic fuels. In this regards, Rudolf Diesel, the founder of the diesel engine at first used the peanut oil as a fuel in diesel engine for demonstration purpose and successfully ended up with ecological and economical biofuel (biodiesel)[1]. Since then number of researchers have reported the use of other vegetable oils as an alternative to conventional diesel fuel[2,3]. However, their direct use in diesel engine causes many problems such as coking formation on the injectors causing improper fuel atomization, plugging of orifices, carbon deposits, thickening or gelling of the lubricating oil etc. The lubricating problems are due to higher viscosity of oils than that of biodiesel. To solve these problems, the oils require further modifications like transesterification, pyrolysis (thermal cracking) or its blending with petroleum derivatives to reduce the viscosity. Among all these processes, transesterification is the most favored one to produce clean and ecologically safe fuel from different types of oils[4].

The overall transesterification reaction of oil is a sequence of three consecutive and reversible reactions in presence of strong acid, base or enzyme catalyst [5, 6]. The transesterification reaction of oil requires 1 mole of oil and 3 moles of the alcohol as shown in scheme 1. However, an excess of the alcohol is used to increase the yields of the alkyl esters and to allow its phase separation from the glycerol formed.



Scheme-1
Transesterification of Oil

Number of parameters such as the type of catalyst, alcohol to oil molar ratio, temperature, purity of the reactants and free fatty acid content (FFA) affects the rate of transesterification reaction[7]. Among the above parameters, the selection of the catalyst mainly depends on FFA's content present in the oil. The base catalyzed transesterification reaction gives better conversion at low temperature in case of oils with lower amounts of FFA's as with high FFA contents, usually soap formation takes place as shown in scheme 2. However, for process economy the base catalysts, such as alkaline metal alkoxides and hydroxides are more preferred[2, 8-9].



Scheme-2
Soap formation

Many processing improvements have been made to increase conversion and yield of oil to biodiesel, to lower the production cost and improve quality of biodiesel. In conventional biodiesel production, dispersion of all reactants occurs primarily near the interface, leading to less interfacial area for the transesterification reaction reducing the biodiesel conversion as compared to that in ultrasonication. Ultrasound is the process of propagation of the compression (rarefaction) waves with frequencies above the range of human hearing [10]. When sound passes through a liquid, it consists of expansion waves and compression waves. These compression and expansion of waves cause bubbles to grow and recompress. Under proper conditions, acoustic cavitation can lead to result implosive compressions in such cavities. These implosive bubbles collapse and produce intense local heating with high pressure in very short period in the reaction mixture, which is useful to form homogeneous mixture of oil and alcohol [11]. Ultrasonication provides the mechanical energy for homogeneous mixing and the required activation energy for initiating the transesterification reactions. A low frequency ultrasonic irradiation is a useful tool for emulsification of immiscible liquids[12]. The effect of ultrasonic energy on vegetable oil biodiesel synthetic processes was studied by Seung Bum Lee et al, 2011. The ultrasound irradiation reduced reaction time and improved biodiesel properties as compared to conventional process[13,14].

A substantial literature is available for the production of biodiesel and for some other applications using ultrasound [10-25]. However, till date very few reports are available on application of simultaneous ultrasonication and mechanical mixing with minimum base catalyst concentration for production of biodiesel from vegetable oils[26,27].

In present study, soybean oil is used as vegetable oil source for biodiesel production due to is high soybean content around 20%. The simultaneous irradiation of ultrasonic energy and mechanical agitation was used for base catalyzed transesterification of soybean oil to shorten the reaction time and to increase the product efficiency. The optimal reaction conditions like sonication power, catalyst concentration, and methanol/oil molar ratio were also investigated.

Material and Methods

Equipment and Materials: The ultrasonic reactor (USB-150) with capacity 150 liter, frequency 40 kHz, total power 350 watts was procured from SPECTRA LAB and was characterized for power and frequency by the SPECTRA LAB Instruments Pvt. Ltd., Mumbai, India. Methanol (99.9%), sodium hydroxide (NaOH) and anhydrous sodium (Na_2SO_4) were purchased from sd Fine Chemicals Ltd., Mumbai, India. The soybean oil was procured from Kansai Nerolac Paints, Mumbai, India.

Analytical methods: The acid value (AV, mg KOH /g) and saponification value (SV, mg KOH /g) were determined by AOCS standard titrimetry method. The molecular weight (MW) of soybean oil was calculated from its saponification value (SV) and acid value (AV). The physical and chemical properties of soybean oil used are as shown in table 1.

Table-1
Characterization of soybean oil

Density at 31^0C	929 Kg/m^3
Molecular weight	885.58
Kinematic viscosity 31^0C	29 mm^2/sec
Saponification value	190 mg KOH/g oil
Iodine value	123 g of ICl/ 100 g oil
Acid value	0.2 mg KOH/g oil
Free fatty acids	< 0.1%
Cetane number	37
Calorific value	37.63 MJ/Kg
Flash point	253 ^{0}C

Experimental: Transesterification of soybean oil using conventional method: In a conventional reaction methyl ester was synthesized using NaOH as a catalyst under identical parameters. The molar ratios of soybean oil to methanol were 1:3, 1:4, 1:5, 1:6 and 1:8. The catalyst concentration 0.2 wt% of oil and mechanical agitation speed 200-300 rpm was maintained throughout the set of experiments. Alcoholic solutions of NaOH was added to 65 g of soybean oil and the reaction mixture was allowed to react in 125 ml three neck round bottom flask for 60 minutes at 45^0C. After completion of the reaction, the resulting mixture of methyl ester and glycerol was separated in separating

funnel. After purification, it was observed that amongst the series, 1:6 molar ratio gave better yield 82.33 %.

Transesterification of soybean oil using ultrasonic reactor: Ultrasound assisted transesterification reactions of soybean oil were carried out using an ultrasonic reactor in which a three neck round bottom flask of 125 ml capacity coupled with a water reflux condenser mounted on it was installed. The ultrasonic reactor was used to generate ultrasound, which when transmitted into a liquid sample caused mixing and provided necessary energy for the transesterification of soybean oil. Transesterification reactions of soybean oil using sodium hydroxide catalyst were performed for 60 minutes at 45^0C in ultrasonic bath at 35-350 W sonication power with simultaneous 200-300 rpm mechanical agitation by keeping other reaction conditions and process parameters was unaltered.

Separation of soybean oil methyl ester (SOME) from byproduct: After completion of 60 minutes, the product was allowed to settle down in a separating funnel for 30 min into two liquid phases, the upper one of SOME and the lower of glycerol. The upper layer of SOME was purified by distilling the unreacted methanol at its boiling point. After distillation; the SOME was washed with hot water and 20% aqueous acetic acid to remove NaOH from the SOME. Repeated water washings were provided till neutral pH of SOME and then filtered by using anhydrous sodium sulfate (Na_2SO_4) to remove the traces of water. Finally the purified SOME was characterized by various analytical methods to identify its physical and chemical characteristics of fuel for its suitability to act as a biodiesel.

Ester characterization: Fatty acid composition of SOME was determined by using Varian 8400 GC equipped with a flame ionization detector and Restek RTX wax column (30 m x 0.25 mm Ld., 0.20 J.lm film thicknesses). Helium gas was purged as at 1 ml/min. The oven temperature was initially held at 150°C for 15 min, and then increased to 210°C at 2°C/min, followed by an increase to 220°C at 50°C/min. The injector and detector temperatures were set at 240 and 270°C, respectively. The SOME was prepared as per the standard method reported by Metcalfe LD, Schmitz [28].

The ester obtained by transesterification of soybean oil was then tested for estimating its fuel properties according to the ASTM standard test method of analysis [29].

Results and Discussion

Base catalyzed transesterification of soybean oil was carried out for process economy and to achieve maximum possible yield of SOME in short time and hence the factors affecting the yield of methyl ester were studied.

Factors affecting the yield of soybean oil methyl ester: Effect of molar ratio: It is one of the most important parameter affecting the yield of methyl ester. The transesterification reaction was observed to be incomplete for 1:3 to 1:5 molar ratios of oil: methanol and no more separation were observed in separating funnel at the time of settling. As the molar ratio of oil to methanol increased from 1:6 to 1:8 there was an increase in the yield of SOME. This can be easily explained from the fact that with the increasing the alcohol content, the reversible reaction of transesterification resulted in increasing the yield of SOME. Amongst the series, 1:6 molar ratio resulting in SOME with 82.33% yield and 6.46 mm^2/sec kinematic viscosity was optimized. The results obtained via conventional process are tabulated in Table 2. Higher amount of methanol interfere with the separation of glycerol because of an increase in solubility, the glycerol remaining in the solution helps to move the equilibrium back to the left side of the reaction resulting in the lower yield of methyl ester. The maximum yield of oil to ester was at the molar ratio 1:6 as reported by some researchers [30, 31].

Effect of sonication power: It was necessary to optimize the amount of energy supplied to the reaction mixture to maximize the yields of methyl ester at the lowest possible energy input for cost effective production of SOME. Hence, the effect of ultrasonic power on the biodiesel formation was studied for different powers input. When the intensity (i.e., ultrasonic power/irradiation area) is increased, the acoustic amplitude increases and a more violent collapse of the cavitation bubble occur. The harsher the collapse of the cavitation bubble, the higher is the jet velocity and micromixing at the phase boundary between the soybean oil and methanol phases. This results in finer emulsion formation, hence a higher mass-transfer coefficient and thus higher the methyl ester formation. Singh et al, 2007 have found similar results in the formation of biodiesel from soybean oil [32].

In transesterification reaction of soybean oil NaOH was used 0.2 wt% of oil as a catalyst. The temperature of the each reaction was not controlled in this set of reactions because the increase in temperature caused by ultrasound helps to increase the rate of methyl ester formation. The temperature of the reaction was 28 ± 1°C at the start of the reaction, but it increased to a maximum of 45°C. The results obtained as shown in table 2, indicate that 94.42% yield for SOME was obtained with sonication power 210 W keeping all other reaction parameters constant. This was observed to be the highest amongst all sonication powers. It was also observed that with increase in input energy of ultrasonic reactor the yield of SOME increased initially, reached the maximum and started declining after sonication power input 210 W. This could be attributed to the fact that higher energy input cause cracking and degradation [32].

Table-2
Effect of molar ratio on yield and properties of soybean oil methyl ester

Conventional Batches without sonication	Power (watt)	Yield of SOME %	Sap value mg KOH/g	Density Kg/m^3	Kinematic viscosity mm^2/sec
Molar ratio					
1:3	-	71.24	188.94	895	7.54
1:4	-	74.81	185.64	890	7.22
1:5	-	77.67	183.21	886	6.64
1:6	-	82.33	175.18	880	6.46
1:8	-	83.10	171.05	878	6.48
Batches on Sonicator					
1:6	35	82.05	182.54	880	5.81
1:6	70	87.67	181.21	878	5.79
1:6	105	91.29	180.72	880	5.31
1:6	140	93.98	180.74	877	4.75
1:6	175	93.84	178.88	874	4.38
1:6	210	94.92	175.35	872	4.13
1:6	245	93.90	178.14	876	5.52
1:6	280	93.11	179.90	875	5.59
1:6	315	92.31	179.90	879	5.87
1:6	346.5	89.03	181.63	881	6.06
1:6	350	88.22	183.40	883	6.24

Effect of catalyst concentrations on yield at optimized sonication power: The effect of NaOH concentration on yield of SOME was studied separately in the range of 0.2 to 1 wt% of oil at optimized sonication power 210 W at 45 ^{0}C for 60 minutes by keeping all other reaction parameters constant. It was observed that sonication helps to increase the yield of SOME and decrease the amount of catalyst. Further, increase in catalyst concentration does not increase the yield but infact adds to cost as it is necessary to remove it from the reaction medium at the end of reaction. The results show that the yield of SOME decreased with increase in the concentration of catalyst as shown in figure 1. Generally the catalyst NaOH reacts with methanol to produce the reactive nucleophile methoxide anions, which attack the tri-, di-, and monoglycerides in presence of methanol. Once a sufficient amount of methoxide anions is formed, the transfer of these anions to the reactant (Triglyceride, Diglyceride, and Monoglyceride) phase becomes more important. The rate of transfer of these radicals depends upon the interfacial area, which in turn depends upon the amount of methanol and oil. As the intensity of cavitation bubble collapse and the number of cavitation events depend upon the properties of the reaction medium, the oil/methanol ratio becomes more important. Santos et al. have observed that there is a relation between the oil/methanol ratio and amount of catalyst. At a lower molar ratio of oil/methanol, the amount of catalyst plays an important role, whereas at a higher molar ratio, it has little statistical significance[33]. However, using higher concentration of catalyst more than 0.2 wt%, the yield of SOME decreased and resulted in no clear separation in separating funnel during settling, while washing with warm distilled water more soap formation was observed due to excess of catalyst favoring the process of saponification.

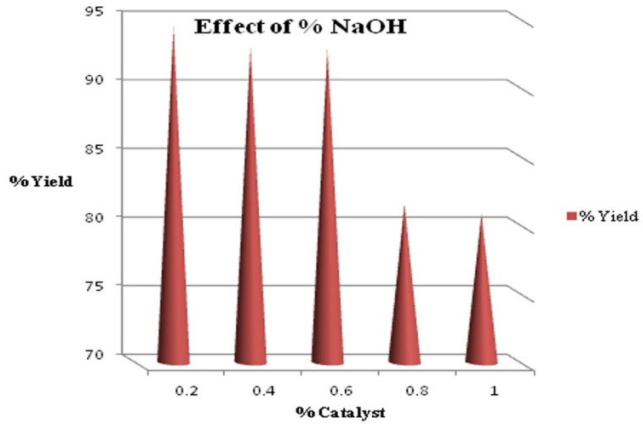


Figure-3
Effect of % NaOH on yield of soybean oil methyl ester

Physical properties of SOME at optimized sonication power

The properties of the SOME were evaluated using standard method of analysis[29]; all results are tabulated in table 3. The first most important property of SOME to act as biodiesel is calorific value 39.76 MJ/Kg within the range of biodiesel according to ASTM D 2015. The higher calorific value of biodiesel indicates lower power consumption by the engine, thus increasing the efficiency of it.

Cetane number of biodiesel indicates the actual quality of compression ignition of diesel fuel. The cetane number of SOME is ~47 within the range of biodiesel according to ASTM D 613. Usually, Cetane number mainly depends on the distribution of fatty acids in the oils from which it was obtained and increases with increase in saturation[34,35]. It influences the performance parameters like combustion stability, drive ability, white smoke, noise ability, emission of carbon monoxide and hydrocarbons when injected in engines.

The SOME synthesized was free from sulfur, thus eliminating the engine corrosion problems occurring due to the combustion of sulfur generating corroding sulfur dioxide, which a conventional sulfur containing fuel usually causes. The other advantage of SOME is quite high flash point 162 ^{0}C can prevent auto ignition and fire hazards at higher temperature during transportation and storage periods. The kinematic viscosity of SOME was investigated to be 4.13mm^2/sec within the range of biodiesel according to ASTM D 445. Higher fuel viscosity affects the flow and atomization characteristics of fuel and affects engine start ability[39].

The acid number of fuel used for detection of free fatty acids (FFA) content. The acid number of SOME was meeting to the standard limit of biodiesel, indicates that FFA will not cause corrosion; pump plugging due to corrosion and its deposition. The copper strip corrosion test was also meeting to standard fuel specifications according to ASTM D 130. Table 3 and table 4 show fatty acid compositions and characteristic properties of obtained SOME at optimized sonication power respectively.

Table-3
Fatty acid composition of soybean oil methyl ester at optimized sonication power

Fatty acid	Composition (wt %)
1. Palmitic ($C_{16:0}$)	11.39
2. Stearic ($C_{18:0}$)	03.92
3. Oleic ($C_{18:1}$)	26.16
4. Linoleic ($C_{18:2}$)	51.78
5. Linolenic ($C_{18:3}$)	06.75

Table-4
Physical and chemical properties of soybean oil methyl ester

Fuel properties	Standards method	Limits of Biodiesel	Soybean oil methyl ester	Standard Diesel fuel
Density at 31 ^{0}C (Kg/m$^{3)}$	BIS	860-900	872	867
Total acid number (TAN),mg KOH/g	D 664	≤0.8	0.1	0.12
Saponification value, mg KOH/g	AOCS	-	175.35	-
Flash point,^{0}C	D 93	≥130	162	55
Kinematic viscosity at 40^0 C,mm^2/sec	D 445	1.9-6	4.13	3.42
Copper strip corrosion,3 Hr,50 ^{0}C	D 130	No. 3	No. 1 b	No. 3
Cetane number	D 613	≥47	46.93	50
Calorific value MJ/Kg	D 2015	39-43.33	39.76	44.5
Sulfated Ash content,wt%	D 5453	≤0.02	Nil	0.89
Ash content,wt %	D 975	0.01	Nil	0.0056
Carbon residue, wt%	D 4530	0.050 max	Nil	0.080

Conclusion

We presented novel method with minimum concentration of base catalyst for transesterification of soybean oil through simultaneous sonication and mechanical agitation for producing better quality soybean oil methyl ester (SOME) to act as a biodiesel. Experimental results indicated that sonication power 210 W of ultrasonic reactor was effective in biodiesel production as against the conventional batches carried out under identical molar ratio. The optimized reaction conditions were soybean oil to methanol molar ratio 1:6, ,catalyst concentration 0.2 wt % of oil, mechanical agitation speed 200-300 rpm and reaction temperature 45 ^{0}C yielding ~ 95 % SOME in 60 minutes. Further, the sonication power [38], total reaction time, temperature, mechanical agitation speed [15,36], catalyst concentration [21, 37, 38] was lower than that reported studies. This method is time saving and cost effective, yielding SOME with potential to be used as a biodiesel conforming to its all fuel properties such as cetane number, calorific value, flash point as well as its sulfate content, ash content, carbon residue value within the range of biodiesel.

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