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A Comparative Analysis of Impact of Hexane, Diethyl Ether, Toluene and Acetone on Biodiesel Transesterification Process

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The aim of this study was to examine the production of biodiesel from waste cooking oil with an acid value of 1.82 mg KOH/g in the presence of methanol and KOH, utilizing cosolvent technology in order to increase biodiesel output by overcoming mass transfer resistance. This study examined the effect of four cosolvents (hexane, diethyl ether, toluene and acetone) on biodiesel yield under optimized reaction conditions, including cosolvent-to-oil weight ratio, reaction time and temperature. The polarity index of cosolvent was determined primarily on its biodiesel yield performance. Highest yield (98.46%) of biodiesel was obtained at 20 wt.% of acetone; 1:6 molar ratio (oil-to-methanol), 1 wt.% KOH at 40 ± 1 ºC for 10 min of reaction time at 600 rpm. The physico-chemical properties of biodiesel such as acid value, density, kinematic viscosity and flash point were evaluated and found to be within ASTM standards.

Keywords: Acetone, Biodiesel, Cosolvents, Transesterification, Waste cooking oil.

INTRODUCTION

After industrialization, it has been emitting more than 1200 gigatonnes of CO_2 into the earth's atmosphere due to human activities worldwide [1]. Globally, most air pollution is generated by fossil fuel combustion for electricity generation and transportation [2]. The same fact has led to the primary source of Greenhouse gas emissions (GHG) for decades. The way that environmental pollution causes respiratory problems and other health issues have been addressed by several researchers [3,4]. Energy services are critical for economic growth and living standards improvement, so an increase in demand is both necessary and desirable. Only through the use of renewable energy can be achieved sustainable development. As a result, a number of developed countries are concentrating their efforts on enhancing or developing long-term renewable energy sources [5]. GHG emission can be reduced by utilizing renewable energy sources. Biofuels and biodiesel, as well as renewable energy sources such as wind, solar, geothermal, hydro and biomass, all contribute significantly to greenhouse gas reduction [6].

It is clear that fossil fuels are non-renewable energy sources as they take millions of years to form and replenish; they are being consumed faster than they are being reproduced. Therefore, renewable energy is vital to replace fossil fuel combustion for power generation. Biodiesel is a widely used renewable energy source to minimize fossil fuel combustion and reduce environmental pollution. There are mainly three advantages of biodiesel. It will reduce the foreign dependency on fossil fuel, produce domestically and have negligible greenhouse emissions compared to the fossil fuel combustion process [7]. Biodiesel is an essential renewable fuel source that can be utilized in standard diesel vehicles without engine modifications, making it a viable alternative to fossil diesel [8].

Biodiesel is produced from edible oils such as coconut oil, palm oil, sunflower oil and so on, as well as non-edible oil sources such as rubber seed oil, Jatropha oil, waste cooking oil (WCO) or animal fat and so on [9]. Biodiesel has significant benefits and disadvantages as a fuel source [10]. Biodiesel has chemical and thermal properties that are comparable to those of fossil diesel fuel. Biodiesel has a higher cetane number than fossil diesel fuel, making it more appropriate for combustion in a diesel engine. Biodiesel is an excellent lubricant and can compensate for the lubrication loss associated with the removal of sulphur from a blend with low sulphur fossil diesel fuel [11]. The main concern is that the production cost is higher than fossil diesel. Therefore, production costs should be reduced or biodiesel yield should be increased to overcome that issue. Production cost can be minimized only by reducing the raw material cost, representing 70-90% of the total cost of production [10]. Therefore, enhancing the yield of biodiesel is the best way to reduce the cost of production.

The widespread use and efficient chemical reaction for biodiesel production are considered transesterification or in other words as alcoholysis. The transesterification reaction can occur as an acid-catalyzed or base-catalyzed reaction, non-catalytic or enzymatic catalysis reaction [12,13].

However, the choice of catalysis depends on the free fatty acid (FFA%) amount and the water contents in the fatty materials [14,15]. During the transesterification reaction, the reactants that participate in the reaction do not form a single-phase reaction medium. Therefore, the transesterification reaction is biphasic due to the immiscibility of oil and methanol in a transesterification system. Accordingly, mass transfer between the two phases becomes a key factor affecting the reaction rate [16]. As a result, it is necessary to maintain a high process temperature and pressure throughout the reaction in order to overcome the mass transfer resistance between alcohol and oil [17]. Although increasing the temperature can improve the miscibility of the two phases, this is an energy-intensive operation [18]. One option to reduce the process's severity is incorporating cosolvents or entrainers [17].

Recently, attention has been focused on ways to improve the transesterification process using solvent technologies. Methanol is an efficient solvent for transesterifying vegetable oils to their fatty acid methyl ester (FAME). However, the reaction occurs when the parent chain carbon atom and the alkyl substituent carbon atom in the triglyceride undergo complete bond cleavage. This is the rate-determining phase of the reaction. When another solvent (cosolvent system) is added to methanol, significant improvements in the mechanism of this nucleophilic substitution have been found [19]. A one-phase reaction can be accomplished by adding a cosolvent, a solvent that increases the solubility of oil [20]. Alcohol, fatty acids and triglycerides are soluble in the cosolvent. Water should not be present in the cosolvent and the more cosolvent added, the more solubility of the oil will be improved. The chosen cosolvent has a boiling point similar to methanol, which simplifies the reaction's termination method [21].

However, the comparison of different cosolvents impact transesterification reaction has to be addressed to compare those and rank the effectiveness of the cosolvents in biodiesel production. Therefore, the present study focuses on ranking cosolvents for transesterification reaction based on the biodiesel yield and efficiency.

EXPERIMENTAL

Pre-treatment: To remove any solid contaminants, the waste cooking oil (WCO) was filtered with a mesh size of 250 µm. Water was removed by heating the WCO to 110 ºC for 10 min to ensure complete evaporation of all water molecules. Following that the WCO was cooled to room temperature.

Free fatty acid (FFA) content and acid value: The FFA content was determined using the ASTM D5555-95 (2007) method. To begin, 1 g of dried WCO was transferred to the titration flask at room temperature (25 ± 1 ºC), followed by the addition of 125 mL isopropyl alcohol. Two drops of phenolphthalein were added to the flask as an indicator and 0.1N KOH was used as the titrant. The titration was conducted until the colourless solution became pale pink, at which endpoint the volume spent in mL was noted. In absence of WCO sample, a blank sample was taken and repeated the same procedure. The volume difference between the blank and sample was used in eqns. 1 and 2 to get the FFA% and acid value, respectively.

$$\text{FFA}\ (\%) = \frac{V \times N \times 28.05}{W} \tag{1}$$

$$\text{Acid value (mg KOH/g)} = \text{FFA}\ (\%) \times 1.99 \tag{2}$$

where, V is the volume of KOH in mL required to neutralize the titrand; N is the normality of KOH solution; and W is the weight of WCO sample.

Transesterification: Transesterification was carried out on a number of co-solvents. Table-1 lists the features of each solvent. A 100 mL of preheated WCO (acid value of 1.82 mg KOH/g) was transferred to a closed reaction bottle equipped with a magnetic stirrer. At room temperature, the mixture of WCO and cosolvent was mixed at 200 rpm for 5 min. After that the solution in the container was heated to the desired reaction temperature to initiate the transesterification. When the solution mixture reached the desired reaction temperature, the catalyst reagent (1 wt.% KOH based oil WCO weight) was added to the bottle as a methoxide form at a molar ratio methanol-to-WCO of 6:1. The transesterification was carried out at desirable reaction temperature with simultaneous stirring at 600 rpm using a hot plate with a magnetic stirrer for desirable reaction time. After the reaction was completed, the products

TABLE-1
PHYSICAL PROPERTIES OF SOLVENT AND REACTANTS USED IN THIS STUDY [Ref. 22-24]

Property	Methanol	Hexane	Diethyl ether	Toluene	Acetone
Physical form	Colourless liquid	Colourless liquid	Colourless liquid	Colourless liquid	Colourless liquid
m.w. (g/mol)	32.04	86.17	74.12	92.13	58.08
Flashpoint (°C)	11	-9	-45	4	-17
Boiling point (°C)	64.7	69	34.6	110.8	56.5
Density at 25 °C (g/cm^3)	0.791	0.659	0.706	0.866	0.791
Viscosity at 20 °C (cP)	0.55	0.297	0.224	0.560	0.32
m.p. (°C)	-98	-94	-116	-95	-94
Electric dipole moment (C·m)	2.87	0.08	1.15	0.31	2.69
Polarity index	5.1	0.1	2.8	2.4	5.1

were moved to a separation funnel and allowed to settle overnight to form two layers. The lower glycerol layer was removed and discarded, while the higher methyl esters layer was collected. The mixture was then purified by successively washing it with warm distilled water (50 ± 1 ºC) to remove the remaining impurities such as catalyst, soaps and glycerol for 20 min at 200 rpm until a pH 7 was obtained. The products were then transferred to a separation funnel and left overnight to form two layers. The water layer was removed and discarded, while the biodiesel layer was collected and dried at 110 ± 1 ºC for 10 min. The washing and drying process was continued until the dried biodiesel reached a neutral pH value. The yield of biodiesel produced was determined using eqn. 3:

$$\text{Biodiesel yield (\%)} = \frac{\text{Biodiesel dry weight}}{\text{WCO dry weight}} \times 100 \qquad (3)$$

Process optimization: To determine the optimal reaction conditions for increased biodiesel yield, the effect of cosolvent-to-oil weight ratio (10, 20, 30, 40 and 50 wt.%), reaction time (5, 10, 15, 20, 25 and 30 min), reaction temperature (30, 35, 40, 45, 50, 60 ºC) and reaction speed (200, 300, 400, 500, 600, 700 rpm) was investigated. On the basis of the results of process optimization for each cosolvent, the optimal biodiesel yield was calculated.

Biodiesel characterization: At the laboratory, the physical characteristics of the biodiesel such as, flash point, density, kinematic viscosity and chemical characteristic of acid value were evaluated followed by the American Society for Testing and Materials (ASTM) analytical technique, identical conditions were maintained for all samples.

RESULTS AND DISCUSSION

Effect of cosolvent-to-oil weight ratio: The effect of the cosolvent-to-oil weight ratio on the cosolvent methanolysis of WCO was explored by testing various cosolvents such as hexane, toluene, diethyl ether (DEE) and acetone with six different cosolvent-to-oil weight ratios (10, 20, 30, 40 and 50 wt.%) as seen in Fig. 1. Other variables were kept constant at 1 wt.% KOH, 6:1 molar ratio of methanol-to-oil, reaction temperature of 60 ºC and 30 min of reaction and a stirring speed of 600 rpm.

Fig. 1 demonstrates an increased in biodiesel production from 77.6% to more than 80% as the weight ratio of cosolvent-to-oil was increased from 0 to 10%. The transesterification reaction is biphasic due to the immiscibility of oil and methanol in a transesterification system. Accordingly, mass transfer between the two phases becomes a key factor affecting the reaction rate [25]. Without cosolvent (cosolvent-to-oil weight ratio of 0%), biodiesel production may be constrained by mass transfer resistance, which was solved by adding a tiny amount of cosolvent to the reaction system. Initially, methanol was less soluble in oil due to a lower cosolvent concentration, resulting in a lesser biodiesel yield. However, it was observed that adding acetone at a concentration of 20 wt.% significantly increased the yield, resulting in an optimum yield of 96.39%. In contrast, other cosolvents such as hexane, toluene and DEE required significantly more quantity to achieve the optimum yields of 90.02%, 92.66% and 93.50%, respectively. Compared to hexane (40 wt.%), toluene and DEE, their optimum yields were achieved at a lower ratio (30 wt.%). This could be attributed to the fact that the polarity index and electric dipole moment coincide with the polarity of chemical substances, as shown in Table-2. The dipole moment of toluene (0.31 C·m) and DEE (1.15 C·m) are comparable to those of methanol (2.87 C·m), showing that these solvents were reasonably miscible and required just a tiny amount of cosolvent to establish a single phase. The dipole moment of hexane (0.08 C·m) are significantly different from methanol, which has a smaller single area, signaling that a large amount of cosolvent must be added to the reaction mixture to achieve homogeneity. Hexane is soluble in WCO but has limited solubility in methanol; as a result, it functions as an antisolvent for WCO in methanol, reducing the yield of biodiesel. However, at high concentrations, hexane mixing of methanol and WCO was improved dramatically and significantly reduced the oil viscosity. Reduced viscosity results in increased mass transfer between methanol and oil, hence boosting product yield [26]. Excess cosolvents in the reaction medium showed no discernible influence on biodiesel yield, while yield decreased with the weight of the cosolvents due to the dilution effect. The mixture's viscosity decreased as the cosolvent concentration increased, indicating that the density difference between the FAME and glycerol lowered the time necessary for phase separation. This behaviour is explained by the concentration-dependent separation of glycerol and excess methanol in the reaction mixture. The concentrations of glycerol and methanol are more significant with a lower cosolvent level than with higher cosolvent content. As a result, the frequency of collisions between glycerol and methanol is increased, allowing them to collide rapidly and form a glycerol phase that separates from the reaction mixture [27].

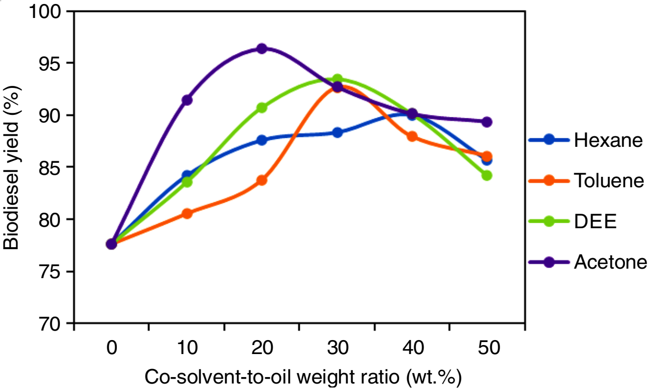

Fig. 1. Effect of cosolvent-to-oil weight ratio on biodiesel yield (catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6: 1; temperature 30 ± 1 °C; reaction time 30 min, reaction stirring speed 600 rpm)

TABLE-2
PHYSICO-CHEMICAL PROPERTIES OF BIODIESEL

Physico-chemical property	WCO methyl ester	ASTM D6751 biodiesel
Density at 15 °C (g/cm^3)	0.887	0.860-0.900
Kinematic Viscosity (cSt at 40 °C)	3.78	1.9-6.0
Acid value (mg KOH/g)	0.25	< 0.5
Flash point (°C)	155	> 130

Effect of reaction time: The duration of the reaction has been identified as a critical factor in the production of biodiesel. It is crucial to regulate the temperature of the transesterification reaction, as the reaction rate constant is strongly influenced by temperature [28]. When potassium methoxide or cosolvent solution was introduced to the vegetable oil at the initial transesterification stage, a distinct cloudy mixture was noticed. This was because the reactants are incompatibly miscible due to mass transfer resistance, demanding sufficient reaction time to overcome the intermolecular and intramolecular interactions between them [19]. However, it has been discovered that adding a cosolvent to the transesterification alcohol reduces the reaction time. To investigate the effect of time on the process, transesterification was carried out using a 6: 1 methanol-to-WCO molar ratio, 20 wt.% cosolvent and 1 wt.% KOH as the catalyst at a reaction temperature of 30°C and a stirrer speed of 600 rpm. The reaction time varied between 5, 10, 15, 20, 25 and 30 min. As the reaction time increased, the biodiesel yield percentage increased. The reaction was initially delayed due to the mixing and dispersion of alcohol into the oil. After a while, the reaction speeded up considerably further. At 10 min reaction time, the most significant yield of 97.25% was recorded and then stabilized as the reaction time was increased further for acetone. As indicated in Fig. 2, hexane, toluene and DEE did not significantly improve biodiesel yield compared to acetone over a short time of reaction. These cosolvents produced the highest yield for a reaction period of 25 min, resulting in 90.57%, 90.90% and 91.01%, respectively, under identical reaction conditions. However, the results demonstrated that while increasing the reaction time from 25 min had no significant influence on biodiesel yield, it did result in a decrease in product yield. This was because a longer reaction time increased the hydrolysis of esters by increasing the reverse reaction of transesterification, resulting in the loss of esters and the formation of additional fatty acids.

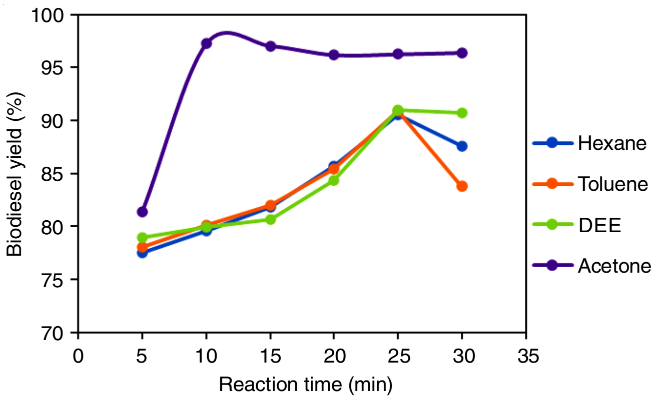

Fig. 2. Effect of reaction time on biodiesel yield (catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 20 wt.%; temperature 30 ± 1 °C; reaction stirring speed 600 rpm)

Effect of reaction temperature: Because the reaction rate constant is a vital function of temperature, optimizing the reaction temperature during transesterification is critical. The increased reaction temperature can significantly improve the mass transfer rate of transesterification by providing sufficient external kinetic energy to oil and methanol. As a result, the number of collisions will increase, effectively completing the reaction [28]. Additionally, as the reaction temperature rises, the degree of hydrogen bonding decreases, resulting in decreased polarity and increased hydrophobicity due to the decreased dielectric constant. As a result, non-polar triglycerides dissolve more readily in alcohol, resulting in the formation of a single phase of triglyceride and methanol [26]. While increasing the temperature of the oil can reduce its viscosity and increase mass transfer and reactivity between the reactants, it can also cause the oil's viscosity to decrease. Additionally, elevated temperatures can enhance saponification, resulting in a reduction in FAME yield [29]. The reaction temperature should be lower than the boiling points of methanol and the cosolvent to prevent these components from evaporating [30]. As illustrated in Fig. 3, the effect of reaction temperature was investigated by varying the reaction temperature between 30 ± 1 ºC and 60 ± 1 ºC. All experiments were conducted at 600 rpm, a 1:6 WCO-to-methanol molar ratios and a 1 wt.% KOH concentration for a reaction time of 30 min. As shown in Fig. 3, biodiesel production increased proportionately as reaction temperature increased, reaching the maximum yield of 98.46% at 40 ºC using acetone as the cosolvent, followed by toluene (97.04% at 60 ºC), hexane (92.41% at 40 ºC) and DEE (90.74% at 30 ºC). This can be explained by the fact that when the reaction temperature exceeds the boiling point of the cosolvent, the cosolvent may evaporate, resulting in the creation of multiple phases in the reaction mixture. As a result, the reaction temperature is crucial in the transesterification of WCO.

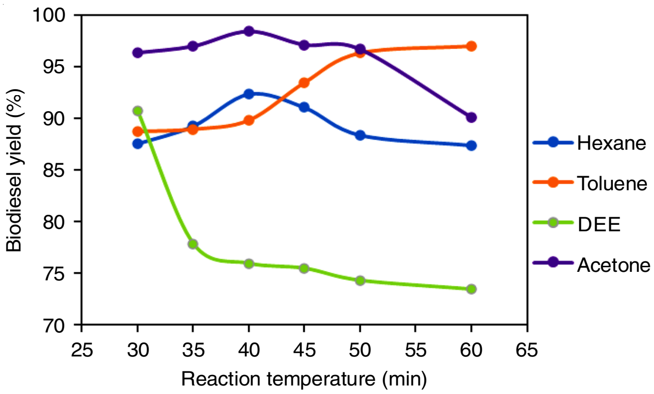

Fig. 3. Effect of reaction temperature on biodiesel yield (catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 20 wt.%; reaction time 30 min, reaction stirring speed 600 rpm)

Effect of stirring speed: Due to the limited solubility of methanol in oil, mass transfer resistance is crucial during the transesterification reaction's first stage, since the reactants form a two-phase liquid solution. Therefore, the reaction is diffusion-controlled, with a slow rate due to poor diffusion between the phases. While the methyl esters are produced to act as a solvent for the reagents, stirring the mixture is required to achieve the desired reaction rate [29-31]. Therefore, the effect of stirring speed on the transesterification reaction was investigated for non-cosolvent and cosolvent-added reaction systems by varying the stirring speed from 200 to 700 rpm. As shown in Fig. 4,

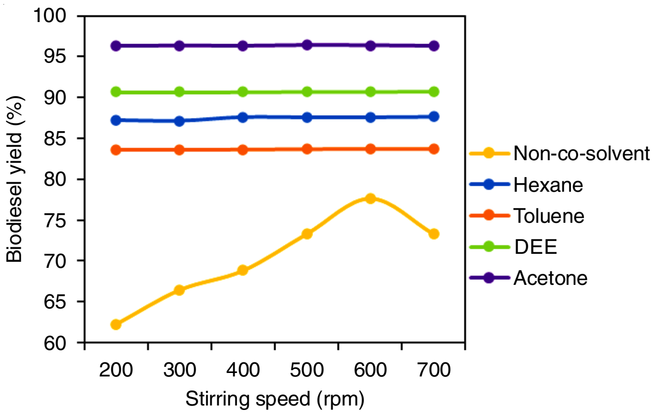

Fig. 4. Effect of stirring speed on biodiesel yield (catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 20 wt.%; reaction time 30 min, temperature 30 ± 1 °C)

increasing the stirring speed from 200 to 600 rpm increased the biodiesel yield significantly in non-cosolvent reaction systems while remaining constant in cosolvent reaction systems. This suggests that the mass transfer resistance of the reactants has a significant effect on their miscibility. However, by adding a cosolvent to the process, mass transfer resistance was overcome and hence stirring speed had no significant effect on the yield. This result indicated that the inclusion of cosolvent increased the miscibility of WCO and methanol at the initial stage, resulting in a nearly homogeneous transesterification process.

Effect of cosolvent on biodiesel yield: Based on the results of the previous optimization, numerous cosolvents were given different optimization values for each reaction parameter in order to increase the rate of mass transfer between the reactants and shorten the reaction time. Fig. 5 depicts the transesterification of WCO using several cosolvents under optimal reaction conditions for each cosolvent. The reaction mixture containing acetone produced the highest yield (98.46%), followed by DEE, toluene and hexane (93.75%, 92.36% and 89.02%, respectively). Acetone was determined to be the optimum cosolvent for the biodiesel synthesis from WCO due to the oil's and methanol's solubility in acetone, which results in a more homogeneous distribution of the reactants. Additionally, when acetone was utilized as a cosolvent, it produced the highest values for each parameter under the simplest conditions compared to the other cosolvents used in this study. Acetone is an aprotic solvent that dissolves both highly polar methanol and nonpolar WCO triglyceride, forming a homogenous reaction system that accelerates the transesterification reaction between the methanol and WCO. Additionally, when KOH is utilized as a catalyst, acetone aids in the stability of the methoxide ions generated. These methoxide ions operate as a reactive intermediate in the S_N2 reaction that converts triglycerides to methyl esters, according to Hájek *et al.* [23]. Due to the comparable boiling temperatures of acetone and methanol (56.5 ºC and 64.7 ºC, respectively), those solvents can be separated concurrently for recycling purposes. While hexane has a similar boiling point (69 ºC) with methanol, it is a non-polar solvent and incompatible with methanol. On the other hand, because toluene has a substantially more significant boiling point than methanol, the separation process would require a proportionately greater quantity of energy. Because DEE has a low boiling point, the reaction temperature is less than 34.5 ºC; consequently, the boiling point of DEE acts as a temperature limiter, preventing the mass transfer barrier from being totally overcome. As illustrated in Table-1, the solvents' polarity index appropriately explained the cosolvent's influence on the biodiesel output. Methanol and acetone have the same polarity index (5.1); however, DEE, toluene and hexane have lower values than methanol.

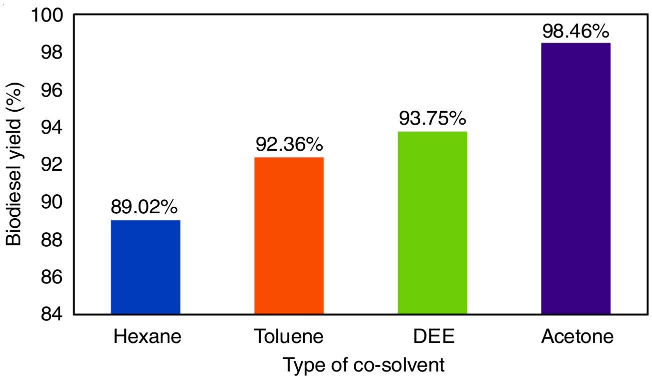

Fig. 5. Effect of cosolvent on biodiesel yield (Hexane: catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 40 wt.%; reaction temperature 40 °C reaction time 25 min, reaction stirring speed 600 rpm, Toluene: catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6: 1; cosolvent-to-oil 30 wt.%; reaction temperature 60 °C; reaction time 25 min, reaction stirring speed 600 rpm, DEE: catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 30 wt.%; reaction temperature 30 °C; reaction time 25 min, reaction stirring speed 600 rpm, Acetone: catalyst amount 1 wt.%; methanol-to-oil molar ratio of 6:1; cosolvent-to-oil 20 wt.%; reaction temperature 40 °C; reaction time 10 min, reaction stirring speed 600 rpm)

Physico-chemical properties of biodiesel: The physical and chemical properties of biodiesel produced from WCO with acetone as a cosolvent were determined using the ASTM D6751 standard. The compliance of the manufactured biodiesel with ASTM standards was demonstrated under optimal conditions, which included 1 wt.% KOH; a methanol-to-oil molar ratio of 6:1; a reaction time of 10 min; a reaction temperature of 40 ºC; a reaction speed of 600 rpm and an acetone-to-oil weight ratio of 20% (Table-2). This result demonstrates the excellent potential for using acetone in the production of biodiesel.

Conclusion

The effect of five cosolvents on transesterification reaction parameters such as cosolvent-to-oil weight ratio, reaction time, reaction temperature and stirring speed, as well as biodiesel yield, was examined. It was found that the addition of a cosolvent improved mass transfer between the phases of the transesterification process. As a result, even at room temperature, large biodiesel yields can be achieved in a short reaction time. Acetone was the most effective cosolvents, followed by diethyl ether (DEE), toluene and hexane. The acetone one might be thought of as an aprotic solvent because it dissolved both highly polar methanol and non-polar WCO triglyceride, improving oil and methanol's solubility. The study found the minimal cosolvent-to-oil weight ratios necessary for complete dissolution in the presence of several types of cosolvents. The optimal cosolvent in the transesterification system was determined to be the one with the lowest cosolvent-to-oil weight ratio. Excessive cosolvent addition to the reaction system was decreased the transesterification product yield and increased the operating costs. Incorporating a cosolvent was shown to reduce the time required for phase separation. Acetone demonstrated a higher biodiesel yield with a shorter reaction time (10 min) than other solvents. On the other hand, a longer reaction time increased the hydrolysis of esters by boosting the reverse transesterification reaction, resulting in the loss of esters and the creation of more fatty acids. Additionally, it was discovered that maintaining the reaction temperature near the boiling point of cosolvent increased biodiesel output. When the temperature of reaction mixture surpasses the boiling point of cosolvent, the cosolvent may evaporate, resulting in the formation of several phases. The physico-chemical properties of the biodiesel produced in the presence of acetone were determined to be within the ASTM limit.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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