

A Review of Biodiesel Production from Renewable Resources: Chemical Reactions

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Biodiesel has become an attractive renewable fuel due to carbon footprint reduction. Greenhouse gas footprint is minimised when the locally available renewable feedstock is utilised for cleaner biodiesel production. This biodiesel has similar characteristics of diesel oil which allows its use in compression motors without extensive engine modifications. One of the greenest choices to address the ever growing demand for cleaner energy is renewable biomass power. Contemporary development and work have been aimed at transforming these bio-based resources into practical gas and liquid fuels that comply with the current petro-based chemical production infrastructure. Biodiesel is a prospective alternative to petroleum fuels due to its clean, renewable and ecologically beneficial characteristics. The transesterification cycle is applied to the processing of cleaner biodiesel manufacturing. Comprehensive research is underway to develop more effective biodiesel processing processes. The different new technologies are both solid catalysts and supercritical processes that are not catalytic. A review of the various methods used for the successful synthesis of biodiesel includes the homogeneous, heterogeneous and enzyme catalyst, microwave and ultrasonic synthesis followed by the supercritical synthesis of biodiesel. The chemical reactions of biodiesel, i.e. transesterification, esterification and interesterification have been presented by this paper and the merits and limitations of these chemical reactions for biodiesel discussed.

1. Introduction

The use of fossil fuel has significantly increased through sustained developments in industrialisation, motoring and shipping (Chuah et al., 2021). Biodiesel is an alternative fuel for the replacement of petroleum diesel solution. Due to its similarity to diesel fuel, such as heated value and viscosity, Biodiesel can be used in a diesel engine without major alterations (Bokhari et al., 2016). There are three chemical reactions viz. transesterification, esterification and interesterification reactions to produce biodiesel (Yusup et al., 2015). Transesterification of oil with alcohol is the most popular process for biodiesel production. One mole of triglyceride required 3 moles of alcohol during transesterification (Chuah et al., 2016). The stepwise transesterification reaction is presented in Figure 1. Excess alcohol is needed to shift the reaction forward to the desired product, i.e. fatty acid methyl ester, due to the reversible nature of the transesterification reaction. Waste oil to alcohol molar ratio 1:6 is the most commonly used for transesterification, which is able to obtain 98 wt.% ester conversion (Issariyakul and Dalai, 2014). However, the optimum molar ratio of oil to alcohol is depending on the oil quality and type of oil used (Asif et al., 2017). An increase of oil to alcohol molar ratio to a certain level does not further increase the conversion during transesterification because the reaction mixture polarity increased and increased the solubility of glycerol back to the ester phase and promoting the reverse reaction between glycerol and ester or glycerides, consequently reducing the ester yield (Issariyakul and Dalai, 2014). In terms of ester conversion percentage in transesterification, potassium hydroxide as alkali catalyst is higher compared to sodium hydroxide, which is 96 (Issariyakul et al., 2007) and 76.9 wt.% (Meng et al., 2008) under operating parameters of waste oil to methanol molar ratio of 1:3, 1 wt.% alkali catalyst, 50 °C and 60 min reaction time. There are various alcohols that can be used in transesterification reaction, such as methanol, ethanol,

propanol, and butanol. Long-chain alcohol such as ethanol and propanol has higher miscibility with oil and lower reaction time compared to methanol. Methanol (short carbon chain) is the most commonly used in transesterification reaction due to its lower cost and higher nucleophilicity compared to ethanol (long carbon chain) (Issariyakul and Dalai, 2014). Issariyakul et al. (2007) reported that the use of methanol in transesterification reaction of waste fryer grease resulted in 82 wt.% biodiesel yield recovery, whereas only 62 wt.% biodiesel yield recovery is observed when using ethanol.

Esterification reaction occurs in a relatively straightforward way, in which one mole of free fatty acid reacts with one mole of alcohol to produce one mole of biodiesel and one mole of water. Both transesterification and esterification reactions (endothermic processes) can be significantly influenced by some operating parameters, such as reaction temperature, loading catalyst and a molar ratio of oil to alcohol. The immiscibility behaviour of glycerides and alcohol is often referred to as mass transfer resistance, which can be overcome by several methods such as rigorous mechanical stirring, supercritical condition, microwave, ultrasonic and hydrodynamic cavitation. In general, the activation energy for the transesterification process is higher compared to the esterification process. It could be attributed to the stability of the triglycerides molecules (esters), which required more energy to break prior to the reaction with alcohol in the transesterification process. Free fatty acid is ready to react in the esterification process due to its less stable molecular structure. Similarly to other carboxylic acids, a free fatty acid is capable of donating the proton (H⁺), which makes them susceptible to the reaction.

The interesterification of triglycerides with methyl acetate instead of methanol provides a promising alternative to transesterification due to the formation of triacetin instead of glycerol (Maddikeri et al., 2014). One mole of triglyceride required 3 moles of methyl acetate during interesterification to produce one mole of triacetin and 3 moles of alkyl esters (Figure 2). Interesterification has been mostly studied in the presence of enzymes or under supercritical conditions. However, both approaches have their own disadvantages. The drawbacks of the enzymatic approach require longer reaction time and higher production costs as well as difficulty at industrial scale due to the need for careful control of the reaction parameters and inherent slowness of the reaction, whereas the supercritical approach requires significantly high pressure (20 - 40 MPa) and temperature (350 – 400 °C) as well as significant excess of methyl acetate with 1:42 molar ratio of oil to methyl acetate (Maddikeri et al., 2014). The high oil to methyl acetate molar ratio under supercritical resulted in a higher cost for the evaporation of the unreacted methyl acetate (boiling point: 56.9 °C). Although hydrodynamic cavitation (one of the intensification processes) has been used for biodiesel production from soybean, vegetable oil, Thumba oil, frying oil and Nagchampa oil based on the esterification/transesterification reactions, however for interesterification reaction, Maddikeri et al. (2014) are the first to attempt to apply hydrodynamic cavitation technically for biodiesel production. Throughout this paper, the chemical reactions of biodiesel are presented. The merits and limitations of these chemical reactions for biodiesel have been discussed in this paper.

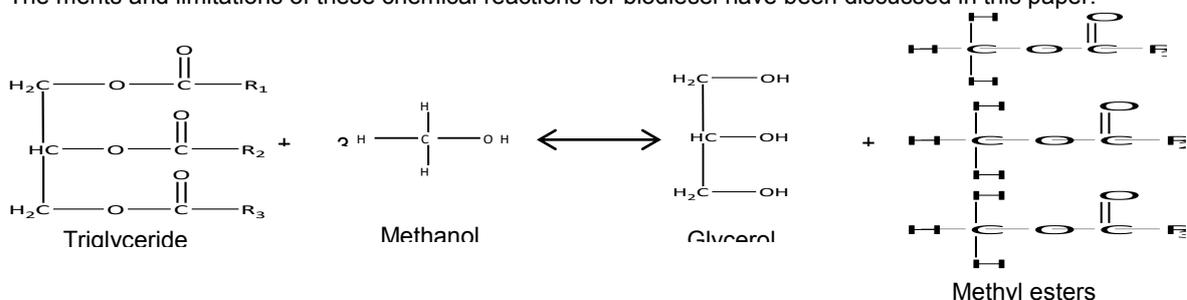


Figure 1: Scheme for stepwise transesterification reaction

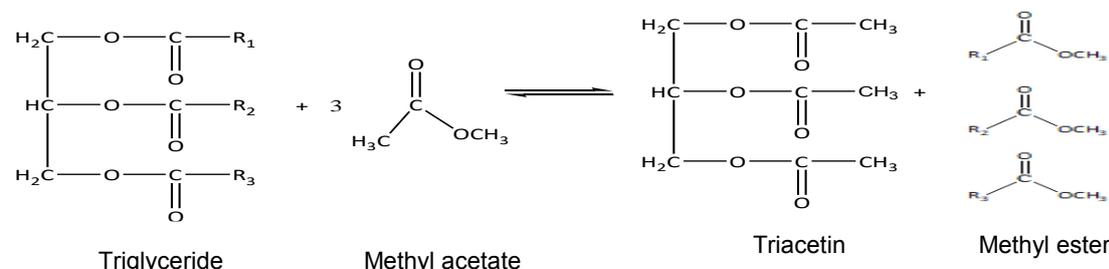


Figure 2: Scheme for stepwise interesterification reaction

2. Chemical reactions

Chemical reactions for biodiesel production from sustainable feedstock is attaining concerns and attention. A detailed discussion of different chemical reactions for biodiesel has been presented and discussed in the following sections.

2.1 Homogeneous-catalysed

Presently, homogeneous alkali catalysed, e.g. sodium methoxide, sodium hydroxide and potassium hydroxide transesterification is the commonly adopted process worldwide at an industrial scale for biodiesel production (Kulkarni and Dalai, 2006). There are several advantages of homogeneous alkali catalysed compared to the acid catalyst in transesterification: (i) widely available and cheap, (ii) high conversion of methyl ester in a shorter time, (iii) reaction can occur at mild reaction condition. Kulkarni and Dalai (2006) reported that homogeneous alkali catalysed transesterification could be 4,000 fold faster compared to the acidic catalyst. Lam et al. (2010) reported that some researchers claimed alkali catalysed could tolerate the free fatty acid value less than 0.5 wt.% or less than 2 wt.% in order to prevent saponification occurrence. The soap formation during reaction resulted in a difficult separation of glycerol from the product, which resulted in ester yield reduction. Shahbazi et al. (2012) investigated biodiesel production from RBD (Refined, Bleached and Deodorised) palm oil with 1:6 molar ratio of oil to methanol, 1 wt.% alkali catalyst (potassium hydroxide and sodium hydroxide) and agitation speed of 600 rpm for 60 min under 60 °C reaction temperature using the mechanical stirring method. They stated that KOH was considered the best catalyst for better conversion of methyl ester during transesterification reaction. Wan Ab Rashid et al. (2014) obtained a biodiesel yield of 91 wt. % through homogeneous base catalysed transesterification of cooking palm oil at 60 °C, 1:23 oil to methanol molar ratio, 5 wt.% catalyst loading and 3 min in milichannel reactor. Kafuku and Mbarawa (2010) studied transesterification of *Moringa oleifera* oil under conventional heating in the presence of potassium hydroxide. It was concluded that 82 wt.% conversion of biodiesel at 1:6 molar ratio of oil to methanol and 1 wt.% catalyst concentration was achieved after 60 min of reaction time. Kumar et al. (2013) reported that 90 wt.% conversion was obtained after only 40 min for conventional heating with 0.5 wt.% sodium hydroxide. With respect to other sources of oil, work on conventional heating for transesterification with sodium hydroxide or potassium hydroxide were reported, achieving 81.0 – 99.4 wt.% conversions in 40 - 90 min and 73.9 – 99.5 wt.% yields in 30 – 120 min. Against conventional heating, it was also confirmed that the ultrasonic and milichannel reactors could enhance transesterification of oil with alcohol, allowing high yields and conversions into biodiesel at a shorter time.

Table 1: Homogeneous catalysed used in biodiesel production

| Source of oil | Reaction condition | Maximum yield/conversion (wt. %) | Method | Reference |
|----------------------------|---|----------------------------------|---------------------|------------------------------------|
| RBD palm oil | 1 wt.% KOH, 60 °C, 60 min, 600 rpm, 1:6 oil to methanol molar ratio | 90 (conversion) | Mechanical stirring | Shahbazi et al., 2012 |
| Waste cooking oil | 1 wt.% NaOH, 50 °C, 90 min, 1:6 oil to methanol molar ratio | 89.8 (conversion) | Mechanical stirring | Meng et al., 2008 |
| Waste cooking oil | 1 wt.% KOH, 70 °C, 60 min | 98.2 (conversion) | Mechanical stirring | Agarwal et al., 2012 |
| Jatropha oil | 1 wt.% NaOH, 60 °C, 90 min, 1:5.4 oil to methanol molar ratio | 98 (yield) | Mechanical stirring | Chitra et al., 2005 |
| <i>Hibiscus sabdariffa</i> | 1.5 wt.% KOH, 60 °C, 60 min, 600 rpm, 1:8 oil to methanol molar ratio | 99.4 (conversion) | Mechanical stirring | Nakpong and Wootthikanokkhan, 2010 |
| <i>Moringa oleifera</i> | 1 wt.% KOH, 60 °C, 60 min, 400 rpm | 82 (conversion) | Mechanical stirring | Kafuku and Mbarawa, 2010 |

2.2 Heterogeneous-catalysed

The most pressing disadvantages of heterogeneous acid catalyst transesterification are high cost for catalyst synthesis, required high reaction temperature, high chemical consumption and long reaction time due to mass transfer limitation between liquid reactants and solid catalyst. In general, the reaction temperature should be kept below the boiling point of the reactant, e.g. methanol (65 °C) and ethanol (78 °C). Heterogeneous acid catalyst required a high temperature up to 200 °C, and pressure is needed to be applied to the reaction mixture in order to maintain the reacting alcohol in a liquid state (Issariyakul and Dalai, 2014). However, the advantages of heterogeneous acid catalyst transesterification are less sensitivity to free fatty acid, and water content in the oil, esterification and transesterification can be performed simultaneously, no washing is required resulted in a reduction of the acidic wastewater, easy separation of catalyst from the reaction mixture, easy recovery, regeneration and recycling of catalyst and less corrosion to the equipment (Chuah et al., 2017). Lotero et al. (2005) stated that the ideal solid acid catalyst for the transesterification of waste cooking oil should have characteristics such as an interconnected system of large pores, a moderate to a high concentration of strong acid sites and a hydrophobic surface. A more in-depth study using a solid acid catalyst in the transesterification of high free fatty acid oil is required to overcome its slow reaction rate and undesired side reaction. Miao and Gao (1997) reported that the acid property could be enhanced by impregnating ultrafine crystalline zirconium oxide (ZrO_2 - metal oxide) with sulphuric acid. Jitputti et al. (2006) claimed that without sulphate of ZrO_2 as a catalyst used in transesterification of crude palm kernel oil and crude coconut oil, only 69.0 wt.% and 54.3 wt.% of methyl ester content were achieved. It could be concluded that modification of metal oxide surface acidity is the key factor in obtaining high methyl ester conversion. Application of ZrO_2 supported La_2O_3 in the transesterification of sunflower oil with methanol to produce biodiesel was also reported by Sun et al. (2010). Transesterification reaction was performed with 1:30 oil to methanol molar ratio with the presence of catalysts (ZrO_2 , La_2O_3 and 7 - 28 wt.% La_2O_3/ZrO_2) at a reaction temperature of 200 °C for 5 h reaction time under magnetic agitation. They found that the highest methyl ester conversion of 96 wt.% was attained with a catalyst of 21 wt.% La_2O_3/ZrO_2 calcinated at 600 °C. However, they also claimed that with a single catalyst (unsupported catalyst), i.e. ZrO_2 and La_2O_3 , in transesterification reaction of sunflower oil, only 31.0 wt.% and 80.8 wt.% of methyl ester conversion were obtained. Table 2 compares the reaction condition and performance of homogeneous and heterogeneous catalysed used in biodiesel production. The insights into the structure-function interaction of heterogeneous catalysts lead increasingly to the conclusion that catalysts are by far not materials with static surface and bulk structures. Instead, they are dynamic entities that can modify their structure (for example surface morphology or composition) depending on the local reaction conditions in the reactor. The process conditions are not affected by the mass flow rate into the process. It is affected due to the presence of free fatty acid content, acid value, triglycerides, saturation or unsaturation in the feedstock oils.

Table 2: Heterogeneous catalysed used in biodiesel production

| Source of oil | Catalyst | Reaction condition | Maximum yield/conversion (wt. %) | Method | Reference |
|------------------------|---------------------|---|----------------------------------|---------------------|-----------------------|
| Crude palm kernel | SO_4^{2-}/SnO_2 | 200 °C, 60 min, 350 rpm, 50 bar under N_2 , 1:60 oil to methanol molar ratio, 3 wt.% catalyst | 95.4 (conversion) | Mechanical stirring | Jitputti et al., 2006 |
| | SO_4^{2-}/ZrO_2 | | 88.3 (conversion) | | |
| Crude coconut oil | SO_4^{2-}/SnO_2 | 200 °C, 5 h, 1:30 oil to methanol molar ratio, 5 wt.% catalyst | 95.8 (conversion) | Mechanical stirring | Sun et al., 2010 |
| | SO_4^{2-}/ZrO_2 | | 93.0 (conversion) | | |
| | ZrO_2 | | 31 (conversion) | | |
| Sunflower oil | La_2O_3 | 200 °C, 5 h, 1:30 oil to methanol molar ratio, 5 wt.% catalyst | 80.8 (conversion) | Mechanical stirring | Sun et al., 2010 |
| | 21% La_2O_3/ZrO_2 | | 96 (conversion) | | |
| <i>Scenedesmus</i> sp. | WO_3/ZrO_2 | 80 °C, 20 min, 350 rpm, 1:45 oil to methanol molar ratio, 4 wt.% catalyst | 71.4 (conversion) | Ultrasonic | Guldhe et al., 2014 |

3. Conclusions

Because of its renewable nature and environmental benefits, biodiesel is a promising and more appealing fuel for diesel engines. The main issue to consider is that biofuels are more expensive than fossil fuels. Using low-quality feedstock, such as non-edible oils and animal fats, which do not compete with food supply and land for food cultivation, is thought to be an effective way of lowering biodiesel production costs. The paper demonstrates that biodiesel feedstock can be non-edible oil or edible oil. Biodiesel is made from edible or inedible oil via a transesterification reaction in which the organic group (alkyl) of alcohol is substituted with the organic group of a triglyceride, resulting in fatty acid alkyl ester and glycerol. Biodiesel production is a simple process that does not require a licence or complicated technology. The alkaline catalysed transesterification method is the most commonly used because it is the quickest reaction, has the highest yield, has the mildest reaction conditions, is the least expensive, corrosive, and toxic. Catalysts are important in increasing yield and conversion, as well as saving energy required to run the reaction at higher temperatures without a catalyst. Acids and alkalis are commonly used as liquid catalysts in the production of biodiesel. Solid catalysts based on transition metals and noble metals are used in the production of renewable fuels. By applying homogeneous catalyst transesterification in biodiesel production, about 5-fold lower methanol consumption, 5-fold shorter time reaction and 3-fold lower temperature reaction compared to heterogeneous catalyst transesterification. The second major contributor to the environmental footprint after the raw material is the energy efficiency in biodiesel production. It has been observed that about 4-fold reduction in the environmental footprint using homogeneous catalyst transesterification compared to heterogeneous catalyst transesterification. In terms of separation time (time-saving) and free fatty acid sensitivity, heterogeneous catalyst transesterification proved to be more advantageous over homogeneous catalyst transesterification. In addition, in-situ (trans) esterification is an alternative route in biodiesel production. It involves lesser steps as it eliminates the need for lipid or oil extraction prior to transesterification. Commercial units are in operation all over the world, producing massive amounts of biodiesel each year, demonstrating that biodiesel is a mature technology for industrial applications, particularly in countries where biomass is abundant. Eventually, the amount of diesel consumed in the world is massive, having a negative impact on the economy and the environment. As a result, accelerating the use of biodiesel will have a significant impact on the environment, vehicle engines, independence from crude oil, investment, and the economy by creating jobs.

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