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REVIEW ARTICLE

Improvements in the stability of biodiesel fuels: recent progress and challenges

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Abstract

Fewerfossil fuel deposits, price volatility, and environmental concerns have intensified biofuel-based studies. Saccharification, gasification, and pyrolysis are some of the potential methods of producing carbohydrate-based fuels, while lipid extraction is the preferred method of producing biodiesel and green diesel. Over the years, multiple studies have attempted to identify an ideal catalyst as well as optimize the abovementioned methods to produce higher yields at a lower cost. Therefore, this present study comprehensively examined the factors affecting biodiesel stability. Firstly, isomerization, which is typically used to reduce unsaturated fatty acid content, was found to improve oxidative stability as well as maintain and improve cold flow properties. Meanwhile, polymers, surfactants, or small molecules with low melting points were found to improve the cold flow properties of biodiesel. Meanwhile, transesterification with an enzyme could be used to remove monoacylglycerols from oil feedstock. Furthermore, combining two natural antioxidants could potentially slow lipid oxidation if stainless steel, carbon steel, or aluminum are used as biodiesel storage materials. This present review also recommends combining green diesel and biodiesel to improve stability. Furthermore, green diesel can be co-produced at oil refineries that are more selective and have a limited supply of hydrogen. Lastly, next-generation farming should be examined to avoid competing interests in food and energy as well as to improve agricultural efficiency.

Keywords Biofuel · Biodiesel stability · FAME composition · Additives · Cold flow improver · Green diesel

Introduction

Many countries around the world depend on fossil fuels to sustain their industries and economies. However, the fossil fuel deposits that the energy and petrochemical industries heavily rely on are diminishing. At present, global natural gas and oil reserves are predicted to completely run out by 2069 and 2067, respectively (BP 2018). Fossil fuels cause serious environmental problems, especially air pollution. The amount of carbon dioxide released by burning fossil fuels significantly contributes to global warming, which leads to various other dangers (Solarin et al. 2018). Meanwhile, other pollutants such as sulfur oxide result in acid rain, which negatively affects crop yields. Apart from that, the numerous oil spills (≈1190 thousand tonnes) that have occurred between 1970 and 1990 have destroyed nearby terrestrial and aquatic ecosystems (Chen et al. 2015; Farrow et al. 2016; Lee and Jung 2015).

Oil price volatility has posed a problem over the last 50 years. Supply, demand, and geopolitical interests are largely believed to be the cause of fluctuating oil prices.

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For instance, oil prices fell into the negatives in April 2020 due to oversupply and extremely low demand following the advent of the COVID-19 pandemic (Jefferson 2020), during which approximately 40% of the world's population was forced to self-isolate in their homes. Therefore, as the road transportation and aviation industry accounted for 58% of the oil consumption in the Organization for Economic Cooperation and Development (OECD) countries, oil demand dropped significantly. At the same time, many oil storage facilities as well as oil tankers were completely full as no one predicted that a global pandemic would occur at the end of 2019. Although oil prices rose to US$25 on 1 May 2020, several oil companies revised their strategies for the future (Agency 2020). For instance, the Chevron Corporation acquisitioned Noble Energy Incorporated, a natural gas company (Blackmon 2020), while British Petroleum plc invested US$5 billion/year in low-carbon power (BP 2020). Only the agricultural sector experienced positive trends during the pandemic as the increasing demand for food and other nutrients (Pu and Zhong 2020). As such, biofuel development has taken center stage over the years.

Figure 1 presents some biofuel options and their processing methods. Biomass predominantly comprises cellulose and lignin, or lignocellulose (Yoo et al. 2020), and can be converted into bio-oil or synthetic gas either via pyrolysis or gasification. Bio-oil can also be used to produce jet fuel post-hydrotreating and refining. However, synthetic gas is an intermediate feedstock that can be used to produce fuel and chemicals via the Fischer–Tropsch synthesis method or gas fermentation. Agricultural waste is another potential feedstock option. For instance, aromatic sugar is an intermediate product that is used to produce ethanol (Galadima et al. 2022c; Zheng et al. 2009). As such, multiple studies have examined improving the efficiency of the sugar production process. Lastly, lipids and vegetable oils can also be extracted from algae species or plants (Karmakar and Halder 2019), and the resulting products can be differentiated into hydrotreated vegetable oils or fatty acid methyl esters, depending on the refining process. Over the last decade, fatty acid methyl esters have quickly become a popular biodiesel fuel option and their demand in the transportation industry has increased (Yue et al. 2014). Studies have indicated that biodiesel can sometimes be produced alongside the sugar fermentation process to lower the investment cost. In this method, the bioethanol derived from corn stover as feedstock is the preferred choice in this route (Shafiei Alavijeh et al. 2020).

Over the years, multiple studies have examined biodiesel fuel properties. This is because the possibility of adapting pure biodiesel or biodiesel blends as fuel in compression-ignition engines has convinced many practitioners and governments to invest in this field (Anis and Budiandono 2019). Furthermore, biofuels emit significantly

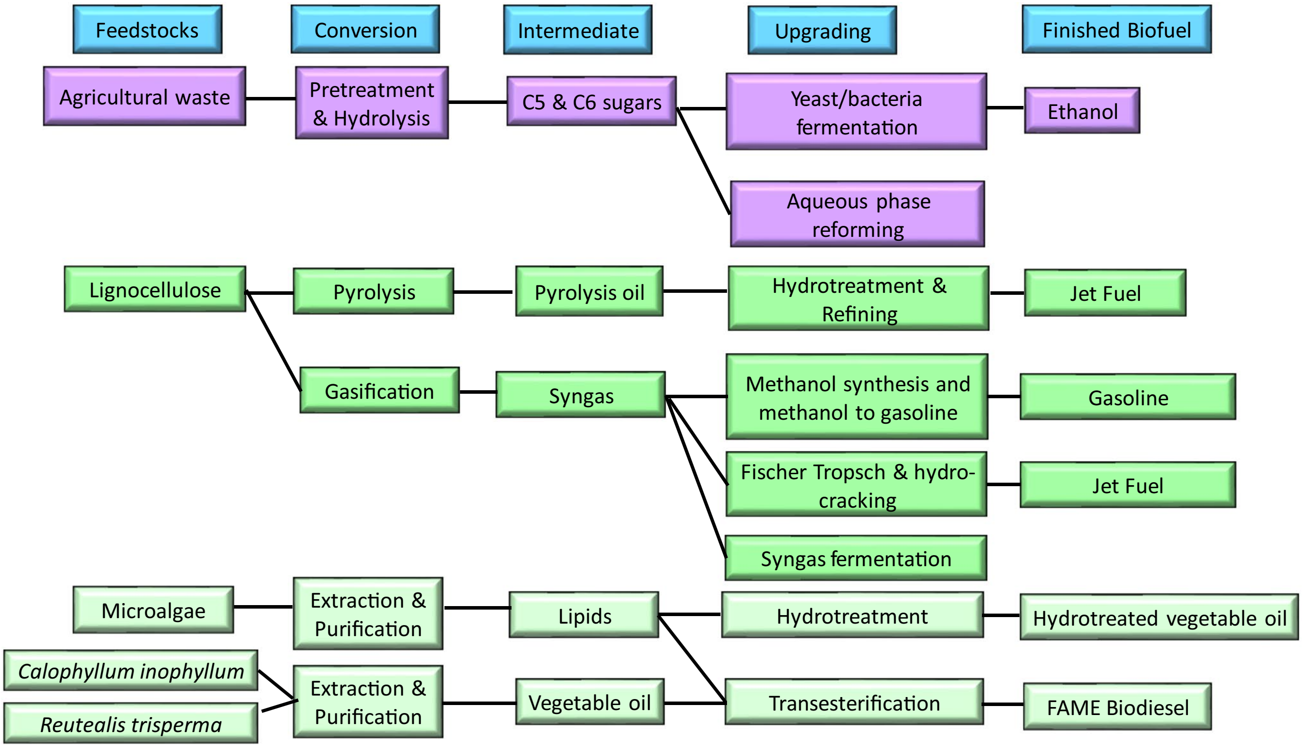


Fig. 1 Conversion of biomass to biofuel

less carbon, nitrogen, and sulfur than fossil fuels, as they contain 11% more oxygen.

However, biodiesel fuels are still plagued with several drawbacks such as poor cold flow properties, higher fuel consumption, poor oxidative stability, material corrosion, and wear (Vicente et al. 1998, 2004). Furthermore, as biofuels form alcohols, carboxylic acid, and aldehydes during oxidation, they formed insoluble deposits that clog the fuel injector pins, resulting in poor atomization and clogs in the fuel filter. Some studies indicated that oxidative degradation must be extensive for density, cloud point, and other physical properties to be severely affected. Apart from that, incomplete fuel combustion is generally caused by an increase in viscosity, which also occurs in fuels that have already undergone severe degradation.

At present, multiple biodiesel-based studies have examined process intensification (Athar and Zaidi 2020), glycerol utilization (Muraza 2019), various biodiesel feedstock sources (Ambat et al. 2018), integrated thermochemical conversions (Casoni et al. 2019), tribology (Sundus et al. 2017), bio-lubricants (Masudi and Muraza 2018), and the use of waste materials in catalyst design (Galadima and Muraza 2020). As the world's largest producer of biodiesel, Indonesia has prioritized increasing the biodiesel-to-diesel blending ratio from B20 to B30. However, although Indonesia has sufficient production capacity (Febriansyah et al. 2020), the high blend of biodiesel has led to several problems such as filter blocks (Komariah et al. 2018) as well as poor heating, cold flow properties, and density (Rodríguez-Fernández et al. 2019). Despite growing global concerns, only a handful of studies have examined biodiesel stability to the best of our knowledge (Fig. 2). Therefore, this present review provides an overview of biofuels and discusses the present state of biofuel stability. The parameters influencing oxidation stability has also been summarized to outline a clear path with which to improve biodiesel stability. Lastly, next-generation farming and the potential of the industrial biomass process are discussed.

Overview of biofuel

Biofuel is a potential energy source of the future as fossil fuel reserves deplete and global warming increases. Biofuel commercialization technologies can be categorized as either lipid-based or carbohydrate-based biofuels (Fig. 3). Carbohydrate-based fuels are mainly based on lignocellulosic biomass from which sugar, bio-oil, and synthetic gas which comprises carbon monoxide and hydrogen that produced via saccharification, pyrolysis, and gasification, respectively. Anex et al. (2010) examined the cost of using the abovementioned three processes to produce 2000 tons of gasoline per day from corn stover. Firstly, the required capital for the processes ranged US$200–610 million. Their studies confirmed that pyrolysis and gasification as the lowest and highest investment, respectively. Since then, technologies have consistently developed into more efficient and cost-effective processes (Anex et al. 2010).

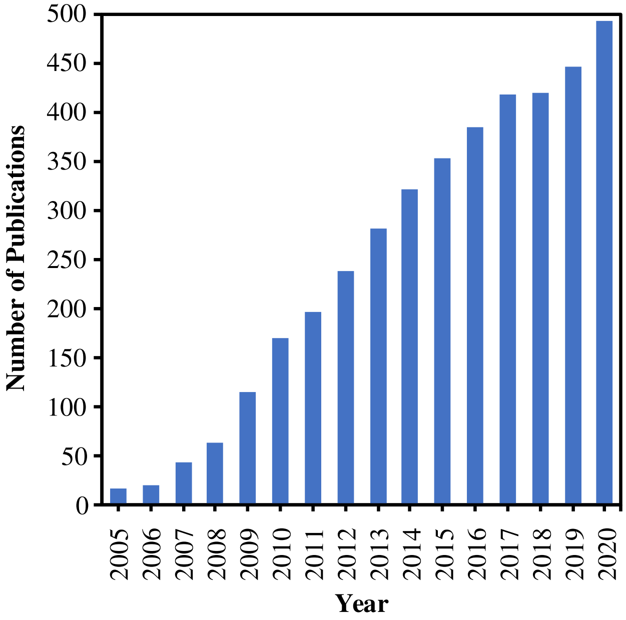


Fig. 2 Trend of publications on biodiesel stability (extracted from Web of Science)

The current trends seen in the sugar industry occurred while searching for better glucose conversion and separation technologies (Galadima et al. 2022b; Vohra et al. 2014). Although lignocellulosic biomass is a potential bioethanol feedstock, not many studies have examined its use (Ali et al. 2022). The use of lignocellulosic biomass, such as grass, woody materials, and plant residue, has been explored to prevent increasing food prices (Barbanera et al. 2018). Agricultural waste needs to be pre-treated to increase the efficiency of the bioethanol production process. Banerjee et al. (2016) used varying concentrations of sodium chloride to pre-treat mustard stalks and straws. The treated sample had a crystallinity index, porosity, and surface area that was higher than that of the untreated sample. Furthermore, the efficiency of the bioethanol production process approached 82%, with a 26% reduction in lignin content. Therefore, sodium chloride is a suitable alternative to the toxic or expensive materials that are commonly used for pre-treatment during bioethanol production (Banerjee et al. 2016). According to Barrera et al. (2016), pre-treatment is especially necessary when biomass has high lignin and hemicellulose content. These two compounds were removed using a hydrolytic system followed by enzymes or other catalysts. The study found that blue agave bagasse was more promising than sugarcane as the former did not require pre-treatment (Barrera et al.

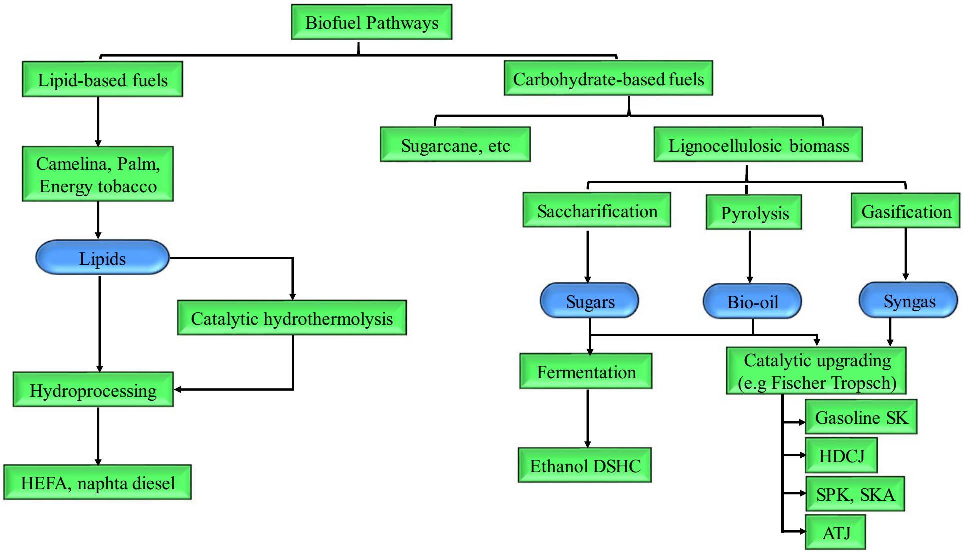


Fig. 3 Technologies for biofuel commercialization

2016). Meanwhile, Kalyani et al. (2017) used birch wood in a saccharification-anaerobic digestion process to produce a high yield of biofuel (Kalyani et al. 2017).

Although bio-oil can be extracted from various water- and oxygen-rich biomass sources, these bio-oils are corrosive to existing crude oil pipelines (Kraiem et al. 2017). Apart from that, the presence of alkali metals and charcoal residues can lead to contamination and lower oil stability. Some studies have examined the use of empty fruit bunches for bio-oil production. For instance, Ferreira et al. (2020) converted empty fruit bunches to bio-oil via slow pyrolysis at 500–700 °C. An optimization analysis indicated that temperature was the biggest influence on liquid yield (Ferreira et al. 2020). Similarly, Folgueras et al. (2017) used 30 s of fast pyrolysis at 500 ° C to treat a bamboo species. The bio-oils obtained comprised several compounds such as aromatics, heterocyclic, esters, acids, alcohols, ketones, and aldehydes. These compounds could then be converted to other chemicals (Folgueras et al. 2017). Meanwhile, García et al. (2015) converted bio-oil from sawdust to a variety of hydrocarbons using Y-type zeolites. The findings of the study led to increased interest in improving the conversion of bulky molecules to hydrocarbons. A study of bio-oil obtained from two species of wood in Northern Mexico examined the optimum temperature, flowrate, and heating rate and determined that the reaction order followed first-order kinetics (García et al. 2015). It also confirmed that the process could open the possibility of producing high-purity hydrogen (Gándara Terrazo et al. 2016). Meanwhile, pre-treatment of sunflower seed hull was preferred using phosphoric acid to obtain a high bio-oil yield (Casoni et al. 2019).

During gasification, additional apparatus are required to prevent hydrogen leaks and minimize exposure to carbon monoxide, a highly toxic gas (Zamboni et al. 2016). Xiao et al. (2020) coupled biomass co-gasification with coal gasification to sustain the auto-thermal process of a dual-loop gasification system. Under optimum conditions, olivine was found to efficiently remove tar. Meanwhile, another study that used a combination of olivine and ferric oxide as a catalyst during dual-bed gasification reported that the process required a high reformer temperature to yield higher gas and hydrogen content (Xiao et al. 2020). The catalyst was also found to carry oxygen from the combustor to the reformer (Pan et al. 2019). Peng et al. (2017) used alkali metal carbonates, specifically sodium bicarbonate, potassium bicarbonate, and potassium carbonate, as catalysts in circulating fluidized bed reactors for dual gasification (Peng et al. 2017). The study reported that the coal-to-biomass ratio plays a crucial role in the feedstock conversion and that potassium carbonate exhibited the best activity as it slower coke deposition during gasification. Meanwhile, another study reported that integrating hydrotreated empty fruit bunches and coal power plants increased energy efficiency by approximately 40% (Darmawan et al. 2017).

A bimetallic catalyst which was prepared by impregnation and calcination of carbon nanofibers with various ratios of iron and nickel showed high tar removal of up to 86% during biomass gasification and only slightly decreased after the 13th cycle. The study concluded that the high mesopore carbon and metal alloy facilitated adsorption and tar cracking via carbon deposition, which facilitated better interaction between the metals (Xie et al. 2018). Meanwhile, another study proposed a novel biomass gasification method that integrated tar steam reforming, methanation, and adsorption to enhance reforming. The nickel-doped α-ferric oxide as the catalyst during methanation and tar reforming yielded high hydrogen and methane content, about 97%. The synthetic gas was transported to the top, together with the tar, and processed during the water–gas shift reaction. The optimum condition was achieved by reducing the temperature gradient of the two processes and using calcined limestone as a carbon dioxide separator (Galadima et al. 2022a; Zhang et al. 2019).

Another study examined the crucial parameters required for the synthetic gas formation and developed an attractive method of producing synthetic gas with high hydrogen content by integrating high-density polyethylene waste and coconut shell biomass gasification. A 1:1 mass ratio of coconut shell and high-density polyethylene was found to produce the highest yield. Furthermore, the use of dolomite and nickel in the fixed and fluidized bed gasifier was found to remove 86–94% of the tar. The carbon conversion efficiency was also found to be approximately 92% under optimum conditions. A nickel catalyst was also found to be capable of converting the biochar from the gasification method into carbon nanotubes (Alipour Moghadam Esfahani et al. 2017). Meanwhile, a separate study reported simultaneously producing synthetic gas and power by chemically looping macroalgae (Zaini et al. 2017).

Another study reported that methanol can be produced via the gasification of forest residue and lignin. However, as alkali impregnation during pre-treatment only slightly increased methanol yield, the techno-economic analysis indicated that the selling price of methanol must be in the range of €90–130/MWh to compete with other methanol production methods. Furthermore, a co-addition of lignin was required when the price was below €25/MWh to generate sufficient profits (Carvalho et al. 2017).

Lastly, lipid extraction can be used to produce two products, namely fatty acid methyl esters and green diesel or hydrotreated vegetable oils. Glycerol is a by-product of the fatty acid methyl ester production process, while hydrogen must be added to produce hydrotreated vegetable oil (Fig. 4). The choice of carrier material plays a crucial role in achieving high deoxygenation activity. de Oliveira Camargo et al. (2020) examined the use of three types of supports, namely protonic zeolite Socony Mobil–5 (H-SBA-15), aluminum-substituted mesoporous Santa Barbara amorphous-15 silica (Al-SBA-15), and ultra-stable Y molecular sieve (USY). The hydrocarbon yield of oleic acid was according to the following sequence: USY (24%) < H-SBA-15 (29%) < Al-SBA-15 (42%). Furthermore, the mesoporous characteristics and the small size of the metal sites contributed to better deoxygenation and catalyst deactivation occurring between 280 and 300 °C, as evidenced by the lower hydrocarbon yield. Meanwhile, another study examined the use of zirconium dioxide, zeolite Socony Mobil–5, and activated carbon as supports for a nickel. The study concluded that zirconium dioxide was the best support with 98% conversion and 75% selectivity to n-pentadecane (de Oliveira Camargo et al. 2020). Furthermore, an observation of the gaseous product indicated that a synergistic carbon dioxide methanation and water–gas shift reaction occurred with the zirconium dioxide support (Hongloi et al. 2019). Upon obtaining potential support as well as identifying several metallic sites, Kamaruzaman et al. (2020) impregnated mesoporous silica with various metals such as cobalt, nickel, and nickel–cobalt alloy. The nickel/mesoporous silica and nickel–cobalt/mesoporous silica samples were found to yield 86% and 88% of liquid hydrocarbon (C_8-C_{17}), respectively. Furthermore, the sintering process of the cobalt was also found to initiate coke formation that reduces catalyst performance (Kamaruzaman et al. 2020).

Another study examined converting canola oil to green diesel using a nickel-molybdenum or cobalt-molybdenum catalysts supported on mesostructured γ-alumina. Both catalysts, which were prepared via wet impregnation, exhibited

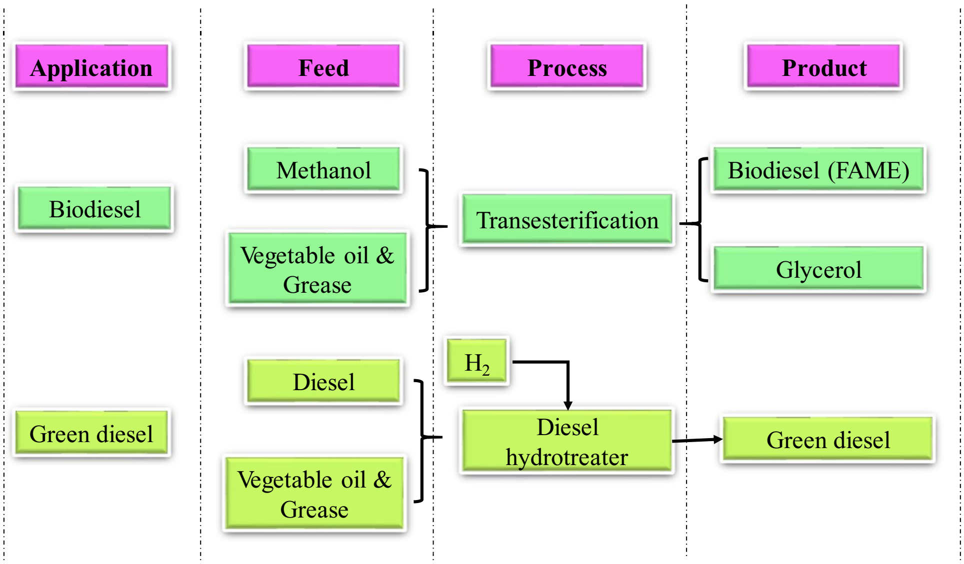


Fig. 4 Lipid extraction method of producing biodiesel and green diesel

homogenous dispersion within catalyst support. At 375 ° C and 450 psi, the canola oil to green diesel conversion reached 100%, with 70% selectivity to C_{15}-C_{18} using a nickel-molybdenum catalyst (Afshar Taromi and Kaliaguine 2018). Meanwhile, macauba oil, a non-edible oil, was converted to green diesel using a cobalt-carbon catalyst. The addition of 10 wt.% of the cobalt-carbon catalyst resulted in 98% deoxygenation within 4 h at 350 ° C and 30 bar of hydrogen pressure (de Barros Dias Moreira et al. 2020). Meanwhile, Ameen et al. (2019) compared the efficacy of wet impregnation and sono-chemical methods in synthesizing a bimetallic nickel-molybdenum catalyst that was supported on a mesoporous γ-alumina at a hydrogen-to-oil ratio of 1000 N (cm^3/cm^3) at 35 bar and 350 °C. The sonochemically supported catalyst was found to exhibit more uniform metal distribution and higher exposure to molybdenum (+IV) species with 81% selectivity to diesel hydrocarbons (Ameen et al. 2019).

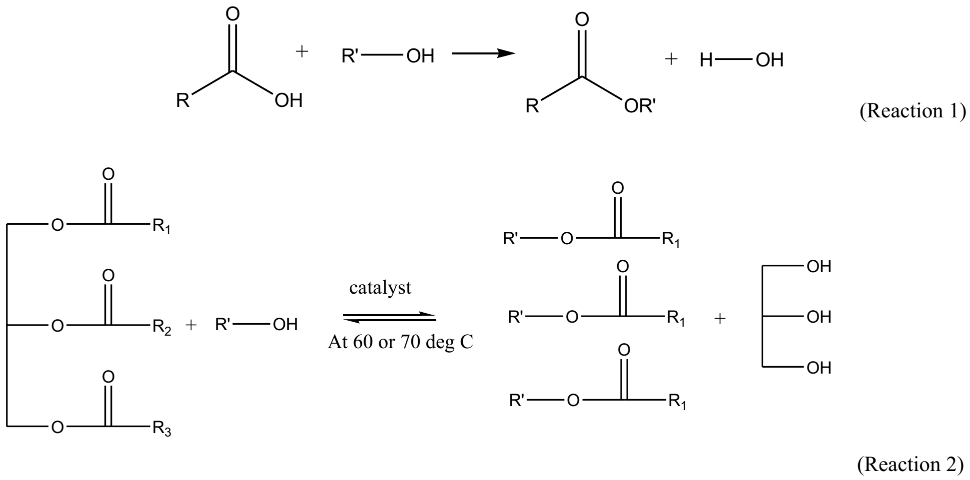
Lower stability, higher cloud point, and the need for purification of remaining metals in the pipeline are just some of the limitations of fatty acid methyl esters. Glisic et al. (2016) examined the techno-economic feasibility and feasibility of producing 100,000 tonnes of hydrotreated vegetable oil per year via non-catalytic supercritical transesterification of hydrotreated vegetable oil from waste cooking oil. Hydrogenation was conducted at 390 °C and 138 bar with a fixed hydrogen-to-waste cooking oil ratio of 1068 Nm^3/m^3, while supercritical transesterification was conducted at 20 MPa and 300 °C. As the process relies heavily on feedstock prices, it was deemed uneconomical to produce hydrotreated vegetable oil alone. This hydrotreated vegetable oil production method was only deemed feasible if the production capacity reached 200,000 tonnes/year and it was installed near a petroleum refinery, where the required infrastructure is already in place (Glisic et al. 2016). Similarly, another study reported that the current capacity of the supercritical transesterification method was less profitable and depends on glycerol prices in the future (Andreo-Martínez et al. 2020).

Biodiesel: production, properties, and challenges

Biodiesel production generally consists of esterification and transesterification. Esterification is a reaction that occurs between alcohol and carboxylic acid to produce an ester and water (Fig. 5, Reaction 1). Methanol and ethanol are the most used alcoholic compounds due to their easier miscibility, long carboxylic chains, and low cost. Meanwhile, glycerolysis is a potential pre-treatment, especially for oils with high free fatty acid content (Elgharbawy et al. 2021). During glycerolysis, a triglyceride reacts with methanol and converts to fatty acid methyl esters and glycerol (Fig. 5, Reaction 2) (Moraes et al. 2020). The catalyst that is used in the transesterification process can either be homogeneous or heterogeneous and acidic or alkaline in nature (Cong et al. 2020; Zhang et al. 2018). Biodiesel feedstock can also be varied such as sugarcane lipids (Arora and Singh 2020) and grease trap waste (Tran et al. 2018), to name a few.

A biocatalyst is a suitable alternative for solvent-free biodiesel production. Lee et al. (2019a, b) used a biocatalyst to co-produce glycerol-free biodiesel as well as optimize the condition. Under optimal conditions, the biocatalyst was able to convert 70% of the glycerol and 80% of the biodiesel. However, although this process eliminated the complexity of product separation and purification, a reaction time of 10 h was required to reach the optimal condition (Lee et al. 2019b). Meanwhile, Andrade et al. (2019) performed an in-depth analysis and compared the efficacy of using a liquid enzyme or an immobilized enzyme in the transesterification of castor oil. The reactor of the first approach required the use of several vessels for the process, distillation, decanting, as a heat exchanger, and for storage as well as 0.3–10

Fig. 5 Typical reaction in biodiesel production



wt.% of the enzyme, while the second approach required the addition of separation vessels to facilitate enzyme recovery. Both approaches were simulated to produce 250,000 tons per year. The first approach was found to yield a profit of US$52 million/year at a production cost of US$0.80/kg, while the immobilized enzyme had to be reused at least 300 times in the second approach to yield the same amount of profit as the first approach (Andrade et al. 2019). However, Elgharbawy et al. (2021) simultaneously conducted esterification and transesterification with lipase, which was an interesting method of further scaling up (Elgharbawy et al. 2021).

The ever-increasing use of biodiesel fuels is still faced with several problems. Some of the obstacles to biodiesel commercialization include production cost, post-production management, engine warranty, poor cold flow properties, and storage stability (Jafari et al. 2019). As the cost of biodiesel production hinges upon oil feedstock prices, cheap feedstocks need to be identified to increase the feasibility of commercialization. It is also a challenge to collect feedstock in specific places and comprehensively analyze the cost of pre-treatment. Issues with post-production management include wastewater treatment and glycerol by-product management. For instance, as biodiesel wastewater comprises organic compounds such as methanol, oil residue, triglycerides, glycerol, and its derivatives, proper wastewater management is required. Furthermore, as 10% by volume of glycerol by-product is produced during the transesterification of triglyceride, integrated glycerol valorization increased the profit of this method of biodiesel production. Although the use of biodiesel fuels has reduced greenhouse gas emissions, it still requires some regulations to improve efficiency. For instance, the company needs to treat palm oil mill effluents post-production as it emits other toxic substances such as methane. Deforestation should also be avoided to preserve the original carbon content of the trees (Szulczyk and Atiqur Rahman Khan 2018). Table 1 simplifies the technical limitations of existing biodiesel fuels. As several extant studies have examined methods of overcoming the abovementioned limitations, this present review only focused on overcoming the issues of poor cold flow properties and storage stability in biodiesel commercialization.

Effect of fatty acid methyl ester composition and blending level

Vegetable oils comprise different fatty acid compositions, types, and saturated fatty acid to non-saturated fatty acid ratios (Jain and Sharma 2010). Table 2 provides the fatty acid profiles of different feedstocks. The properties of biodiesel fuel are highly dependent on the fatty acid profile of the vegetable oil. For instance, unsaturated fatty acid chains are more susceptible to free radical attacks, which then react with other fatty acid chains (Pantoja et al. 2013). This reactive site is not directly affected by the number of double bond chains, but by the number of bis-allylic methylene groups adjacent to the double bond or cis–trans configuration. Furthermore, although allylic methylene positions are significantly less reactive than bis-allylic positions, they are just as susceptible to free radical attacks. Moreover, polyunsaturated methyl esters and cis-configuration are more susceptible to free radical attacks.

Tang et al. (2008) investigated the effect of fatty acid profile on biodiesel stability by examining vegetable oils and animal fats such as soybean, cottonseed, palm, yellow grease, selected white grease, and poultry fat (Tang et al. 2008). The study found that fatty acid composition affects oxidative stability and the amounts of antioxidants required to meet biodiesel standards. Meanwhile, another study used kinematic viscosity, acid value, peroxide value, and induction time to observe the effect of the fatty acid composition.

Table 1 Problems and solutions for biodiesel fuel utilization

Problem	Solution	Ref
Deposit on injector affected to fuel spray pattern	The injector should be cleaned by qualified mechanics with specialized training and instrument the injector	Liaquat et al. (2014)
Lubrication oil becomes diluted	The lubrication oil needs to be changed faster than fossil fuel with regular check	Liu et al. (2019b)
Longer to turn on the engine, especially in a cold climate	(1) Install a pre-heater to warm up the fuel tank and filter (2) Blend with kerosene or add additive to improve cold-weather properties	Kumar and Singh (2020)
Fuel leaking from its line	The materials in the engine should be changed to flour-based elastomers such as Viton or Teflon	Chandran et al. (2017)
Clogs in the fuel filter	The filter needed to be changed before using biodiesel. Then, if the filter clogs again, add an additive or filling fuel at the end of the usage on that day	Cardeño et al. (2020)
High capital investment	Some promising non-edible feedstocks should receive more concern with analysis of their fluctuating prices	Gebremariam and Marchetti (2018)

Table 2 Fatty acid profile of several oil feedstocks

Fatty acid	Vegetable oil source				
	Palm	Soybean	Rapeseed	Jatropha	Sunflower
Capric ($C_{10:0}$)	0.48	-	0.56	-	-
Lauric ($C_{12:0}$)	-	0.09	0.09	0.09	0.10
Myristic ($C_{14:0}$)	1.05	0.09	-	0.28	0.10
Palmitic ($C_{16:0}$)	41.59	11.07	4.04	14.36	6.31
Palmitoleic ($C_{16:1}$)	0.19	0.18	0.09	0.93	0.10
Stearic ($C_{18:0}$)	3.6	3.72	2.1	5.88	3.55
Oleic ($C_{18:1}$)	40.41	22.61	57.26	38.94	21.39
Linoleic ($C_{18:2}$)	9.3	51.33	20.69	34.89	63.35
Linolenic ($C_{18:3}$)	0.29	5.63	8.08	0.28	1.46
Arachidic ($C_{20:0}$)	0.29	0.27	0.37	0.19	0.29
Eicosenoic ($C_{20:1}$)	0.1	0.27	2.02	0.09	0.19
Behenic ($C_{22:0}$)	0.1	0.27	0.28	0.19	0.58
Erucic ($C_{22:1}$)	-	0.09	0.46	0.09	0.10
Total saturated	48.85	15.70	7.06	23.58	11.22
Total unsaturated	50.28	80.39	88.80	75.31	88.69
Ref	Aung et al. (2018)	Prabakaran et al. (2018)	Bocianowski et al. (2012)	Jonas et al. (2020)	Akkaya (2018)

The plant commodities studied were rapeseed, soybean, and flaxseed, which mainly contained oleic acid (C18:1), linoleic acid (C18:2), and linolenic acid (C18:3), respectively. The study found that fatty acid methyl esters from highly unsaturated fatty acid raw materials such as flaxseed tend to oxidize faster. Increasing the temperature was also found to reduce the induction time as it was difficult to remove the polymeric substances that formed due to peroxide radical polymerization (Yamane et al. 2007).

Hydrogenation or isomerization to the cis–trans configuration can be used to reduce the number of unsaturated bonds in biodiesel fuel. Liu et al. (2019a, b) used a p-toluene sulfonic acid catalyst to successfully produce the cis–trans configuration in various edible oil feedstocks such as safflower, camellia, sunflower, and soybean. The higher percentage of unsaturated fatty acid than saturated hydrocarbon chain in the bio-oil feedstock affected the final biodiesel stability. Heating the mixture to 100 °C for 30 m under nitrogen flow at atmospheric pressure was found to yield a conversion of almost 80% without changing the position of the double bond. The mixture was then diluted with alkali to neutralize the excess acid before it was treated with diethyl ether. Furthermore, the addition of 0–20% water and less than 200 ppm of antioxidants did not affect the nature of the biodiesel. Therefore, this is a promising method for improving biodiesel stability (Liu et al. 2019a).

A systematic study recently examined the effect of the fatty acid methyl ester composition of selected edible oils, non-edible oils, low molecular weight oils, and animal fat feedstocks on cold filter plugging points. The fatty acid methyl ester sources were mixed and studied using the statistical regression method. Upon using several of the proposed modeling equations, the study concluded that long-chain saturated fatty acid methyl esters significantly affected the cold filter plugging point. Apart from that, C16:0, C18:0, and C20:0 content were responsible for the production of biodiesel fuels that were within the requirements of the cold filter plugging point (Yuan et al. 2017).

Another study developed three correlations to calculate the cold filter plugging point of biodiesel fuels based on their fatty acid profiles. The study developed a model derived from pure biodiesel fuels as well as blends containing 24 admixtures of feedstocks such as palm, canola, yellow grease, and soybeans. Although the established models yielded good results during the pre-validation stage with fewer experimental data, the results deviated significantly when the samples contained high amounts of monoacylglycerols. This is because monoacylglycerols are soluble in biodiesel; however, they formed solid residues that are problematic in engines as it clogs the filters. Therefore, monoacylglycerols have to be removed to improve biodiesel stability (Dunn 2020).

Oxidation stability is an important parameter for biodiesel commercialization. Oxidation in biodiesel occurs through three stages of auto-oxidation that formed peroxides, then the peroxides decomposed to aldehydes, dimer, acids, or other polymer derivatives (Hazrat et al. 2021), as presented in Fig. 6. Biodiesel oxidation generally initiated with the metal-catalyzed decomposition of hyperoxides. The oxidation in biodiesel causes several impacts on its properties. The affected properties were acidity, calorific, and induction period. Oxidation increases biodiesel acidity due to

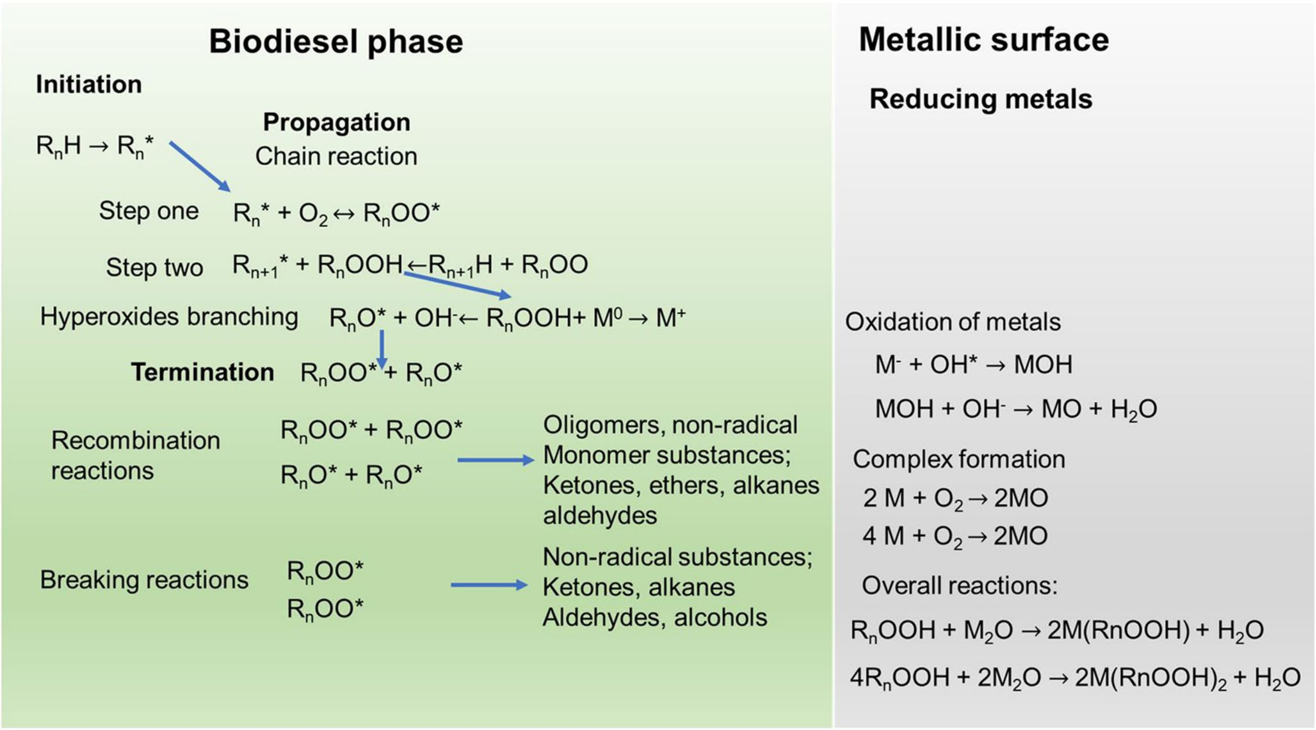


Fig. 6 Detail mechanism of biodiesel oxidation (adapted from (EN 2012) with permission from Elsevier)

the formation of carboxylate, aldehyde, and ketone. Calorific value (CV) has a linear correlation with combustion efficiency. Oxidation increases the amount of heteroatom in the fatty acid chain, consequently decreasing biodiesel efficiency. Meanwhile, the induction period (IP) is a direct measurement of biodiesel stability. Hence, the longer IP indicated the longer stability of the fatty acid chain with minimum IP of 6 h according to EN14112 (Longanesi et al. 2022).

The stability of a biodiesel fuel can also be prolonged by blending with petroleum diesel. The B40 jatropha oil biodiesel meets the American Society for Testing and Materials (ASTM) D7467 standards, while B30 provides a balance between cost and oxidative stability. The B5 jatropha oil biodiesel did not show any signs of oxidation despite three years of storage compared to biodiesel that had been treated with 100 ppm of tert-butylhydroquinone while the B20 jatropha oil biodiesel also exhibited showed similar properties when the feedstock had an induction time of six hours. However, there are few references for higher proportions of biodiesel blends (McCormick et al. 2007).

Blending with conventional diesel has also been found to accelerate deposit formation. For instance, a pure biodiesel fuel was found to generate only 0.9 mg/100 mL of deposits, while a B20 blend generated 17 mg/100 mL (Fang and McCormick 2006). Another study comprehensively examined the effect of biodiesel blends from various feedstocks. Three types of biodiesels were blended with four types of conventional diesel that have a low sulfur content to create the following: sample B-1 which contained 65% canola oil and 35% waste cooking oil; B-2 which contained 36% waste cooking oil, 20% palm oil, and 44% soybean oil; and B-3 which contained 32% waste cooking oil, 32% soybean oil, 36% canola oil, and four other blends with an additive. The compositions of these biodiesel blends were categorized according to the ratio of total saturated to unsaturated fractions. The minimum induction time for biodiesel blends with diesel was 20 h, based on the margins of the EN ISO 4259 methodology. When blended with 5–10% biodiesel, the properties of the B-1 sample were still below the margin, while that of the B-2 and B-3 samples were above the margin. The low properties of the B-1 sample were due to the high content of unsaturated carbon chains in the rapeseed oil and the short organic chains in the waste cooking oil, which tends to oxidize and form aldehydes and ketones. Furthermore, fatty acid composition affected not only biodiesel stability but also to several other parameters that will be exhaustively clarified in the subsequent section part (Karavalakis et al. 2010). Meanwhile, Li et al. (2019) investigated the effect of biodiesel blends, specifically, B0, B5, B20, and B50, on thermal degradation and reported that the thermal degradation of samples that contained less than 20% of aldehyde and ester was insignificant compared to that of samples containing more than 20% (Li et al. 2019).

Biodiesel stability can also be improved via blending with higher-stability fatty acid methyl esters. For instance, one study added a 60% palm oil fatty acid methyl ester to jatropha oil to meet the required standards (Sarin et al. 2007), while another study that added a 20% of crambe oil fatty acid methyl ester reported a 44% decreased in the viscosity of soybean oil (Wazilewski et al. 2013). Meanwhile, binary blends of sativa oil methyl ester and pork lard methyl ester have exhibited excellent stability after 19 months of storage (Lebedevas et al. 2013). Pölczmann et al. (2016) blended 10%, 30%, and 50% of waste cooking oil biodiesel with gas oil to determine which combination provided the best characteristics. Although the 10% sample yielded the best condition, it was still not recommended to store it for more than a year without a stabilizer. The study also suggested the use of floating roof tanks for storage to minimize contact with air (Pölczmann et al. 2016).

Removal of monoacylglycerols

Biodiesel is a fatty acid methyl ester that is derived from triacylglycerols and methanol. A minimum of three moles of methanol are required to completely convert triacylglycerols to fatty acid methyl esters (Reaction 2). The conversion of triacylglycerol to glycerol occurs via two intermediates, namely diacylglycerols and monoacylglycerols. This reaction forms two phases consisting of biodiesel (non-polar) and glycerol (polar). However, diacylglycerols and monoacylglycerols are only slightly soluble in fatty acid methyl esters, which can be mixed with the desired biodiesel. Unfortunately, monoacylglycerols can lead to several problems such as solid precipitate formation, suspension of bubbles, and emulsions in the fuel. Additionally, monoacylglycerols also affect the cloud point, which causes engine problems due to filter clogging (Dunn 2012). Therefore, monoacylglycerols must be removed to enhance biodiesel fuels.

The quantification and limits of monoacylglycerol content in biodiesel were in accordance with ASTM D6751-10 standards. However, as it only focuses on $C_{16\text{-}C18}$ monoglycerides, it is limited to certain oil feedstocks. Therefore, advances in chromatographic instruments and techniques provide avenues for increasing the detection limit to a broader range. For instance, Alleman et al. (2019) produced biodiesel using non-conventional feedstock. However, they received some retention time errors when using the existing ASTM standard. They, therefore, developed a calibration standard that could observe the retention times of individual monoacylglycerols that could be present in the feedstock. This provided a more depth analysis to predict major monoacylglycerol components. Furthermore, the data accuracy of the developed calibration standard provided better accuracy than the existing standard (Alleman et al. 2019).

Another study comprehensively examined the effect of monoacylglycerols on the cold flow properties of fatty acid methyl esters. This study confirmed the significant role of monoacylglycerols in cloud point as the addition of 1 wt.% of monoacylglycerol increased the cloud point by 13 ° C while the addition of the same amount of monoolein only increased the cloud point by 0.094 ° C (Dunn 2012).

Meanwhile, Padhi et al. (2012) investigated if transesterification with lipase could effectively remove monoacylglycerols from biodiesel fuel. The study used a combination of glycerol mono-palmitate and glycerol mono-stearate as a monoacylglycerol sample. As such, 12 mg of lipase G, a partial glyceride-eliminating enzyme produced by *Penicillium camembertii*, was added to the monoacylglycerol sample and stirred continuously for 24 h at 50 ° C. More methanol was then added during the reaction to ensure that enough methanol was available. As 90% of the monoacylglycerols were successfully converted to fatty acid methyl esters, it indicated the high performance of the lipase. Furthermore, the amount of fatty acid methyl esters did not change significantly after the addition of methanol in the middle of the reaction, indicating that an adequate amount of methanol has been present during the initial reaction. However, a setback of the enzymatic transesterification method was its high cost, which could be lowered by binding to support to immobilize the enzyme (Padhi et al. 2012). Lee et al. (2019a, b) posit that the surface of the support would require modification when used with certain functional groups to prevent activity loss. As such, activated carbon was used as a support that functionalized with glutaraldehyde to form a covalent bond between amine groups and enzymes. This approach enabled to maintain 80% activity after 20 cycles during fatty acid methyl ester production from various feedstocks (Lee et al. 2019a). However, the enzymatic catalyst for biodiesel production still has several drawbacks such as a slower reaction rate and the high cost of the enzyme. Additionally, the enzyme that is immobilized to support also requires complex preparation and low yield (Kalita et al. 2022).

Oxidation stability of fatty acid methyl esters

Biodiesel stability is one of the major issues limiting the biofuel consumption of various continents. This is because fatty acid methyl esters are more prone to oxidation which changes the physicochemical and tribological properties of fatty acid methyl esters than conventional diesel. To better elucidate the oxidation process, Alves-Fortunato et al. (2020) oxidized rapeseed and soy biodiesel in an autoclave reactor and then evaluated the functional groups, viscosity, and density of the biodiesel sample. Oxidation was found to form two phases that have a tendency of forming a complex oil-oil emulsion.

The supernatant and deposit phase were then separated and studied using Fourier transform infrared spectroscopy, which revealed that polarity influenced the separation rate. Therefore, the solid deposit was not only induced by surface contact with the fuel but also stimulated in the fatty acid methyl ester liquid phase (Alves-Fortunato et al. 2020). Figure 7 presents the biodiesel oxidation process.

Another study conducted a more comprehensive analysis of biodiesel degradation pathways using Fourier transform infrared spectroscopy, nuclear magnetic resonance, and thermogravimetric analysis to monitor the mechanism of biodiesel oxidation. The study found that oxidation occurred due to reverse transesterification or methyl ester decomposition. Furthermore, impurities such as glycerine derivatives (mono-, diglyceride, or glycerine) may react with the methyl ester and decrease the amount of fatty acid methyl esters. Meanwhile, fatty acid methyl ester decomposition may change to a per-ester basis after reacting with the hyperoxides. Peroxides or hyperoxides are the end products of a series of reactions via the radical route. These oxygen-based radicals reacted and formed various carbon compounds such as alcohols, aldehydes, ketones, or carboxylic acids. Carboxylic acids and alcohols decreased the total acidity and flash point, respectively. Dimer and trimer acids formed when carboxylic acid reacted with hyperoxides. This leads to the formation of deposits that cause injector failures and filter blocking (Fang and McCormick 2006).

According to the European EN 15,751 standards and the ASTM D6751 biodiesel fuel standard, the induction period can be used to deduce oxidation stability (Knothe 2007). Therefore, the oxidation stability of the biofuel was measured using an induction period at a minimum of 6 h using Rancimat or PetroOXY oxidation stability measurements. Meanwhile, another study used the oil stability index to monitor oxidation stability (Knothe and Dunn 2003). Biodiesel oxidation can be caused by a reaction with oxygen, photo-oxidation, or reverse transesterification. As such, some of the factors affecting oxidation as well as methods of slowing oxidation are presented below.

Temperature

Several studies have correlated oxidation with temperature. One study concluded that increasing the temperature decreased the fatty acid methyl ester stability of various oil feedstocks such as olive, corn, peanut, soybean, lear, and safflower. This indicates that temperature altered the fatty acid methyl ester properties of oil regardless of its fatty acid composition and content (Hasenhuettl and Wan 1992). Another study also corroborates that temperature changed the properties of fatty acid methyl esters and their by-products (Dunn 2008). However, changes in fatty acid methyl ester products can also be caused by the generation of hydroperoxides, which leads to radical routes that expedite oxidation (Yamane et al. 2007).

Jain and Sharma (2012) established a correlation between thermal stability and biodiesel oxidation using activation energy (Ea) and induction period (IP) as parameters for thermal stability and oxidation, respectively. The study found that decreasing the thermal stability decreased the activation energy, which indirectly increased the induction period. Therefore, the correlation between these two parameters is linear, with a good correlation coefficient. Interestingly, the study also found that the correlation depends on the type of metal as well (Jain and Sharma 2012). This corroborates the findings of another study which found that copper most adversely affects biodiesel fuel characteristics (Sarin et al. 2010b).

Another study also investigated the correlation between thermal stability and metal contaminants using pyrogallol,

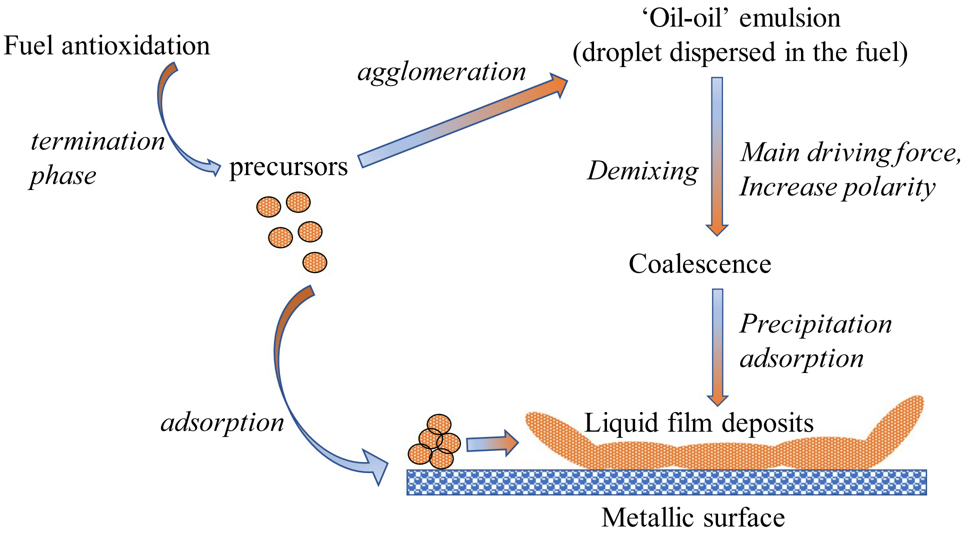


Fig. 7 Mechanism of biodiesel oxidation (adapted from (Alves-Fortunato et al. 2020) with permission from Elsevier)

which a previous study found was the optimum antioxidant for various metals of different concentrations (Jain and Sharma 2011). The established correlation was found to depend on the type of metal contaminant. Equation (1) presents an example of the correlation:

$$\text{IP or Ea} = a\ (M)^{b}\ c\ (A)^{-d} \tag{1}$$

where a, b, c, and d are constant from linear regression of experimental data, while M and A are the metal content (ppm) and the amount of antioxidant (ppm), respectively. This equation can be used to predict the amount of antioxidants required for metal-contaminated biodiesel. The same method also confirmed the correlation between metal contaminants and the oxidation stability as well as metal contaminants and the storage stability of jatropha oil biodiesel (Jain and Sharma 2013).

Light

Although biodiesel fuel is commonly stored in closed containers, light can still penetrate the chamber at times. The effect of light penetration on palm olein was elucidated using Fourier transformation infrared spectroscopy, and the oxidation rate was indirectly monitored by measuring the peroxide, acid, and iodine value. This found that light reduced antioxidant performance and slightly increased the acid value. It has been well established also that light can have a pro-oxidizing effect on fatty derivatives. Therefore, it was not surprising that some antioxidants were rendered less effective when stored in daylight (Almeida et al. 2019).

Apart from exposure to light, open biodiesel containers are also in constant contact with fresh air. This promotes oxidation during storage. Christensen and McCormick (2014) compared the properties of waste cooking oil and sunflower oil-based biodiesel fuel that had been stored in both open and closed containers for 30 months. After six months of storage, the peroxide and acid levels of the exposed biodiesel containers were significantly different from that of the closed biodiesel containers. However, the viscosity of both containers only differed slightly, indicating that peroxide cracking occurred but did not cause oligomerization. Interestingly, the oxygen was in radical form, which significantly accelerated oxidation (Christensen and McCormick 2014). The radical oxygen was formed via photo-reaction, which is 30,000 times faster than autoxidation in oleic acid and 1500 faster than linoleic acid methyl ester (Knothe 2007).

Water

Fatty acid methyl esters are a type of ester that is usually produced after a carboxylate acid and alcohol reaction. As this type of process is an equilibrium reaction, fatty acid methyl ester hydrolysis can be used to reverse the reaction. For instance, Fang and McCormick (2006) were able to shorten the induction period by 25% by adding 100 ppm of water (Fang and McCormick 2006). Therefore, the reverse reaction caused fatty acid methyl ester degradation and oxidation to occur. Another study also found that water reduces the amount of fatty acid methyl ester biodiesel, especially that produced with a calcium oxide catalyst. The yield was decreased by 9% after 180 days of storage (Dias and Ramos 2021).

Metal content and antioxidant

Metals, either from the oil itself, the production method, or the storage tank, have been found to negatively impact the stability of biodiesel fuels, which reduces the induction period. Sarin et al. (2010a, b) evaluated metals, such as iron, manganese, nickel, cobalt, and copper, and found that the presence of metals accelerated free radical oxidation and that copper was the most detrimental metal. Furthermore, high and low metal content were found to yield similar oxidation stability. Therefore, metal destruction occurs even when the metal content is low. However, the oxidation stability of fatty acid methyl esters that contain metals increased with the addition of antioxidants. A minimum of 250 ppm of antioxidants was required to extend oxidation stability from 2.54 to 6 h in fatty acid methyl esters that had been contaminated with 2 ppm of metal (Sarin et al. 2010b).

Another study examined the effect of metals such as iron, copper, cobalt, nickel, and manganese on manketti oil methyl ester. During the experiment, a specific amount of metal was placed inside plastic bottles containing manketti oil methyl ester, and the oxidation stability was monitored once a month for the six-month period. The study found that copper and iron had the most and least detrimental effects on stability, respectively. In the case of iron-contaminated manketti oil methyl ester, 500 ppm of pyrogallol was required to yield biodiesel with an oxidation stability of 6.2 after six months. This was higher than the standards outlined in the ASTM D6751 (3 h) and the South African National Standard (SANS) 1935 (6 h) (Kivevele 2020).

Meanwhile, another study examined the effect of aluminum on a B20 cotton seed oil biodiesel and reported a 10% reduction in brake thermal efficiency and a 15% increase in brake-specific fuel consumption compared to conventional diesel. Furthermore, the presence of metal also increased the nitrogen oxide pollution of biodiesel by 16% (Puniyani et al. 2019). A comprehensive study of the effect that several transition metals and their metal oxides on biodiesel stability concluded that copper causes the fastest oxidation, while molybdenum and rhenium slower the oxidation process. The molybdenum metal oxides were also found to inhibit oxidation, while that of gold, chromium, rhenium, and copper accelerated oxidation (Knothe and Steidley 2018).

According to Fernandes et al. (2019), carbon steel is a potential biodiesel storage vessel. The study stored *Moringa oleifera Lam* biodiesel under conditions of room temperature and the absence of light. Two types of carbon steel were investigated, namely, CS1015 (99.48% iron, 0.13–0.18% carbon) and CS4140 (96.78–97.77% iron, 0.38–0.43% carbon). This study concluded that CS4140 was more suitable for biodiesel storage as it prevents iron from dissolving into the biofuel. Therefore, carbon is able to slow the oxidation process. Nevertheless, a tert-butylhydroquinone antioxidant still had to be added to the *Moringa oleifera Lam* biodiesel to meet the European EN 14,112 standards (Fernandes et al. 2019). According to Zhao et al. (2014), metal contaminants can originate from the soil that a biofuel source was planted in. The study planted sunflowers in urban marginal land, which contains an excessive amount of permitted heavy metals, to produce biodiesel. The sunflower plantation showed positive energy gains, especially when the community contributed to harvesting; 5–15 people are required to harvest 0.2 ha of marginal land, which could be an alternative option for food and urban farming (Zhao et al. 2014).

Biodiesel contains small amounts of organic molecules that act as natural antioxidants. Antioxidants slower biodiesel oxidation by generating radical species that react with hydrocarbons to form a stable intermediary and disrupt the destruction route (Miyata et al. 2004). Alcohol formation is favored over aldehyde or acid formation as it reduces polymeric deposition. The natural antioxidants found in biodiesel originated from the vitamin E of vegetable oil, which comprises tocopherols. Tocopherols prolonged the induction period of several types of fatty acid methyl esters. Furthermore, natural antioxidants perform better than synthetic antioxidants (Donoso et al. 2020).

Generally, antioxidants acted as hyperoxides decomposers, chain breakers, or free radical terminators. Sarin et al. (2010a, b) investigated the stability of *Pongamia pinnata* oil biodiesel. *Pongamia pinnata* oil mainly consists of 72% oleic acid ($C_{18:1}$) and 16% saturated fatty acid. The induction period of the produced fatty acid methyl esters was less than 3 h, which was still lower than the worldwide standard. Therefore, the natural antioxidant was incapable of slowing oxidation. However, the induction period increased to more than 6 h when tert-butylated hydroxytoluene was used. Therefore, tert-butylated hydroxytoluene can be used for phenol inhibition for biodiesel stability (Sarin et al. 2010b).

Antioxidants such as tert-butylated hydroxytoluene, tert-butylhydroquinone, tert-butylphenolic derivatives, as well as octylated and butylated diphenylamine were investigated in the presence of metal. The study found that tert-butylhydroquinone was the best antioxidant as it plays the crucial role of hydroxyl groups to slow biodiesel oxidation. Furthermore, the amount of antioxidants required was influenced by the type and concentration of metals in the fatty acid methyl esters (Sarin et al. 2010a). Another study concluded that 1,2,3 tri-hydroxy benzene and 3,4,5-tri-hydroxy benzoic acid (gallic acid) were the best antioxidants to slow the oxidation of karanja, neem, and jatropha oil biodiesels. This indicates that the phenol site is the crucial functional group in antioxidants (Agarwal et al. 2015).

Comin et al. (2017) added two to three types of antioxidants to a soybean oil biodiesel and investigated the efficacy of using tert-butylhydroquinone, alizarin, and citric acid to stabilize the B100. Even a small amount of the combined antioxidant was found to yield better performance than a single antioxidant, even at the highest concentration. Therefore, this may be a method of reducing the antioxidant cost, which was obtained via a synergy of several antioxidants (Comin et al. 2017).

Devi et al. (2018) examined the use of 100–250 ppm of a potato peel antioxidant in *Mesua ferrea* L oil biodiesel. The study found that a minimum concentration of 150 rpm was required to yield an induction period of up to 6.21 h. A comparison indicated that the performance of the potato peel antioxidant was comparable to that of a synthetic antioxidant such as tert-butylhydroquinone. Therefore, greener compounds are powerful antioxidants (Devi et al. 2018). Meanwhile, another study examined to replace synthetic antioxidants with *Thuja oreantalis* L leaf extract in waste cooking oil biodiesel. Characterization using nuclear magnetic resonance, Fourier transform infrared spectroscopy, and high-performance liquid chromatography confirmed that the extract contained flavonoid and phenolic compounds, which slow lipid oxidation. Furthermore, a minimum of 100 ppm of the extract was required to yield a biodiesel that met both the European and American standards with an induction period of 6.79 h (Devi et al. 2019). Table 3 provides a summary of antioxidants that could potentially prolong the oxidation stability of biodiesel fuels.

Antioxidants could also be originated from amine structures rather than phenolic compounds. The aromatic amine was responsible for free radical inhibition (Varatharajan and Cheralathan 2013). Since direct monitoring of radical formation and inhibition was challenging, they observed hydrocarbon and heteroatom content in exhaust emission using DPPD (N,N′-diphenyl-1,4- phenylenediamine) and NPPD (N-phenyl-1,4-phenylenediamine). NPPD was identified as the best antioxidant due to its lower NO pollution with B20 and B100 of soybean oil.

Cold flow improvers

Another obstacle in the commercialization of biodiesel fuels is the longer period required to start an engine, especially in the winter. This is because the fuel in the tank

Table 3 Potential antioxidants for biodiesel fuels

Oil feedstock	Antioxidant	Concentration (ppm)	FAME properties	Remark	Ref
Pongamia pinnata	tert-butylated hydroxytoluene (BHT)	250	IP: 6.2 h	Tocopherol: < 50 ppm	Sarin et al. (2010b)
Pongamia pinnata	tert-butylated phenol (TBP)	250	IP: 5.3 h	Tocopherol: < 50 ppm	Sarin et al. (2010b)
Refined palm oil (RPO)	tert-butyl hydroxquinone (TBHQ)	150	IP: 6.6 h	FAME has a high tendency for copper corrosion	Sarin et al. (2010a)
Soybean	Alizarin (ALZ), TBHQ, citric acid (CA)	50, 100, 50	IP: 13.7 h	The optimum composition reduced 70% of the final cost	Comin et al. (2017)
Mesua ferrea L	Potato peel	150	IP: 6.21 h	Comparable performance to TBHQ	Devi et al. (2018)
Waste cooking oil	*Thuja oreantalis* L	100	IF: 6.79 h	Rich in phenolic compound	Devi et al. (2019)
Soybean	N,N′-diphenyl-1,4-phenylenediamine (DPPD)	750	NO reduces by 9%	Amine attach to aromatic ring influenced the exhaust pollution	Varatharajan and Cheralathan (2013)

IP, induction period

requires a longer period to warm up, thereby decelerating fuel mobility. Cloud point, pour point, and cold filter plugging point are three characteristics in biodiesel standards that correlate with flow characteristics. Cloud point (ASTM D2500) and pour point (ASTM D97) are the lowest temperature tested, where the oil demonstrates fluid behavior (pours), not the temperature where it loses flowability. Meanwhile, the cold filter plugging point (ASTM D6371) relates to the filter limit required to work efficiently and can be defined as the lowest temperature at which 20 mL of a sample can flow through a 45 mμ wire mesh filter within 60 s. Studies rarely provide a phase diagram of the multiple components in biodiesel. An interesting observation can be conducted using Newtonian heating and a computation analysis (Evcil et al. 2018). The three characteristics of cold flow properties were plotted in one graph, instead of separate graphs, with the use of a computer. The solid fraction was also estimated using the significant increase of solid fraction at the cold filter plugging point. Therefore, this method is a promising tool to monitor and understand the biodiesel solidification process and develop methods of minimizing the formation of solid fractions. Cavalheiro et al. (2020) conducted an exhaustive investigation of biodiesel solid fractions and concluded that the amount of solid residue did not correlate linearly with oxidation stability. Therefore, the oxidation process did not predominantly produce solid precipitates. The study also revealed agglomeration of monoglycerides and free glycerine. Furthermore, low temperatures, near to non-polar sites, and storage time were identified as the primary accelerators of agglomeration (Cavalheiro et al. 2020). Table 4 summarizes the three types of cold flow improvers.

Polymers

Extant studies recommend blending with fossil fuels, winterization, or cold flow improvers to improve the cold flow properties of biodiesel fuels. Cold flow improvers, which function by delaying crystallization at certain stages for a certain period, are the most popular choice that could be classified as pour point depressants or cloud point depressants. Mohanan et al. (2017) added a polymeric pour point depressant and vegetable oil crystallization modifier to soybean oil fatty acid methyl ester. The pour point depressant was poly(lauryl methacrylate), while the vegetable oil crystallization modifier was a vegetable oil derivative, triacylglycerol. These two additives lowered the pour point to 31 ° C by slowing nucleation and agglomeration. Meanwhile, the vegetable oil crystallization modifier inhibited nucleation and formed crystal arrangements that selectively adsorbed the pour point depressant. This reduced agglomeration, thereby lowering the pour point significantly. The study concluded that the amount and structure of the vegetable oil crystallization modifier plays a crucial role as all the fatty acid methyl esters were attracted to big molecules with varied structural orientations. This type of depressant also has the potential for commercialization and created a new method of controlling the use of specific additives to prevent crystallization (Mohanan et al. 2017). Meanwhile, another study that combined Span™ 80, a commercial pour point depressant, with polymethacrylate concluded that the

Table 4 Cold flow improvers in biodiesel

Feedstock	Additive	Amount	Decreased by (° C)	Ref
Soybean	Poly Lauryl Methacrylate (PLMA) and triacylglycerol (TAG)	0.5% PLMA and 7% TAG	CFPP: 30	Mohanan et al. (2017)
Waste cooking oil	span80 and polymethacrylate (PMA)	• 0.5% • Mass ratio: 2: 1	CFPP: 7	Xue et al. (2017)
B5 from soybean	Poly (tetradecyl acrylate)	1000 ppm	CFPP: 29	Muniz-Wypych et al. (2016)
B20 from soybean	Poly (tetradecyl acrylate)	1000 ppm	CFPP: 18	Muniz-Wypych et al. (2016)
Waste cooking oil	Methyl acetoacqetate	20% vol	CFPP: 5	Lian et al. (2017)
Biodiesel blends	Triacetin	20% vol	CFPP: 2–10	Elias et al. (2016)
Palm oil	Ethanol	20% vol	CP: 13.7 PP: 15.6	Verma et al. (2016)
Soybean oil	Poly (lactic acid) oligomer (OLLA)	10% vol	CP: 6 PP: 2	Tesfaye and Katiyar (2016)
Soybean	Ethyl lactate	5% vol	CP: 6 PP: 2	Tesfaye and Katiyar (2016)
Waste cooking oil (B50)	Ethyl acetoacetate (EAA) and iso-octyl methacrylate (IOMA)	2.5% vol EAA and 10% vol IOMA	CFPP: 11 PP: 12	Zhao et al. (2016)
Milkskum	Acetone or diethyl ether	20% vol	PP: 7 CP: 12 CFPP: 13	Srikanth et al. (2019)
Palm oil (B20)	Poly (dodecyl acrylate-co-tetradecyl methacrylate)	1000 ppm	PP: 7	Muniz et al. (2019)

PMTAG, cross-link palm oil triacylglycerol

optimum additive amount was 0.5%m with a 2:1 mass ratio. The additives were also found to aid the formation of small crystals, thereby increasing the mobility of the biodiesel fuel (Xue et al. 2017).

Meanwhile, another study evaluated the ability of methyl acetoacetate concentrations of 0–20% as a fluid flow improver in waste cooking oil biodiesel (Muniz-Wypych et al. 2016). Twenty percent was determined to be the optimum methyl acetoacetate concentration as three crucial cold flow parameters, cold filter plugging point, pour point, and cloud point, decreased by 5 ° C, 5 ° C, and 7 ° C, respectively. Furthermore, the low melting point of the methyl acetoacetate (– 80 ° C) inhibited crystal growth, thereby lowering the temperature. This was confirmed using differential scanning calorimetry and polarity measurements (Lian et al. 2017).

A series of polymer additives based on methyl tetraethyl ester-maleic anhydride has also been evaluated as potential cold flow improvers. Five hundred ppm was determined to be the optimum dose as it lowered the cold filter plugging point by 8 ° C. Therefore, molecular structure and wax crystal interactions are important to obtain optimum depressant performance. These interactions were further enhanced as the chain structure of the cold flow improver was similar and exhibited similar polarity to that of the biodiesel (Zhou et al. 2016). Apart from that, Muniz et al. (2019) used poly(dodecyl acrylate-co-tetradecyl methacrylate) as a cold flow improver as it maximizes interactions with biodiesel and conventional diesel (Muniz et al. 2019). It comprised homopolymers and copolymers that were longer than 14 carbon atoms and small molecules that acted as spacers in the long hydrocarbon chain. One thousand ppm of poly(dodecyl acrylate-co-tetradecyl methacrylate) was found to lower the pour point of B5 and B20 palm oil biodiesel to – 35 ° C and – 14 ° C, respectively. The structural configuration of the polymer was also found to play a crucial role, as some of the copolymer compounds did not perform well. However, the best polymer had a negligible effect on B100 and, therefore, required further enhancement.

Meanwhile, another study evaluated the ability of a biodegradable polymer and its derivative, namely, poly(lactic acid) oligomer and ethyl lactate, as depressants and enhance the flow properties of soybean oil biodiesel. Although the cold flow improvers did not change the kinematic viscosity and heating properties of the soybean oil biodiesel, the addition of 3 wt.% of ethyl lactate improved its pour point and cloud point by 3 °C and 6 °C, respectively. Meanwhile, the addition of poly(lactic acid) oligomer was similar to that of ethyl lactate, indicating the crucial role of the chemical backbone. Furthermore, increasing crystal nucleation at low temperatures enhanced the flow characteristics of the soybean oil biodiesel (Tesfaye and Katiyar 2016).

Surfactants

Surfactants are another potential cold flow improver as it displays hydrophobic and hydrophilic properties. Madihalli et al. (2016) used mannosylerythritol lipid to improve the flow characteristics of refinery-grade diesel, consumer-grade diesel, and biodiesel. Surfactants decreased the cloud points of all three fuels. More specifically, the cloud point of biodiesel decreased by 3.2 ° C. The proposed mechanism was developed by observing the thermal behavior of the biodiesel fuels and fuel with surfactant. The study posits that the interactions mainly occurred with the major components of the fatty acid in the fatty acid methyl esters, which began with physical interactions and were reversible in nature (Madihalli et al. 2016).

Meanwhile, Zhao et al. (2016) investigated the ability of cold flow improvers such as iso-decyl methacrylate, ethyl acetoacetate, and iso-octyl methacrylate to improve the cold flow properties of B10 to B50 waste cooking oil biodiesel blends. A combination of ethyl acetoacetate and iso-octyl methacrylate was the optimum cold flow improver. This was caused by the synergistic behavior of the lowest freezing points of the two cold flow improvers, i.e., – 45 ° C for the ethyl acetoacetate and – 50 ° C for the iso-octyl methacrylate. The ethyl acetoacetate-iso-octyl methacrylate cold flow improver was also found to reduce crystal aggregation and preferred to form small crystals at low temperatures, thereby decreasing the cold filter plugging point. However, the addition of the cold flow improver only changed the characteristics of the biodiesel slightly. It is noteworthy that a ternary mixture of the cold flow improver did not have a synergistic effect in decreasing the cold filter plugging point (Zhao et al. 2016).

To monitor the crystallization process, another study used differential scanning calorimetry to observe and compare the low-temperature behavior of a biofuel that had been treated with a side product-based cold flow improver and other commercial cold flow improvers. The side product-based cold flow improver could act as a cold flow improver and provide economic benefits that were comparable to that of commercial cold flow improvers. Furthermore, the addition of 5% of the side product-based cold flow improver improved the cold filter plugging point of the biofuel by 2 ° C. However, higher amounts of the side product-based cold flow improver significantly changed the characteristics of the biofuel. More specifically, it lowered the heating value of the biofuel (Lapuerta et al. 2019). Meanwhile, another study used X-ray diffraction analysis, differential scanning calorimetry, and polarized light microscopy to examine the crystallization behavior of methyl palmitate that had been blended with a 1,3-dioleoyl-2-palmitoyl glycerol additive. The addition of 1,3-dioleoyl-2-palmitoyl glycerol was found to interrupt the nucleation and crystal growth of methyl palmitate by altering its crystal structure, microstructure, and polymorphism (Mohanan et al. 2015).

Small molecules

Some studies have evaluated the cold flow properties of biodiesel blends with triacetin as a cold flow improver. The biodiesel blends comprised saturated-saturated and saturated–unsaturated carbon chains. The study produced fatty acid methyl esters by reacting a triglyceride with methyl acetate, which produced triacetin as a by-product. The overall cloud point of the first blend was the component with the lowest cloud point, as it was easier to form a solution than crystallize. However, when the biodiesel blends comprised carbon chains of similar numbers, the total cloud point varied significantly from the component with the lowest cloud point. Interestingly, the overall cloud point of the second blend was that of the component with the highest cloud point. Therefore, triacetin could be used as an additive at various weight loadings as it has a similar structure to fatty acid methyl esters, which is also influenced by the type of biodiesel blend (Elias et al. 2016).

Verma et al. (2016) reported that the addition of fossil fuel improved the cold flow properties of biodiesel. The study found that blending 80% of fossil fuel with B20 biodiesel increased the cloud point and pour point of the B20 biodiesel by 60% and 71%, respectively. However, blending with conventional diesel did not enhance biofuel. Meanwhile, the addition of 20% ethanol, a potential cold flow improver that can be produced from biomass, to palm oil biodiesel lowered its pour point and cloud point from 21 ° C and 19.7 ° C to 7.3 ° C and 4.1 ° C, respectively (Verma et al. 2016). Another study found that adding a large amount of branched-chain fatty acid diluent only decreased the cloud point and poured point of biodiesel from several feedstocks by 2 ° C (Dunn et al. 2019).

Biodiesel can also be derived from industrial waste such as daily washed milk scum. One study collected the discharged water during milk washing and created biodiesel. The biodiesel was then treated with cold flow improvers such as acetone and diethyl ether. The study found that acetone and diethyl ether behaved similarly, and the cold flow properties of the milk scum biodiesel were similar after the addition of either cold flow improver. For instance, the pour point, cloud point, and cold filter plugging point of the milk scum biodiesel decreased by 7 °C, 12 °C, and 13 ° C, respectively. However, as the lowest freezing point of the acetone (– 95 ° C) was lower than that of the biodiesel, it lowered the initial coagulation temperature. Furthermore, the characteristic of the milk scum biodiesel in the presence of a cold flow improver was still within the acceptable range of the biodiesel standard. The milk scum biodiesel was also treated with ethyl acetoacetate and ethyl levulinate. However, the decrease in cold flow properties was still less than that of the acetone due to their higher freezing points (Srikanth et al. 2017). Meanwhile, another study reported that the addition of 20% of ethyl levulinate to a canola oil biodiesel decreased its cloud point to − 7 ° C or by 17 ° C, indicating that it could be used in cold climates. Therefore, the low freezing point of the ethyl levulinate significantly improved the cold flow properties of the canola oil biodiesel.

Janakiraman et al. (2020) examined the efficacy of adding 25 ppm of cerium oxide, zirconium oxide, and titanium oxide via ultrasonication to a B20 *Garcinia* seed biodiesel (Janakiraman et al. 2020). Tests with a Kirloskar TAF-1 single-cylinder engine revealed that the additives improved brake thermal efficiency and lowered ignition delay. Similarly, another study reported that these nano additives were able to increase heat transfer, reduce pollution, and increase the oxidative stability of the fuel blend (Soudagar et al. 2018).

Conclusion and outlook

Many countries have expressed a significant interest in fatty acid methyl esters or biodiesel. However, the stability of biodiesel is still in doubt and requires further improvements. This review addressed the several parameters affecting biodiesel stability in detail. Isomerization is a potential method that can be used to reduce the number of unsaturated carbon chains in the fatty acid profile of a feedstock. Meanwhile, transesterification with an enzyme can be used to remove monoacylglycerol. As several metals speed up lipid oxidation, stainless steel, aluminum, or carbon steel storage containers could be used to increase stable storage duration. Natural antioxidants that are rich in phenolic or flavonoid compounds are promising substitutes for synthetic antioxidants. Meanwhile, cold flow improvers are required to improve the cold flow behavior of biodiesel fuels. Cold flow improvers such as polymers, surfactants, and small molecules are preferred over substances with low melting points. As most of the existing pipelines and vessels contain many impurities that adversely affect biodiesel stability, adding a combination of two antioxidants is more promising than one antioxidant with double the concentration.

Despite the significant progress in biodiesel production and stability improvement, there are some issues for further commercialization of biodiesel. The existing biodiesel production is still dominated by local fish plantations. Therefore, future studies should examine other potential feedstocks. Furthermore, as biodiesel demand is forecasted to increase, seasonal crops such as corn and peanuts should be investigated as a short-term plan. Next-generation farming, where plants can be grown with minimum space, more advanced sensing, and automation to fertilize and water them, needs to be developed in many countries. Tamanu (*Calophyllum inophyllum*) and *Reutealis trisperma* are promising feedstocks in next-generation farming as they provide more yield with lower investment. For the long term, biorefineries should be established to produce fuel and chemicals by preparing more advanced industrial processes. Additionally, since it is still a challenge to stably store B40 and B50 fatty acid methyl esters, there is an urgent need to combine deoxygenated vegetable oil and fatty acid methyl esters. Finally, to lower the investment, producing green diesel at existing petroleum refineries is a promising process for the future.

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