A Review on the Recent Breakthrough Methods and Influential Parameters in the Biodiesel Synthesis and Purification

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Abstract. Biodiesel has recently received much attention as an energy source with numerous benefits such as high degradability, negligible toxicity, and minimal emissions of carbon monoxide gases as well as particulates. Therefore, this research aims to compare, review, and summarize the conventional and advanced methods of biodiesel production. Currently, some emerging processes that were developed for advanced biodiesel production include microwave-assisted synthesis, ultrasonic-assisted synthesis, supercritical transesterification, and liquid phase plasma discharge technology. The types of feedstocks, catalysts, and operating conditions as the influential parameters in biodiesel synthesis are also discussed. Moreover, in the purification process, the effectiveness of purification depends on the type of catalyst applied in the synthesis process. This research also reviewed and compared several commonly used purification methods such as wet and dry washing, ion exchange and precipitation, complexation, and membrane-based separation that have shown significant results along with the impacts of biodiesel production on environmental and economic sectors.

Keywords: Biodiesel synthesis, biodiesel purification, catalysts, advanced biodiesel production, biodiesel impact

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1. Introduction

Biodiesel has been widely promoted one of the most promising renewable energy sources. Basically, biodiesel is a blend of fatty acid and methyl esters (FAME) that can be used in standard diesel engines with little modification. It has mostly been created through the esterification of free fatty acids (FFAs) and/or homogenous basic or acid transesterification of triacylglycerols (TAGs) from diverse raw materials such as used cooking oils, plant, and animal oils (Athar and Zaidi, 2020). The TAGs conversion into methyl esters takes approximately one hour at ambient temperature and pressure conditions due to the high activity of conventional basic catalysts and the moderate operating condition of reaction (around 60–65°C) (Athar and Zaidi, 2020; Fayyazi et al., 2021; Syafuddin et al., 2020). However, the disadvantages of these procedures include the generation of soap, a decrease in catalytic effectiveness due to catalyst depletion, a rise in viscosity, and the development of gels.

In the wet washing process, a large amount of water is required to remove the remaining catalyst and purify the biodiesel, leading to a substantial amount of wastewater that needs to be effectively handled (Fayyazi et al., 2021; Šalić et al., 2020; Sokač et al., 2020). This extra stage in the synthesis of the biodiesel process raises the total cost of production, which makes it uncompetitive with petroleum-based diesel generation (Kumar et al., 2020; Noriega and Narváez, 2020). Lastly, the cost of producing biodiesel is approximately three times higher than that of petroleum. Moreover, one of the most significant disadvantages of homogeneous catalysts is that they cannot be regenerated (Hariprasath et al., 2019; Kasirajan, 2021; Shankar et al., 2017).

Previous research has stated that the conventional biodiesel production methods such as catalytic transesterification and esterification have achieved production high capacity, however, it requires high expenditure, especially for the purification process (Veljković et al., 2015). This becomes the main reason biodiesel has not been able to compete with diesel oil from petroleum products in the transportation market (Keera et al., 2018; Lee and Saka, 2010; Marchetti et al., 2007). Therefore, the need for the development of novel, clean, eco-friendly, and efficient processes is increasing in past decades to produce the standard biodiesel product with a shorter time and easier purification method. To solve this problem, numerous approaches of biodiesel production from the conventional to the advanced methods such as microwave-assisted synthesis, ultrasonic-assisted ultrasonic, supercritical transesterification, and plasma discharge were deeply reviewed, compared, and discussed. This research also reviews the influential parameters in biodiesel syntheses such as feedstocks, catalysts, and
operating conditions. Furthermore, several commonly applied purification methods were discussed and summarized to present a holistic understanding of biodiesel production. The methods presented were wet and dry washing, ion exchange and precipitation, complexation, membrane-based separation, and simultaneous synthesis and purification techniques along with their benefits and drawbacks. Similarly, the impacts of biodiesel production on environmental and socio-economic aspects were also briefly analyzed.

**Fig. 1.** Overview of the review’s contents.

**Transesterification reaction**

\[
\begin{align*}
&\text{Triglycerides (oil)} \\
&\text{Alcohol} \\
&\text{Biodiesel (esters)} \\
&\text{Glycerol}
\end{align*}
\]

**Esterification reaction**

\[
\begin{align*}
&\text{Free fatty acid (FFA)} \\
&\text{Alcohol} \\
&\text{Biodiesel (esters)} \\
&\text{Water}
\end{align*}
\]

**Fig. 2.** The transesterification and esterification reactions in biodiesel production (Chozhavendhan et al., 2020).
2. Production process of biodiesel

In this section, the conventional and several advanced methods in biodiesel production are discussed, as shown in Fig. 1. Conventional methods from the literature include homogeneous and heterogeneous processes, while advanced methods such as microwave-assisted, ultrasonic-assisted, plasma discharge, and supercritical processes in biodiesel production are reviewed and compared.

2.1. Conventional method of biodiesel production

Algae, plant oil, food sources, lignocellulosic materials, and other sources can be used to produce biodiesel (Abo et al., 2019; Alami et al., 2021; Baskar et al., 2018; Keera et al., 2018; Li et al., 2012; Saputra Nursal et al., 2021). Moreover, biodiesel is commonly made through the transesterification process, which involves reacting oils with short-chain alcohol to form alkyl esters and glycerol with the use of a catalyst. The reaction can be occurred at a temperature of (50-70 °C), and it is usually heated by external thermal heater (Fayyazi et al., 2021). Transesterification is a three-step process that occurs in a sequential (Al-Saadi et al., 2020; Hariprasath et al., 2019; Sokaē et al., 2020), which involves the transformation of triglyceride in the oil to diglyceride, followed by monoglyceride, and glycerol in the first stage. A significant amount of oil to alcohol (usually 1:3) molar percentage is constantly operated in the system to promote the forward reaction and a large amount of alcohol is typically applied to tip the balance to the right side (product). The reaction flow is divided into two stages, namely biodiesel product (upper) and glycerol rich phase (lower) at the end of the transesterification procedures (Fayyazi et al., 2021). It was also reported that the unreacted alcohol is distributed in both phases (Al-Saadi et al., 2020; Hariprasath et al., 2019; Sokaē et al., 2020). The general reaction mechanisms for producing biodiesel (methyl esters) through the transesterification and esterification process can be seen in Fig. 2. Further details regarding homogeneous and heterogeneous catalytic process in biodiesel synthesis can be seen in part 3.2.1 and 3.2.2, respectively.

2.2. Advanced method of biodiesel production

In conventional methods, energy in form of thermal is used to heat the reactants to a specific temperature to force the chemical reaction. Meanwhile, the activation energy is fulfilled using electric, sound, and electromagnetic wave powers in the current methods of biodiesel production. In this section, the current technology of biodiesel production is discussed.

2.2.1. Microwave-assisted biodiesel synthesis

In common biodiesel production, the reactors are designed based on conventional heat transfer, which transfers some amount of energy to the material’s surface and initiates the reaction. However, this method is thermodynamically inefficient and time-consuming, because it requires a large amount of energy and longer time to achieve a uniform temperature distribution (Lawan et al., 2020). Meanwhile, microwave technology can irradiate the electromagnetic energy directly to the molecular level for early chemical reaction (Mamo and Mekonnen, 2020). Research has shown that microwave energy transfer is faster and more efficient than conventional heating technology (Binnal et al., 2021; Mamo and Mekonnen, 2020). In the practical situation, it is essential to identify the dissipation factor of the material that is used in a microwave system because its efficiency of heat transfer depends on the ability of the material to absorb the electromagnetic energy and initiate the chemical reaction. Therefore, homogeneous and heterogeneous catalysts are still used in microwave-assisted biodiesel synthesis to provide higher efficiency (Tangy et al., 2017). In the synthesis process, the microwave energy is directly transferred to the reactants and degraded into different side products. This makes it necessary to maintain the input power of microwave irradiation to achieve a good result.

Microwave-assisted transesterification process using can be applied using homogeneous and heterogeneous catalysts. Homogeneous acid catalyst process is the best choice for the feedstock that containing high level of free fatty acid and water (Zhang et al., 2010; Zhang, 2003). Several homogeneous acid catalysts including sulfuric acid (H2SO4), hydrochloric acid (HCl), and boron trifluoride (BF3) were found useful for the biodiesel synthesis from high acid level feedstocks (Dall’Oglio et al., 2015; Zhang et al., 2010). Other polyacids such as H3PW12O40, H2SiW12O40, and Na4PW12O40 can be dissolved in alcohol, forming the homogeneous system (Zhang et al., 2010). The polyacids have strong Bronsted acidity than that of H2SO4 and other acids with uniform acid sites. A study conducted by Dall’Oglio and coworkers revealed the results of comparative study on microwave-assisted biodiesel synthesis using maize oil as feedstock and applied some acid catalysts such as HCl, H2SO4, and CISO4H was found to have > 85% of conversion (Dall’Oglio et al., 2015). The better conversion using H2SO4 showed the better mobility of ions and counterions in the reaction system that supported by microwaves compared to the conventional system (Dall’Oglio et al., 2015; Zhang et al., 2010). Cheng and coworkers studied the comparation of microwave-assisted biodiesel synthesis of chlorella pyrenoidosa using two different processes namely, (i) microwave-assisted transesterification followed by a conventional solvent extraction, and (ii) microwave-assisted extraction followed by a conventional transesterification (Cheng et al., 2014). It was found that in the first approach the FAME concentration was found to have a maximum content of 95% by using 30 min of 500 W microwave power (Cheng et al., 2014). Other process conditions were 6 mL/g alcohol to oil ratio, 3% H2SO4, and 90 °C of temperature. It was concluded that microwave was useful to boost the FAME yields (Cheng et al., 2014). Since the microwave does not interrupt the bonds at molecular levels, but just helping to cause the electron excitation, thus, the reaction pathways of a microwave-assisted biodiesel synthesis follow the same route as the conventional process (Dehghan et al., 2019; Huang et al., 2015). Despite acid catalysts showed improvements in microwave-assisted biodiesel production, some drawbacks were still found such as it requires higher alcohol to oil ratio, formation of water molecules, complex product separation and purification, and the spent acids in final product (Dehghan et al., 2019).

Due to many drawbacks of acid catalysts in the homogeneous microwave-assisted system, researchers gave more attention to the alkali catalysts (Nayak et al., 2021).
The main profit of using homogeneous alkali catalyst in microwave-assisted biodiesel synthesis is a higher yield of biodiesel in a shorter time compared to the conventional heating method (Qu et al., 2021). This is promoted by the direct absorption of electromagnetic energy of microwave by the hydroxyl (-OH) groups, that subsequently break the structure of two-tier of oil and alcohol, which improved the solubility of these reactants (Lin and Chen, 2017; Sharma et al., 2016). Numerous alkali group I materials such as KOH, NaOH, NaOCH3, which are dissolved in alcohol and can be applied for microwave-assisted biodiesel synthesis (Nayak et al., 2019). Under bombardment of electromagnetic power from microwaves, due to the higher dielectric characteristics, alcohol quickly heated to reach the boiling point and provide sufficient energy to initiate the transesterification reaction (Rokni et al., 2022). A study performed a microwave-assisted biodiesel synthesis using papaya oil found nearly 99% biodiesel conversion (Nayak and Vyas, 2019). This process was conducted by applying 0.95%-wt NaOH as catalyst, 62.3 °C of temperature, 9:5:1 alcohol to oil ratio, 450 W of microwave power for 3.5 min (Nayak and Vyas, 2019). An attempt was performed using microalgae as feed oil with 0.5%-wt NaOH in methanol as catalysts, 12 ml n-hexane, constant agitation of 600 rpm, 45 °C, with 350 W of microwave power (Chen et al., 2015). In addition, the main advantage of microwave-assisted biodiesel synthesis using the homogeneous alkali catalysts is the faster process compared to the acid catalysts and can be performed for wet biomass feedstocks such as microalgae by a single stage (Chen et al., 2015; Qu et al., 2021).

Microwave-assisted biodiesel synthesis using acid and alkali homogeneous catalysts reported to have an improvement in shorter reaction time, but the homogeneous catalytic process suffers from problems regarding product separation process, treatment of acid or base disposals, and catalyst reactivation process (Kamel Ariffin and Idris, 2022; Zhang et al., 2022). This problem can be solved by using heterogeneous catalysts (Delghan et al., 2019). Various study on the microwave-assisted transesterification using heterogeneous catalysts have been reported (Alcañiz-Monge et al., 2018; de Aguiar et al., 2017). Acidic metal oxides, alkali metal oxides, cation exchange resins, clay supported catalysts, and carbon supported catalysts have been utilized for advanced microwave-assisted biodiesel synthesis (Alcañiz-Monge et al., 2018; de Aguiar et al., 2017; Guldhe et al., 2017; Nayak et al., 2019). The presence of catalysts can reduce the required microwave energy level, maintain the reaction equilibrium, and conduct the reaction at lower input energy input that provides a fast conversion rate (Nayak et al., 2019). The operating condition in microwave-assisted biodiesel synthesis depends on the feedstock properties, fatty acid content, reaction time, mixing speed, and catalyst dosage (Mamo and Mekonnen, 2020; Tangy et al., 2017). Some of these parameters have been optimized in the previous research (Thirugnanasambandham et al., 2017). Table 1 summarizes the most recent application of microwave in biodiesel synthesis, where it was concluded that microwave-assisted biodiesel production technology is one of the most promising new technologies with faster and high conversion. However, some obstacles such as high capital cost and complex equipment requirement decelerate the development of this method in the industry.

2.2.2. Ultrasonic assisted biodiesel synthesis

Another modern technology in biodiesel production is ultrasonic-assisted biodiesel synthesis, which is carried out to homogenize the reactants and catalysts. The higher level of homogenization between reactant and catalyst can provide a better contact area to convert reactants into biodiesel with a higher conversion rate (Salamatinia et al., 2012). The use of ultrasonic radiations in transesterification reaction does not disturb the chemical equilibrium and thermodynamic conditions (Sajjadi et al., 2015; Stavarache et al., 2007). Similarly, it can also enhance the mass transfer, thereby improving the overall rate of kinetic (Sajjadi et al., 2015). This process radiates the acoustic energy, generates the cavitation mechanism and increases the molecular kinetic energy (Gogate, 2008). To apply the ultrasonication in biodiesel production, there is a need to understand the required intensity, produce the optimized cavitation, and distribute a uniform ultrasound condition across the mixture to obtain a better result. In an ultrasonic system, the electrical energy is converted into acoustic/ sound energy through piezoelectric transformers, which are emitted into the chemical transformation (Luo et al., 2014). Therefore, an optimized design is needed to minimize the energy loss during the conversion from electrical into sound energy.

The physical effects of ultrasonic-assisted transesterification reaction have been reported by a huge number of investigations. Physically, the ultrasonic waves cause bubble cavitations close to the boundary layer between oil and alcohol, which generating a vast number of micro bubbles. The bubbles’ collapse produces microturbulence and disrupts the phase boundary layer. The speed of collapse of the bubbles can reach up to 200 m/s, generating the immiscible mixing and the emulsification between the oil and alcohol. The mass transfer rate increases due to this emulsification as well as generating faster reaction kinetics. The use of an ultrasonic system in the transesterification process enhances the reaction efficiency and attributes to the less ratio of oil to alcohol (Florez Marulanda and Ortega Alegria, 2019). Meanwhile, one of the superiorities of the ultrasonic system in biodiesel production is the massive formation of oil and alcohol emulsion, which is carried out by simple mixing in the conventional method. The recent developments of ultrasonic-assisted biodiesel production are listed in Table 2. In the homogeneous catalytic process, the ultrasonic energy does not directly enhance the reaction rate; however, the ultrasonic waves intensify the mass transfer by providing massive emulsion of reactants and catalysts to achieve an 80% faster process. Moreover, it can also be seen that the operating temperatures to conduct the biodiesel synthesis using an ultrasonic system are relatively low compared to the conventional process that needs at least 60°C to initiate the reaction (Asakura et al., 2008).

It is noteworthy that, even though transesterification assisted with ultrasonic wave to synthesize biodiesel is very helpful, further studies are required to overcome the technical drawbacks.
## Table 1.
Comparison of several microwave-assisted biodiesel productions.

| Feedstock       | Type of catalytic process | Catalyst          | Operating conditions | Ester yield (%) | Ref.                                      |
|-----------------|----------------------------|-------------------|----------------------|-----------------|-------------------------------------------|
| Waste cooking oil | Homogeneous                | NaOH (1%)         | P = 700 W, t = 10 min, T = 60°C | 88.9            | (Eguchi, 2015)                           |
| Microalgae oil   | Homogeneous                | H₂SO₄ (2.5%)      | P = 450 W, t = 11 min, T = 60°C | 97.1            | (Binnal and Nirguna Babu, 2019)           |
| Dairy scum oil   | Homogeneous                | KOH (1%)          | P = 600 W, t = 5 min, T = 60°C | 93.5            | (Binnal et al., 2021)                     |
| Waste cooking oil | Homogeneous                | KOH (1%)          | P = 750 W, t = 2 min, T = 80°C | 96              | (Sawangkeaw et al., 2011)                 |
| Maize oil        | Homogeneous                | CISO₂H (3%)       | P = 700 W, t = 20 min, T = 80-84°C | 93.1            | (Dall'Oglio et al., 2015)                |
| Chlorella vulgaris oil | Homogeneous            | H₂SO₄ (1.5%)      | P = 700 W, t = 40 min, T = 60°C | 91.8            | (Sharma et al., 2016)                     |
| Yellow horn oil  | Homogeneous                | H₂SO₄ (1%)        | P = 500 W, t = 30 min, T = 90°C | 95.2            | (Zhang et al., 2010)                      |
| Yellow horn oil  | Homogeneous                | H₂PW₂O₁₄ (1%)     | P = 500 W, t = 10 min, T = 90°C | 96.2            | (Zhang et al., 2010)                      |
| Algae oil        | Homogeneous                | NaOH (0.5%)       | P = 800 W, t = 20 min, T = 50°C | 83.3            | (Chee Loong and Idris, 2014)              |
| Waste cooking oil| Homogeneous                | CH₃ONa (0.75%)    | P = 750 W, t = 3 min, T = 65°C | 97.9            | (Chen et al., 2012)                       |
| Tallow           | Homogeneous                | NaOH (0.6%)       | P = 700 W, t = 5 min, T = 55°C | 96.3            | (Murillo et al., 2019)                    |
| Jathropa oil     | Homogeneous                | NaOH (1%)         | P = 312 W, t = 7 min, T = 65°C | 98.7            | (Nayak et al., 2019)                      |
| Safflower oil    | Homogeneous                | NaOH (1%)         | P = 300 W, t = 6 min, T = 60°C | 98.4            | (Duz et al., 2011)                        |
| Waste cooking oil| Heterogeneous              | SrOSIO₄ (41%)     | P = 100 W, t = 15 min, T = 60°C | 99.2            | (Tangy et al., 2017)                      |
| Yellow horn oil  | Heterogeneous              | Ag₃PW₁₂O₄₀ (1%)   | P = 500 W, t = 10 min, T = 60°C | 95.7            | (Zhang et al., 2010)                      |
| Castor oil       | Heterogeneous              | Cs₂H₂O₃PW₁₂O₄₀ (15%)| P = 300 W, t = 4 h, T = 70°C | 90.0            | (Yuan and Shu, 2013)                     |
| Acidified oil    | Heterogeneous              | CERP/PES (3%)     | P = 360 W, t = 90 min, T = 60°C | 97.4            | (Zhang et al., 2012)                      |
| Waste oil        | Heterogeneous              | Amberlyst-15 (25%)| P = 360 W, t = 30 min, T = 75°C | 85.5            | (Ayas and Yilmaz, 2015)                   |
| Rapesseed oil    | Heterogeneous              | K₁₀montmorillonite| P = 1000 W, t = 60 min, T = 170°C | 10.2            | (Mazzochia et al., 2004)                  |
| Oleic acid       | Heterogeneous              | Sulphated metakaolin (5%) | P = 400 W, t = 40 min, T = 115°C | 96.5            | (de Oliveira et al., 2013)                |
| Jathropa oil     | Heterogeneous              | [MMBIM]HSO₄ (20%) | P = 245 W, t = 12 min, T = 80°C | 96.2            | (Soni et al., 2014)                       |
| Microalgae oil   | Heterogeneous              | Goat bone nano-catalyst | P = 600 W, t = 3h, T = 60°C | 92              | (Abomohra et al., 2017)                   |
| Waste lard       | Heterogeneous              | CaO/zeolite (8%)  | P = 595 W, t = 1.25 h, T = 70°C | 90.9            | (Lawan et al., 2020)                      |
| Feedstock                | Type of catalytic process | Catalyst           | Operating conditions          | Ester yield (%) | Ref.                                           |
|-------------------------|----------------------------|--------------------|--------------------------------|-----------------|------------------------------------------------|
| Waste cooking oil       | Homogeneous                | KOH (1%)           | P = 240 W, f = 20 kHz, t = 8 min, T = 60 °C | 88.4            | (Martinez-Guerra and Gude, 2015)               |
| Canola oil              | Homogeneous                | NaOH (1.25%)       | P = 150 W, f = 20 kHz, t = 2 min, T = 50 °C | 96.8            | (Fayyazi et al., 2014)                        |
| Karanja oil             | Homogeneous                | NaOH (1%)          | P = 500 W, f = 20 kHz, t = 45 min, T = 50 °C | 98              | (Parida et al., 2016)                        |
| Waste cooking oil       | Homogeneous                | NaOH (1%)          | P = 500 W, f = 20 kHz, t = 1 min, T = 55 °C | 99              | (Khosravi et al., 2016)                       |
| Waste cooking oil       | Homogeneous                | KOH (1%)           | P = 500 W, f = 20 kHz, t = 1 min, T = 50 °C | 99              | (Khosravi et al., 2016)                       |
| S. triguga oil          | Homogeneous                | Ba(OH)\(_2\) (3%) | P = 250 W, f = 20 kHz, t = 80 min, T = 50 °C | 90.8            | (Sarve et al., 2016)                         |
| Waste cooking oil       | Homogeneous                | KOH (1%)           | P = 400 W, f = 25 kHz, t = 10 min, T = 60 °C | 96.5            | (Aghbashlo et al., 2018)                      |
| R. trisperma oil        | Homogeneous                | H\(_2\)SO\(_4\) (2%) | P = 1000 W, f = 40 kHz, t = 150 min, T = 55 °C | 95.3            | (Wongwuttanasatian and Jookjantra, 2020)      |
| Castor oil              | Homogeneous                | KOH (3%)           | P = 560 W, f = 20 kHz, t = 6 min, T = 42 °C | 97              | (Florez Marulanda and Ortega Alegria, 2019)   |
| Palm oil                | Heterogeneous              | CaO (8%)           | P = 140 W, f = 68 kHz, t = 37 min, T = 50 °C | 96.2            | (Aghbashlo et al., 2018)                      |
| Rapeseed oil            | Heterogeneous              | Li/Fe\(_2\)O\(_3\) | P = 1000 W, f = 37 kHz, t = 35 min, T = 35 °C | 99.8            | (Fallah Kelarijani et al., 2020)              |
| Waste cooking oil       | Heterogeneous              | CaO (1.5%)         | P = 1000 W, f = 20 kHz, t = 60 min, T = 70 °C | 98              | (Aghbashlo et al., 2018)                      |
| Waste cooking oil       | Heterogeneous              | CaDG (1%)          | P = 120 W, f = 22 kHz, t = 30 min, T = 60 °C | 93.5            | (Gupta et al., 2015)                         |
| Karabi oil              | Heterogeneous              | CaO (5%)           | P = 50 W, f = 20-30 kHz, t = 120 min, T = 60 °C | 94.1            | (Yadav et al., 2018)                         |
| Jatropha oil            | Heterogeneous              | K\(_2\)PO\(_4\) (1%) | P = 600 W, f = 20 kHz, t = 45 min, T = 50 °C | 98              | (Jogi et al., 2016)                          |
| Jatropha oil            | Heterogeneous              | Sr-D-CaO (6%)      | P = 210 W, f = 20 kHz, t = 30 min, T = 65 °C | 95.4            | (Ali et al., 2017)                           |
| Jatropha oil            | Heterogeneous              | TPA/AC (4.23%)     | P = 400 W, f = 20 kHz, t = 40 min, T = 65 °C | 91              | (Badday et al., 2013a)                       |
| Waste cooking oil       | Heterogeneous              | SO\(_3\)-H-CD (1.5%) | P = 300 W, f = 20 kHz, t = 8.8 min, T = 117 °C | 90.8            | (Badday et al., 2013b)                       |
| Oleic acid              | Heterogeneous              | PTA-MOF (30%)      | P = 100 W, f = 20 kHz, t = 15 min, T = 30 °C | 98              | (Nikseresht et al., 2017)                     |
| Kernel oil              | Enzymatic                  | Lipase (3%)        | P = 140 W, f = 25 kHz, t = 4h, T = 25 °C | 55.2            | (Murillo et al., 2019)                       |
| Waste cooking oil       | Enzymatic                  | Lipozyme TLIM (3%) | P = 80 W, f = 25 kHz, t = 3h, T = 25 °C | 86.1            | (Subbedar and Oogate, 2016)                   |
| Crambe oil              | Enzymatic                  | Novozym® 435 (20%) | P = 80 W, f = 20 kHz, t = 6h, T = 30 °C | 98.2            | (Tavares et al., 2017)                       |
Yasvanthrajan and coworkers conducted an ultrasonic assisted transesterification method to boost the process efficiency of biodiesel production from waste cottonseed oil using the immobilized lipase enzyme as the catalyst (Yasvanthrajan et al., 2021). This approach effectively decreased the time for reaction to 6 h with low amount of enzyme (5 %w) and relatively milder temperature condition (45 °C). Moreover, the immobilized lipase remains intact during this ultrasonic assisted transesterification process and has a great reusability up to four consecutive cycles. Therefore, this emerging approach can be adapted to provide the more efficient biodiesel production process. Another study by Oliveira and co-workers reported that the removal of external heating source and mechanical agitation is beneficial for ultrasonic assisted transesterification process (Oliveira et al., 2021).

2.2.3. Supercritical transesterification

The supercritical transesterification process is one of the modern methods in biodiesel production, which is carried out without the presence of catalysts. This process allows direct transesterification of triglycerides and esterification of free fatty acid into biodiesel (Farobie and Matsumura, 2017). The supercritical process also improves the solubility and mass transfer properties of the reaction. In this process, the reactants containing oil and alcohol are transferred into a reactor with supercritical operating conditions. The temperature and pressure conditions are above the critical point of the alcohol, with methanol having a critical temperature and pressure of 240 °C and 1140 psi, respectively (Deshpande et al., 2017). In this supercritical condition, the chemical and physical properties of methanol change such as density, diffusivity, polarity, and viscosity (Bernal et al., 2012). Previous research has shown that some influential parameters in supercritical transesterification include pressure, temperature, type of alcohol, reaction time, and oil to alcohol ratio (Deshpande et al., 2017; Farobie and Matsumura, 2017). According to the previous research on biodiesel production using supercritical transesterification, it was discovered that in the supercritical region, methanol disintegrated to a free monomer due to the weakening of hydrogen bonding (Lee and Saka, 2010). The free methanol monomer reacts with carbonyl groups of the triglyceride that generates methoxide transfer and form the methyl ester and diglyceride that further transform into biodiesel and glycerol.

The supercritical transesterification was firstly studied by Lee and Saka and they found that the thermal decomposition of biodiesel (ester) may occur at high temperature condition and ambient pressure, which resulting in lower biodiesel quality (Lee and Saka, 2010). However, at high temperature and pressure condition in supercritical process, the transesterification reaction is easier to occur due to the formation of free monomer of the reactants. Yin and coworkers investigated a biodiesel synthesis using soybean oil in supercritical condition in a high pressurized vessel (Yin et al., 2008). They found that the ester conversion was 95% at 10 min of process at a temperature of 350 °C, whereas at 260 °C, it was only 30% of ester conversion after 60 min. Another study by Tsai and coworkers reported that waste cooking oil showed better efficiency compared to the refined cooking oil due to its free fatty acid content (Tsai et al., 2013). The ester yield was 65% at 300 °C and 100 bar within 4 min.

Even though the supercritical process seems to generate many benefits, including, no pretreatment needed, catalysts free, no soap formation, wastewaster free, and faster process. However, the high expensive operational cost is a significant limitation due to the supercritical process that requires high temperature and pressure (Han et al., 2005; Kusdiana and Saka, 2004; Yin et al., 2008). Some simulation, environmental life cycle assessment, and feasibility studies reported that the supercritical process provided high biodiesel production capacity, faster process, technical benefits, and significantly reduce wastes compared to the conventional process in biodiesel production (Han et al., 2005; Kusdiana and Saka, 2004; Lee and Saka, 2010; Yin et al., 2008b). The operational efficiency of supercritical technology can be achieved by planning an efficient heat flow plant using Pinch technology (Pięt et al., 2015).

2.2.4. Liquid phase plasma discharge technology

In recent years, plasma discharge technology has been applied in biodiesel production. Moreover, plasma is the ionized gas phase that contains electrons, ions, as well as neutral particles (Mostaghimi and Boulou, 2015), and can be obtained in two forms, namely, thermal and cold plasma (Takai, 2008). The thermal plasma is generated when at a high temperature of at least 3500 °C, which is the equilibrium temperature between ions and electrons (Mostaghimi and Boulou, 2015). However, it cannot directly be used for reaction due to its high temperature, which decomposes all reactants, and makes the reaction uncontrollable. Currently, liquid phase plasma discharge (LPPD) was introduced as the controllable plasma technology and has been used in several applications such as nanomaterials, catalyst, organic compounds degradation, and triglyceride cracking for biodiesel production (Du et al., 2007; Meeprasartsagoog et al., 2017). The energy input of LPPD can easily be adjusted for certain purposes. The plasma introduction in biodiesel production can be conducted in the non-catalytic and catalytic systems.

The plasma can transform triglyceride into diglyceride and monoglyceride for easy ester formation through transesterification (Khani et al., 2015), which also increases the rate of reaction due to the massive triglyceride bond breaking (Gharibi et al., 2015). During the plasma discharge assisted transesterification process, electrons with high energy run through the electrode with high voltage to the other electrode in ground state and excite the bulk gas molecules. This gas molecules excitation generates the ionization of atoms and forming the metastable species such as the reactive radical molecules (Wu et al., 2020). The collisions between the reactive species and the metastable species with the reactant molecules result in the production of variety compounds. Specifically, during the plasma assisted transesterification of oil feedstock, the C-C bonds in the triglyceride split, with the presence of methanol as the oxygen donor in the reaction, the oxygen molecules replace the C-C double bonds, subsequently result in the saturated
molecules (Kongprawes et al., 2021). Istadi and coworkers conducted a study of plasma assisted transesterification process using palm oil as a feedstock. They found that using a plasma voltage of 10 kV, electrode distance of 1.5 min, and a reaction time of 2 min, they achieved ester yield of 75.65% (Istadi et al., 2014). Similarly, another study also reported that plasma introduction in the transesterification is a promising method for efficient biodiesel synthesis (Almarashi et al., 2020).

Table 3 shows the recent biodiesel production using plasma discharge technology. This showed that plasma technology provides a new approach to initiate biodiesel formation using a different form of energy compared to the conventional method. It has process flexibility that can be carried out in the catalytic and non-catalytic systems. However, further researches are needed to determine the optimum operating conditions and understand the reaction mechanisms to make the method more controllable. As a summary for an overview of advanced technology in biodiesel production, the comparison including advantages and disadvantages of the discussed advanced process is summarized in Table 4.

Table 3
Comparison of several plasma discharge-assisted biodiesel productions.

| Feedstock          | Type of catalytic process | Catalyst          | Operating conditions                                      | Ester yield (%) |
|--------------------|----------------------------|-------------------|----------------------------------------------------------|-----------------|
| Palm oil           | Non-catalytic              | -                 | Coaxial plasma discharge, Voltage = 220 V, Flowrate = 0.1318 L/min | 10.1            | (Fan et al., 2018) |
| Mixed oil          | Non-catalytic              | -                 | Dielectric barrier plasma discharge, Voltage = 10.2 kV, t = 120 min, 40 °C | 65              | (Nabilla et al., 2019) |
| Palm oil           | Non-catalytic              | -                 | Dielectric barrier plasma discharge, Voltage = 10 kV, t = 120 min, Ar gas | 56.25           | (Nabilla et al., 2019) |
| Mixed oil          | Non-catalytic              | -                 | Dielectric barrier plasma, Voltage = 10 kV, t = 120 min, 40 °C | 60.72           | (Nabilla et al., 2019) |
| Used oil           | Non-catalytic              | -                 | Dielectric barrier plasma, Voltage = 10 kV, t = 120 min, 50 °C | 56.25           | (Zara et al., 2020) |
| Used oil           | Non-catalytic              | -                 | Dielectric barrier plasma, Voltage = 10 kV, t = 100 min, ambient condition | 70              | (Zara et al., 2020) |
| Soybean oil        | Homogeneous                | NaOH (0.8%)       | LPDP plasma, V = 1.2 kV, Flowrate = 2.7 mL/s | 99.5            | (Wu et al., 2019b) |
| Ethyl acetate      | Homogeneous                | NaOCH₂ (1%)       | Dielectric barrier plasma, Voltage = 15.6 kV, t = 90 min, ambient condition | 77              | (Oliveira Palm et al., 2022) |
| Ethyl acetate      | Homogeneous                | H₃PMo (1%)        | Dielectric barrier plasma, Voltage = 15.6 kV, t = 90 min, ambient condition | 90              | (Oliveira Palm et al., 2022) |
| Oleic acid         | Homogeneous                | H₂SO₄ (1.25%)     | Dielectric barrier plasma, Voltage = 2.2, t = 4 min, ambient condition | 80              | (Wu et al., 2020) |
| Oleic acid         | Homogeneous                | H₂SO₄ (1%)        | Dielectric barrier plasma, Voltage = 2.12, t = 2 min, ambient condition | 78              | (Bashir et al., 2021) |
| Waste cooking oil  | Homogeneous                | NaOH (1%)         | Flying jet plasma DBD, Voltage = 2.5 kV, t = 45 min | 95.4            | (Abdul-Majeed et al., 2016) |
| Rape straw         | Heterogeneous              | Zn-Ti-Zeolite (3%)| Dielectric barrier plasma, Voltage = 5 kV, t = 1.25 min, 65 °C | 25              | (Wu et al., 2019a) |
| Soybean oil        | Heterogeneous              | K₂O/CaO-ZnO (6%)  | Dielectric barrier plasma, Voltage = 5 kV, t = 1.25 min, 65 °C | 77.19           | (Buchori et al., 2017a) |
| Soybean oil        | Heterogeneous              | Activated carbon  | Dielectric barrier plasma, Voltage = 7 kV, t = 5 min, 65 °C | 92.3            | (Buchori et al., 2017b) |
| Soybean oil        | Heterogeneous              | Sulfonated ZnO    | Dielectric barrier plasma, Voltage = 7 kV, t = 1.25 min, 65 °C | 56.91           | (Buchori et al., 2017b) |
| Sunflower oil      | Heterogeneous              | MgO (3%)          | Glow discharge plasma, 45 min | 95              | (Rahmani Vahid et al., 2017) |
Table 4
Comparison of the advanced process of biodiesel production.

| Advanced method               | Main properties | Pros                                                      | Cons                                   | Ref.                  |
|--------------------------------|-----------------|-----------------------------------------------------------|----------------------------------------|-----------------------|
| Microwave-assisted method      | T = 40 – 100 °C | • T = 5 - 60 min                                          | • Efficient heating                    | (Binnal et al., 2021;) |
|                               | t = 5 - 800 W   | • Catalyst = 0.5-5%                                       | • Uniform heat distribution            | (Mamo and Mekonnen, 2020) |
|                               | Yield = 80 - 99 | • Energy = 500 - 800 W                                     | • Faster process                       |                       |
| Ultrasonic-assisted method     | T = 40 – 65 °C | • Frequency = 0.25 – 60 kHz                               | • High yield                           | (Florez et al., 2019;) |
|                               | t = 10 – 240 min| • Catalyst = 1-6%                                        | • Low wastes                           | (Marulanda and Alegria, 2019;) |
|                               | P = 1.4 kW/m³   | • Energy = 1.4 kJ/min                                      | • Easier separation                    | (Stavarache et al., 2007) |
|                               | Yield = 90 - 99 | • Frequency = 25 – 60 kHz                                  | • Less energy lost                     |                       |
| Supercritical transesterification | T = 280 – 350 °C | • T = 20 – 30 min                                        | • No pretreatment                      | (Deshpande et al., 2017;) |
|                               | t = 5 – 40 MPa  | • Catalyst = catalyst free                                | • Short residence time                 | (Lee and Saka, 2010;)  |
|                               | P = 1.0 - 94%   | • Energy = 5 – 10%                                        | • Low wastes                           |                       |
| Plasma discharge technology    | T = 20 – 50 °C  | • T = 0.02 - 2 min                                        | • Available for all types of           | (Mostaghimi et al., 2015;) |
|                               | t = 0.02 - 3 min | • Catalyst = 0.5 – 3%                                     | • Feedstocks                          | (Takai, 2008;)         |
|                               | P = 100 – 300 W | • Energy = 100 – 300 W                                    | • Less loss of energy                  |                       |
|                               | Yield = 78 - 99 | • Frequency = 240 min                                     | • Faster reaction                      |                       |

3. Parameters in biodiesel production

Several factors affecting the performance of biodiesel production have been previously reported. Meanwhile, the type of biodiesel feedstock, type of catalysts, and operating conditions are discussed in this following sections as previously described in Fig. 1.

3.1. Types of feedstocks

The initial generation of biodiesel derived from agricultural goods such as peanut, soybean, and canola have implications on food production and the environment for people’s use. In the early 1940s, 5% of biodiesel with gasoline as an alternative fuel was used for vehicles. Fig. 3 shows the classification of the biodiesel feedstock generations. In this first generation of biodiesel, all raw oily feedstocks were derived from edible agricultural products such as soybean, palm oil, and peanuts, due to their ease of handling and large availability (Kumar et al., 2020; Noriega and Narváez, 2020). Meanwhile, the second-generation biodiesel was generated using cellulose substrates such as short-rotation trees, grassland plants, and urban trash. Jatropha is a non-edible feedstock, which necessitates a large quantity of farmland and produces low oil (Baskar et al., 2018; Keera et al., 2018; Saputra Nursal et al., 2021). Based on current research, the development of alternative ways to lessen food farmland rivalry was continued until the discovery of microalgae as a reliable and rich biofuel feedstock recognized as the third-generation of biodiesel. Microalgae-based biodiesel is a feasible third-generation option since it has no impact on food sources (Abo et al., 2019; Alami et al., 2021; Saputra Nursal et al., 2021). It can also be used for both effluent phytoremediation and fabrication of biodiesel and also to remove carbon dioxide from the air (Abo et al., 2019). Furthermore, algae may be cultivated on any accessible surface such as lands, lakes, oceans, and generate more triglycerides that have been identified as a possible source for the production of biodiesel.

The accessibility of oil sources is a crucial determinant of biodiesel production’s economic viability because it contributes approximately 80% of the overall cost of biodiesel (Kumar et al., 2020; Noriega and Narváez, 2020; Hadiyanto et al 2020). Various attempts have been devoted to determining a low-cost feedstock that is available throughout the decade. Compared to standard fuels, FAME from algae, plant oils, and animal fats have been demonstrated to be a source of biodiesel synthesis due to enhanced combustion behavior, volatility, and viscosity. Fish oils and beef tallows are the most common animal fat sources, while vegetable oils such as rapeseed oil, castor berry, palm pulp, palm kernel oil, sunflower seeds, coconut kernel, cottonseed, peanut grain, and canola seed were used in the biodiesel manufacturing process (Athar and Zaidi, 2020; Pinzi et al., 2014; Yusuff et al., 2021). Microalgae can increase their biomass in 24 hours, with the quickest half-life of approximately 3.5 hours, which makes them an attractive sustainable source for biodiesel synthesis (Abo et al., 2019; Alami et al., 2021; Saputra Nursal et al., 2021). Its enormous amount and use of richer
nitrogen, as well as phosphorus in wastewater as an inexpensive source of nutrients, benefits from algae cultivation. Algal species have the disadvantage of being obligate photrophs, which indicate that they need light to survive. The amount of FFA and contaminants in biodiesel also affects the kind of manufacturing technique employed and the amount of fuel produced. Similarly, lipid leftovers such as waste cooking oil and non-edible beef tallow have recently attracted much attention from the biodiesel industry. However, the discovery of new additional options that do not interfere with food sources is critical.

3.2. Types of catalysts

3.2.1. Homogeneous catalysts

Acids and alkalis are included in the homogeneous catalyst, where the alkalis such as sodium hydroxide (NaOH), sodium methoxide (NaOCH3), and potassium hydroxide (KOH) are usually used (Vicente et al., 2004). Meanwhile, in the acidic homogeneous catalyst (H2SO4), sulfuric acid has been commonly used (Wongwuttanasatian and Jookjantra, 2020). KOH behaved well in the transesterification reaction of Karanja oil, yielding methyl esters above 95% at one percent of catalyst dosages in an hour of reaction at 60°C with a mixing rate of 7 rps (Boey et al., 2011). Saponification arises due to an undesirable by-product in a homogeneous catalytic reaction mechanism, which necessitates an extra separation procedure to eliminate catalytic pollutants and increases the final operating costs. In the homogeneous alkali catalytic reactions, (i) the optimal temperature is adjacent to the boiling temperature of the alcohol employed, (ii) sufficient alcohol is required to facilitate excellent conversion, where 1:6 is regarded as the ideal oil:Me-OH molar ratio (Dias et al., 2008). Furthermore, various basic catalysts such as NaOCH3, KOH, NaOH, and KOH for methanolysis of sunflower oil were compared with all reactions in an agitated reaction vessel and the following purification stages in a decanter under the test condition. Methoxides are more efficient than hydroxides, with a yield of around 100% (Boey et al., 2011). The benefits of using a homogeneous catalyst for biodiesel synthesis include low reaction temperatures, the high catalytic performance of base catalysts, and conversion in a relatively short period. The basic type of heterogeneous catalysts is also substantially more active than acid catalysts. After the processing and treatment of wastewater, saponification generates a stable colloid, the used catalysts that cannot be regenerated, basic catalysts which are delicate to the concentration of water and FFA are the key restrictions faced by homogeneous catalysts. The comparison of used catalyst for biodiesel production is listed in Table 5.

3.2.2. Heterogeneous catalysts

Heterogeneous catalysts have the potential for the transesterification process of plant oils to make biodiesel. Meanwhile, heterogeneous catalysts without homogeneous catalysts can be regenerated, renewed, and employed in continuous operations (Ganesan et al., 2021; Helmi et al., 2021; Jayakumar et al., 2021). Since their employment in the transesterification process substantially simplify and optimizes the post-treatment of the products, there seems to be a recent surge in the invention of acidic or basic solid heterogeneous catalysts for FAME generation (Jayakumar et al., 2021; Xie and Wang, 2020). Generally, heterogeneous catalyzed biodiesel manufacturing techniques have fewer processing steps, post-treatment stages, and do not entail neutralization. The kind of catalyst (acid or base), the catalyst quantity, the flow reaction time, the extent of stirring and mixing, the oil/alcohol percentage, and the quality of the feed affect heterogeneous catalysis (Deeba et al., 2020; Helmi et al., 2021; Xie and Wang, 2020). The detailed advantages and disadvantages of heterogeneous catalysts to homogeneous catalysts can be seen in Table 6.

Furthermore, the use of a solid catalyst can lead to faster and less expensive separation procedures, minimum wastewater discharge, lower production and energy expenditures (Rezania et al., 2022; Siddiquee et al., 2011). Solid catalysts can be carried out in the packed bed reactors as the catalyst bed that simultaneously perform catalytic reaction and separation. Therefore, the extra physical separation processes and their related operational expenses can be eliminated. Heterogeneous catalysts are also less harsh and are used in fixed-bed reactors, leading to a safer, better cost-effective, and eco-friendly process. Their advantage is that they consume less energy during the transesterification process (Awogbemi et al., 2021; Jayakumar et al., 2021; Rezania et al., 2022; Siddiquee et al., 2011). In this process, 10 tons of sodium hydroxide are needed to make 1,000 tons of biodiesel, while the heterogeneous requires only 0.7 tons of magnesium oxide solid catalysts to make 12,500 tons of biodiesel (Jayed et al., 2009). Although the heterogeneous process has shown a more effective catalyst to biodiesel product, there are still some challenges, which includes slow reactions, partial conversions, short lifespan, and expensive prices (de Oliveira and Coelho, 2017; Eguchi et al., 2015; Krishnan et al., 2021). Therefore, the homogeneous process is widely selected for biodiesel synthesis in the current industrial processes. The major drawback of a heterogeneous catalyst, which is its slower reaction rate compared to homogeneous catalysis can be solved by increasing the methanol-to-oil proportion, temperature, and pressure of the process (Alagumalai et al., 2021; Hamzza et al., 2021; Krishnan et al., 2021; Zailan et al., 2021). These treatments have been reported to attribute to the faster rate and higher biodiesel yield using the heterogeneous catalytic process (Awogbemi et al., 2021; R. Ganesan et al., 2021; Hamzza et al., 2021; Jayakumar et al., 2021; Krishnan et al., 2021; Patiño et al., 2021). To protect combustion damage to the engine, several pollutants such as unreacted FFAs, TAGs, glycerol, catalyst, mono- and diglyceride, water, and other impurities need to be separated from unrefined biodiesel by post-production purification stages, such as adsorption, filtration, ion-exchange, and other methods (Catarino et al., 2020; Gomes et al., 2015; Hajra et al., 2015; Li et al., 2012; Wang et al., 2021).

Inorganic-synthesized catalysts such as calcium oxide (CaO), silicon dioxide (SiO2), zircon oxide (ZrO2), iron oxide (Fe2O3), and others are usually used due to their low cost, accessibility, safety, robustness, and ease of regeneration (Hadiyanto et al., 2016). Meanwhile, current studies have focused on low-cost, environmentally friendly, and incredibly efficient heterogeneous-base
catalysts, where calcium oxide-based from waste or natural sources has shown the most potential (Baskar et al., 2018; Jayakumar et al., 2021; S. Silviana et al., 2021b; Xie and Wang, 2020). Calcium-compounding shells, limestone, and lime mud have been proven to have superior catalytic characteristics, which make them appropriate for biodiesel generation (Ajala et al., 2020). Furthermore, they make the reaction more cost-effective, ecologically beneficial, and also reduce waste. This occurs because they needed a relatively small amount of catalyst for the biodiesel synthesis, which generates smaller liquid waste compared to the homogeneous catalyst process. Strong acid such as sulfuric acid that simultaneously facilitates both transesterification and esterification processes are particularly important for biodiesel synthesis from low-cost raw stocks with high FFA concentration (Binnal and Nirguna Babu, 2019). Therefore, the invention of the novel, more powerful, and less expensive solid catalysts is expected to reduce the overall cost of producing biodiesel from the various oily feedstock. A better catalyst activity can also be accomplished through porous materials support, which increases TAG and FFA transport to reactive acid/basic sites and improves the rate of the reaction. In addition, environmentally friendly innovations such as membrane reactors, ultrasounds, and microwaves can also be used to solve operational issues such as excessive power consumption, long response times, and poor catalytic performance, which increases the costs of production.

Table 5
Biodiesel production using several types of feedstocks, catalysts, and stoichiometric ratio of methanol and oil.

| Type of catalyst | Feed oil          | Catalyst addition (%wt) | Oil to methanol ratio | Ester yield (%) | Ref.                  |
|-----------------|-------------------|-------------------------|-----------------------|----------------|----------------------|
| Homogeneous catalysts |                   |                         |                       |                |                      |
| Sodium hydroxide (NaOH) | Sunflower oil     | 1.00                    | 1:6                   | 97.0           | (Vicente et al., 2004) |
| Sodium methoxide (NaOCH3) | Sunflower oil     | 1.00                    | 1:6                   | 99.0           | (Vicente et al., 2004) |
| Potassium hydroxide (KOH) | Sunflower oil     | 1.00                    | 1:6                   | 91.7           | (Vicente et al., 2004) |
| Potassium methoxide (KROH) | Sunflower oil     | 1.00                    | 1:6                   | 98.0           | (Vicente et al., 2004) |
| Sulfuric acid (H2SO4) | Oleic acid        | 1.25                    | 1:3                   | 80             | (Wu et al., 2020)      |
| Sulfuric acid (H2SO4) | Oleic acid        | 1.00                    | 1:3                   | 78             | (Bashir et al., 2021)  |
| Barium hydroxide (Ba(OH)2) | Microalgae       | 3.00                    | 1:6                   | 90.8           | (Sarve et al., 2016)   |
| Chloro-sulfonic acid (ClSO3H) | Maize oil        | 3.00                    | 1:6                   | 93.1           | (Dall’Oglio et al., 2015) |
| Heterogeneous catalysts |                   |                         |                       |                |                      |
| Calcium oxide (CaO) | Sunflower oil     | 1.00                    | 1:12                  | 91.0           | (Boey et al., 2011)    |
| Potassium fluoride (KF) | Tallow seed       | 4.00                    | 1:6                   | 96.8           | (Jayakumar et al., 2021) |
| Potassium nitrate (KNO3) | Rape oil          | 1.00                    | 1:12                  | 98.0           | (Jayakumar et al., 2021) |
| Titanium oxide (TiO2) | Canola oil        | 6.00                    | 1:30                  | 100            | (Jayakumar et al., 2021) |
| Magnesium oxide (MgO) | Mutton fat        | 4.00                    | 1:22                  | 98.0           | (Foroutan et al., 2020) |
| Aluminum oxide (Al2O3) | Palm oil          | 5.97                    | 1:12                  | 98             | (Boey et al., 2011)    |
| Zinc oxide (ZnO) | Used cooking oil  | 1.50                    | 1:14                  | 90             | (Yussuff et al., 2021)  |
| CeO2/Li/SBA-15 | Cotton seed oil   | 10                      | 1:40                  | 98             | (Malhotra and Ali, 2018) |
| Cu impregnated TiO2 | Palm oil          | 2                       | 1:20                  | 90.93          | (De and Boxi, 2020)    |
| NaAlO2/Al2O3 | Palm oil          | 10.89                   | 1:21                  | 97.65          | (Zhang et al., 2020)   |
| Ca/ABP-700 | Waste cooking oil | 5                       | 1:30                  | 93.4           | (Wang et al., 2019)    |
| LiFeO8-LiFeO2 | Soybean oil       | 8                       | 1:36                  | 96.5           | (Dai et al., 2018)     |
| Enzymes |                   |                         |                       |                |                      |
| Lipase from Rhizopus oryzae | Soybean oil    | 1.00                    | 1:3                   | 85.0           | (Adewale et al., 2017)  |
| Lipase from Aspergillus niger | Canola oil   | 1.00                    | 1:3                   | 69.0           | (Andrade et al., 2019)  |
| Lipase/APTES-magnetite | Rapeseed oil       | 20                      | 1:6                   | 89.4           | (Gojun et al., 2021)    |
| Thermomyces lanuginosus/ magnetite/Au NPs | Waste tomato oil | 20                     | 1:6                   | 98.5           | (Sarno and Iuliano, 2019) |
| Novozyme 435 | Fish oil          | 50                      | 1:35                  | 82.9           | (Marín-Suárez et al., 2019) |
| Palatase | Soybean oil       | 10                      | 1:3                   | 71             | (Pedro et al., 2020)    |
| Immobilized CLEAs of Km 12 lipase | Waste cooking oil | 0.3                    | 1:3                   | 71             | (Badoei-dalfard et al., 2019) |
| NS 40116 lipase | Chicken fat       | 0.3                     | 1:4.5                 | 77             | (da Silva et al., 2018) |
| NS 40116 lipase | Soybean oil       | 3                       | 1:6                   | 96             | (Mibielli et al., 2020) |
| Rhizopus oryzae lipase | Palm oil          | 1.5                     | 1:3                   | 91.3           | (Muanruksa and Kaewkammet, 2020) |
| Lipase immobilized Fe3O4-poly (GMA-co-MAA) | Soybean oil | 1.5                     | 1:4                   | 92.8           | (Xie and Huang, 2020)   |
| Nano-catalysts |                   |                         |                       |                |                      |
| Nano-CaO | Soybean oil       | 1.00                    | 1:7                   | 96.0           | (Foroutan et al., 2020) |
| Nano-MgO | Soybean oil       | 3.00                    | 2:3                   | 99.0           | (Foroutan et al., 2020) |
| Nano-KF | Canola oil        | 3.00                    | 1:15                  | 82.1           | (Jayakumar et al., 2021) |
| Ni doped ZnO nanocomposite | Castor oil     | 1.40                    | 1:12                  | 91             | (Baskar et al., 2018)   |
| Diatomite CaO@MgO | Waste cooking oil | 6                       | 1:15                  | 96.47          | (Rabie et al., 2019)    |
| Nano sized waste animal bone | Honie oil | 7                       | 1:12                  | 96             | (Chingakkham et al., 2019) |
Table 6
Comparison of biodiesel production using conventional homogeneous and heterogeneous catalytic process.

| Type of production | Pros                                                                 | Cons                                                                 |
|--------------------|----------------------------------------------------------------------|----------------------------------------------------------------------|
| Homogeneous        | • Mild operating condition<br>• The mixture of FFA and water has little effect on acid catalyst<br>• Hydroxides are ineffective compared to methoxides<br>• Catalysts that are alkaline are substantially more active than those that are acidic<br>• Both esterification and transesterification can use the same acid catalyst | • After-reaction separation issues with wastewater treatment<br>• Acid catalysts are corrosive and have a sluggish reaction rate<br>• The existence of FFA and water makes basic catalysts sensitive<br>• The used catalyst cannot be regenerated<br>• Needing more operating cost than heterogeneous process |
| Heterogeneous      | • Product separation is easier, selectivity is better, and catalyst life is longer<br>• Eco friendly, noncorrosive, reusable, and less problematic in terms of disposal<br>• The existence of FFA and water has little effect on acid catalysts<br>• Relatively low-cost compared to homogeneous process<br>• Possibly used in continuous process using fixed-bed reactors | • The multi-phase reaction system has mass transfer restrictions<br>• In comparison to conventional homogeneous base catalysts, it is apparently less effective<br>• Acid catalysts are more expensive than basic catalysts<br>• The raw oil with low water and FFA content are required for basic catalysts<br>• Temperature and pressure, as well as a high alcohol-to-oil molar ratio, may be necessary<br>• The catalyst is possibly leached out during process |

Fig. 3. Classification of biodiesel feedstock generations.

A transesterification method with base catalyst is used when an oil feed contains high water, FFA, and contaminants, such as used cooking oils. Extra consideration must be given to the production planning and catalyst selection especially more favorable on that condition with high contents of water, FFA, and contaminants (Deeba et al., 2020; Siddiquee et al., 2011). However, the design process needed an extremely high temperature, longer time of reaction, and equipped in corrosion resistance vessel. To overcome these challenges, a pre-esterification must be carried out using an acid catalyst to convert FFAs into esters, followed by transesterification with a basic catalyst to also synthesize TGAs. Since both processes are reversible, a large amount of alcohol is frequently used to drive the process to the synthesis of methyl ester. Meanwhile, biodiesel synthesis is carried out in a single step through a bi-functional catalyst that can perform the processes simultaneously (Al-Saudie et al., 2020). Table 6 shows the advantage and disadvantage of homogeneous and heterogeneous biodiesel synthesis method relative to each other.

3.2.3. Enzyme-based catalysts

Lipases are currently the most used enzyme in biodiesel synthesis, grouped into the hydrolases process, which is an enzymatic process that hydrolyzes the
molecules of triglyceride into fatty acids and glycerol. The lipase enzyme can be obtained from numerous sources such as bacteria, plants, fungi, and animals. Recently, the lipase enzyme from microbes (bacteria and fungi) is commonly used as biodiesel catalysts (Adewale et al., 2017; Andrade et al., 2019; Chang et al., 2021). Some advantages of enzyme-based catalysts in biodiesel synthesis include an environmentally friendly type of catalysts that are derived from microbes and can carry out the transesterification reaction in the high level of free fatty acids and water. Moreover, the enzymatic catalysis reacts in the mild ambient condition that caused a lower energy requirement, which shows the sustainability of this process for biodiesel production (Marín-Suárez et al., 2019). Immobilized lipase differs from pure lipase because it can be easily recovered from the reaction medium, allowing for repeated usage. The efficiency of the enzymatic process is great, and the enzyme can be possibly immovable upon the solid matrix, which includes the amount of biodiesel that is retrieved (Marín-Suárez et al., 2019). There are several reaction pathways of the biodiesel synthesis using enzyme catalysts. The active sites on the enzymes attributing the positive or negative charge initiate the reaction by accepting or releasing protons via the Bi-model mechanism based on the enzyme types (Pedro et al., 2020). The lipase enzyme plays as the proton acceptor of hydroxyls (OH), while on the amine group-based enzymes, they release protons (Pedro et al., 2020; Sarno and Iuliano, 2019).

The main drawback in the use of enzymatic catalysts is the high cost of extraction from the sources which usually appears in low yield. Therefore, research was carried out on the integration of enzymes with several nanomaterials to increase the catalyst activity and the yield of biodiesel. This includes the use of the amino-coated iron oxide (FeOx) material, which was crosslinked with the Km12 lipase enzyme (Sarno and Iuliano, 2019). It was discovered that when stored for 24 days at a temperature of 4 °C, it gave a 60% higher initial enzymatic activity compared to the pristine enzyme. The current works of enzymatic catalyst utilization in biodiesel production with their findings are summarized in Table 5. By carefully examining the data in Table 5, this indicated that enzymatic catalysts averagely gave lower biodiesel yield compared to homogeneous and heterogeneous catalysts. Furthermore, the results showed that different types of enzymes, feedstocks, and oil to alcohol ratios gave different yields of biodiesel. Therefore, selecting an appropriate enzyme and operating parameter is essential to achieve a desirable yield of biodiesel.

3.2.4. Nano-catalysts

The heterogeneous catalysts have a different phase from the reactants in biodiesel production, which can be attributed to the acidic and alkali properties based on the chemical composition (Athar and Zaidi, 2020; Jayakumar et al., 2021; Kumar et al., 2010). Their use considerably reduces the number of separation treatments compared to the homogeneous ones. However, lower conversion carried out by the common heterogeneous catalysts initiated the scientists and engineers to develop higher efficient catalyst by synthesizing in form of nanoparticles. The heterogeneous catalysts that appeared as nanomaterials are commonly named the nano-catalyst (Naveenkumar and Baskar, 2020). Meanwhile, research has been carried out on the influence of nano-catalyst on triglycerides transesterification to FAMEs (Ashok et al., 2018; Dehghani and Haghghi, 2020; Foroutan et al., 2020; Liu et al., 2020), as the result can be seen in Table 5. The transesterification process of soybean oil production is improved using MgO as a catalyst (Rahmani Vahid et al., 2017). This process uses ferromagnetic ZnO nanocomposite as a solid catalyst for generating biodiesel from castor oil as a feedstock (Baskar et al., 2018). Research on X-ray diffraction (XRD) showed that the nanoparticles were in a single phase. However, after six cycles of soybean oil and four cycles of poultry fat, biodiesel made from soybean oil using CaO nanoparticles under ambient temperature exhibited a poor speed of the reaction and needed six to 24 hours to achieve a high yield (Foroutan et al., 2020). The use of nanomaterial is costly, but it produces more than 90% of biodiesel conversion. Nanomaterials can provide larger active site for catalytic reaction compared to the conventional heterogeneous catalysts.

3.3. Operating condition

3.3.1. Temperature

One of the most critical elements that affect the output of biodiesel synthesis is the process temperature. This is because an increase in temperature and a decrease in oil viscosity produced a faster response speed. However, increasing the temperature above the optimal level reduces biodiesel yield caused by the higher temperature, which accelerates triglyceride soap formation. To minimize the alcohol loss, the operating temperature must be lower than the alcohol’s boiling temperature. Generally, the optimal temperature for biodiesel synthesis is between 50 and 60°C based on the oil quality and the catalyst (Al-Saadi et al., 2020; Deeba et al., 2020; Krishnan et al., 2021).

3.3.2. Alcohol and oil ratio

Alcohol and oil commonly have a 3:1 stoichiometric ratio, however, excessive use of alcohol is harmful to the advanced transesterification reaction (Sawangkeaw et al., 2011). The most widely known alcohols used in the transesterification process are methanol and ethanol. In a 2.5-hour reaction period at 65°C with a 10:1 methanol/oil stoichiometric ratio and 450 rpm of stirring, approximately 80% biodiesel production was achieved from soybean oil with 0.1 percent NaOCH3. It was also stated that waste cooking oil converted to FAME in 69 hours at a temperature of 65°C and a stoichiometric ratio of 1:3 oil to methanol (Marchetti et al., 2007; Sawangkeaw et al., 2011).

3.3.3. Agitation

Since the mixing of the oil feed and catalyst combination promotes the process, agitation speed is significant in the synthesis of FAME (Demirbas, 2010; Elgarhi et al., 2020). For illustration, the mixing intensities of 200-800 rpm were selected for 1 hour, while other variables remain unchanged and the final product conversion rate was greater at 400 rpm (Demirbas, 2010). Moreover, soap production happens at greater mixing speeds, while byproduct creation occurs at slower agitation.
speeds due to the transesterification reaction's reversible character.

4. Biodiesel purification method

The purification treatment aims to separate the generated soap, glycerol, water, unreacted alcohol, and catalysts from biodiesel products. The generated esters should be free of those impurities to fulfill the standard product of biodiesel. Moreover, the different production processes of biodiesel caused a variation in the product specification, as shown in Table 5. Based on the mechanisms, the purification techniques that have been used since last decades for biodiesel until now can be grouped into wet washing, dry washing, ion exchange and precipitation, complexation, membrane-based separation, adsorption, as well as simultaneous synthesis and purification.

4.1. Wet washing method

The wet washing method is operated using pure or acidified water (Díaz-Ballote et al., 2020; Meadow et al., 2012; Shirazi et al., 2013). Before the process, water is used at ambient temperature or as heated as possible, and the extra alcohol is occasionally removed by distillation. The use of acidified water was reported to be more effective than pure water (Iglesias et al., 2014). Meanwhile, several dilute acids that are commonly used include phosphoric acid, hydrochloric acid, and sulfuric acid. Although the process needs a lower amount of acidified water than pure water to treat the same amount of unrefined biodiesel, the use of acidified water can acidiﬁ the biodiesel product. In industry, this problem is usually addressed by adding the pure water wash at the end of the acidified water washing to remove the spent acid content in the biodiesel product (Gomes et al., 2015). Currently, the ionic liquid compounds have been used in the wet washing process such as organometallic substances, which were added into the wash water to improve the efficiency and effectiveness (Veljković et al., 2015). The benefits of the wet washing method include a very convenient and straightforward approach for biodiesel refining, extremely useful separation of glycerol and alcohol, as well as the ability for using aqueous acids (Veljković et al., 2015). Table 7 shows some reported works regarding the wet washing process. However, some drawbacks still appear such as the need for a large amount of water, product drying to separate the detectable water content, increase in the consumption of energy, greater surface area for washing unit, and generating a large amount of wastewater that contain hazardous substances, which is difficult to be treated.

4.2. Dry washing method

Dry-cleaning was created to replace biodiesel purification with no-water purifying processes that are more eco-friendly, where chemicals such as adsorbents and acidic polymers are used to remove contaminants from unrefined biodiesel (Catarino et al., 2020; Gomes et al., 2015). After mixing for 20 minutes at 55°C, the unrefined biodiesel was treated with 2% magnesium and the adsorbent was recovered by filtering (Zhu et al., 2006). However, to increase the separation efﬁ ciency, the reﬁ ned biodiesel from dry washing process is sometimes washed with pure water in a stirred tank unit (Ilim et al., 2017). The biodiesel phase is separated from the mixture by decantation and centrifugation.

Another type of adsorbent that has been applied in biodiesel puriﬁ cation was silica derived from geothermal solid waste, which is one of the potential silica sources (Silviana et al., 2021). Previous research reported that the silica adsorbent has an optimum glycerol adsorption capacity of 10.06 mg/g (Silviana et al., 2021a). The beneﬁ ts of dry washing include no threat of water, continuous process, a reduction in overall process time, and wastewater. However, the drawbacks include adsorbent that do not eliminate alcohol, the requirement for additional apparatus, and slightly higher operating expenses.

4.3. Ion-exchange and precipitation

Precipitating agents are used to separate Ca²⁺ ions from raw biodiesel in this technique. If a precipitation chemical, like oxalic and citric acid are introduced to calcium-containing unrefined biodiesel, an insoluble substance forms in the mixture (Musial et al., 2011; Vieira et al., 2017). Another study showed that washing the crude biodiesel in a mixture of sodium carbonate (Na₂CO₃) and methanol at boiling temperature. Calcium soap have a more non-polar property, which being less soluble in water and more soluble in biodiesel, thus increases the difﬁ culty in separation. After exchanging calcium ions by sodium ions, the generated sodium soaps can be easier to remove using wet or dry method (Musial et al., 2011; Vieira et al., 2017). This process was carried out to exchange the calcium ions from the crude biodiesel with the sodium ions from sodium carbonate to form the water-insoluble calcium carbonate, with reaction as follows (Veljković et al., 2015):

\[
\text{Ca}^{2+}_{(aq)} + \text{Na}_2\text{CO}_3(s) \rightarrow \text{CaCO}_3(s) + 2\text{Na}^+_{(aq)}
\]

Methanol is used to prevent ester hydrolysis and as a reagent in the transesteriﬁ cation process. The formed calcium carbonate has water-insoluble properties that are easily separated while the remaining sodium ions can be removed using wet or dry washing methods.

Further development in ion exchange and precipitation method has been carried out as a pretreatment process, followed by subsequent acid resin, ceramic membrane, and wet washing (Hajra et al., 2015; Li et al., 2012). At the beginning of the process, the crude biodiesel was treated using methanol containing sodium carbonate and was mixed under a constantly stirred vessel at 60°C for 5h at a speed of 1,200 rpm. The calcium ions were exchanged by sodium ions to generate the calcium carbonate. The calcium carbonate and unreacted sodium carbonate were separated using conventional ﬁ ltration, while methanol was separated with gravitational settling treatment. Moreover, centrifugation or ﬁ ltration can be used to remove the deposited component from pure biodiesel. The microﬁ ltration membrane and wet washing method (pore size 0.1 µm) were also used to remove the sodium ions and the results are compared (Li et al., 2012). Overall, the pretreatment and membrane ﬁ ltration or wet washing methods were efﬁ cient in sodium and calcium removals from crude biodiesel.

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| Ref | Cat | Ope. Cond. | Purification | Findings |
|-----|-----|-----------|--------------|----------|
| (Kim et al., 2011) | ZnO/ZrO<sub>2</sub> | Autopressure, 200°C, 2 h | Washing and product drying | The product meets the ASTM diesel fuel standard for sulfur content below than 500 ppm |
| (Salamatinia et al., 2012) | SrO<sub>2</sub> | Stirred batch reactor, 65°C | Washing, centrifuging, and drying | Wet washing slightly reduced the biodiesel density, while increased in viscosity |
| (Jitputti et al., 2006) | Sulfonated ZrO<sub>2</sub> | Standard batch reactor, 200°C, 50 kPa, 1 h | Washing and drying | The product meets the ASTM standard for biodiesel |
| (Kumar et al., 2010) | Na/SiO<sub>2</sub> | Ultrasonic equipped batch reactor, 24 kHz, 15 min | Washing and drying | The product meets the ASTM standard for biodiesel |
| (Iglesias et al., 2014) | SBA-15 functionalized ZrO<sub>2</sub> | Up flow fixed-bed reactor, 200°C, 70 kPa, 6 h | Washing and drying | The product meets the ASTM standard for biodiesel |
| (Fayyazi et al., 2021) | KOH | Continuous centrifugal contactor separator (CCCS) 10 kHz, 35°C | Washing and centrifugation | Optimized result was 98% of ester yield and 75% energy saving. |
| (Fayyazi et al., 2018) | TransZyme A | Continuous centrifugal contactor separator (CCCS) | Washing and centrifugation | Optimized result was 83.2% of ester yield and productivity of 638 kg/m<sup>3</sup>h |
| (Ilmi et al., 2017) | KOH | Continuous centrifugal contactor separator (CCCS) | Washing and centrifugation | 85% mol of ester yield was achieved during 9 h of process |
| (Bashir et al., 2018) | KOH | Prewashing and wet washing | Washing and centrifugation | Purified biodiesel meets the standards required to be a replacement for diesel. This approach reduces 68% of fresh water in the purification process after 15% less water consumption as compared to two rounds of biodiesel production operations. Considerably less wastewater generation toward zero discharge-based operation |
| (Jaber et al., 2015) | KOH | Wet washing and repeating microfiltration | Washing and centrifugation | Wet washing and repeating microfiltration |
This showed that the benefits of the ion exchange method include a good output of pure biodiesel, a lower volume of water, and easy filtering of the residue. The drawbacks include the dependence of the effective precipitation on operational parameters; therefore, additional investigations are necessary to improve the precipitation settings.

4.4. Complexation

Complexation is a method for biodiesel purification, causing the incorporation of chemical groups, atoms, or ions to form a larger molecule. This method is currently used to separate Ca\(^{2+}\) ions using ethylenediaminetetraacetic acid (EDTA) as the initiator to form the complexation with a molar ratio of 1:1 of EDTA to calcium ions. When the EDTA solution is added to the raw biodiesel product, the reaction takes place in a stirring condition and complex substances between EDTA and Ca\(^{2+}\) are formed (Zhu et al., 2006). The Ca-EDTA residue can be removed from the mixture through centrifugation, where supernatant is biodiesel and the residue is the Ca-EDTA (Zhu et al., 2006). Decalcification can achieve 84% of efficiency with 92% yield in biodiesel. Some benefits of using this method include a high amount of calcium ions that can be separated from the raw biodiesel and soluble in water. However, the disadvantages include the difficult preparation of EDTA and are not fully eco-friendly.

4.5. Membrane-based separation

Membrane filtration is a well-known modern technology that is usually applied in water science, protein separation, and purification purposes. The technology is commercially used in aqueous mixture separations, however, the purification of the non-aqueous mixture by the membrane is currently thriving. The membrane can be in form of organic, inorganic, or a combination of both. Since they are thermally and chemically stable, the former type especially for the inorganic ceramic membrane has been applied in a cross-flow system to separate triglycerides from FAMEs mixture (Shirazi et al., 2013; Sokać et al., 2020; Bansod et al., 2021; Goswami and Pugazhenthi, 2021). The pore diameter for the oil emulsion was set to be about 40 microns. Ultrafiltration and tubular ceramic membrane are the most efficient for biodiesel purification that attributed the environmentally friendly technology compared to the other type of membranes. Several benefits of the membrane-based technology in biodiesel purification include high rejection of unreacted alcohol and generated soap, simple and easy to operate, low energy consumption, easy to control and scale-up, and eco-friendly. However, some of its drawbacks include the membrane that needs to be cleaned up periodically and flux reduction gradually over time. The collective information regarding previous studies results about biodiesel purification can be seen in Table 8.

4.6. Simultaneous synthesis and purification of biodiesel

Research has shown that the simultaneous synthesis of biodiesel formation and purification enhances the overall manufacturing process. Such coupled mechanism has been studied so far (Gojun et al., 2021; Song et al., 2021). It can possibly overcome several drawbacks of current purification methods for biodiesel as shown in Table 8. During the ester formation, they introduced ion exchange preceded by precipitation. It has also developed a bench-scale biodiesel synthesis and purification simultaneously using a reactor equipped with packed anion- and cation-exchange resins at a temperature of 50°C. The cation-resin was firstly introduced to the reactor to accelerate the esterification of FFA, while the anion-resin was loaded after that for transesterification of TAG and also as the adsorbent for the purification treatment of the generated biodiesel. Subsequently, the biodiesel was generated at a flowrate of 0.23 l/h having a TAG content lower than the standard (Veljković et al., 2015). The glycerol, water, alcohol, and unreacted FFA as well as TAG were adsorbed onto the anion exchange resin. Therefore, this process is called simultaneous biodiesel formation and purification because they take place at the same time. Several advantages of this method include shorter time for the processing of the oily feedstock to biodiesel, does not require additional equipment, and is relatively cheaper than the conventional method. However, it has some drawbacks such as the resins needed to be regenerated periodically and properly designed to adsorb various types of impurities with different chemical properties.

5. Impacts of biodiesel

In previous years, a substantial body of knowledge about the socioeconomic and environmental implications of biodiesel has been accumulated. The petroleum-based fuel reserve will be drained in the next few years approximately at 2030 due to the rapid increase of people’s needs, which will generate the greatest issue of the 21st century (Edwan Kardenia, 2015). Based on the Organization of Petroleum Exporting Countries (OPEC), global fuel oil consumption is forecast to reach approximately 100 million barrels a day by 2040, with 5.0 million barrels of diesel fuel consumption a day. Furthermore, the unpredictable petroleum-based fuel prices are jeopardizing energy supplies and degrading the payment balance by increasing the cost of imported energy. This has led to a resurgence of curiosity in the manufacturing and use of energies derived from organic waste and plantations. Therefore, biodiesel is an environmentally sustainable fuel source and energy with several potentials that can be used as an alternative source identical to petroleum. The use of biodiesel instead of coal and petroleum has the potential to provide several advantages. This is because biodiesel can be generated locally, reducing the demand for petroleum importation, lowering the cost, and ensuring the country’s economic stability. The negative effects of supply interruptions can be minimized when biodiesel demand and use to minimize the application of petroleum fuels. Moreover, biodiesel manufacturing provides countries without oil reserves energy sovereignty. The financial effects of biofuel are not restricted to the biodiesel industry and agriculture because of the interdependencies across producing areas that influenced the national economy (Anwar, 2021; NaveenKumar and Baskar, 2020).
The use of biodiesel is also considered environmentally beneficial since the greenhouse gasses emission is remarkably reduced. In the process of biodiesel formation, the use of heterogeneous catalysts is environmentally safer than the homogeneous catalyst process due to the minimized generated waste. The purification step in the heterogeneous catalyst process is easier to conduct, however, the process still generated some wastes (Alagumalai et al., 2021; de Mello et al., 2017; Jayed et al., 2009). This includes wastes from the product separation such as used catalyst and adsorbent, fouled membranes during the application, and complexation product of Ca-EDTA. The second type of waste is from the reactivation of regeneration of catalyst or ion-exchange resins such as solvents, used alcohol, and wastewater. Therefore, there is a need to develop the stability of catalyst properties through its use over several consecutive cycles and the handling catalyst. This approach is useful for minimizing the generated wastes upon the application of the heterogeneous method in biodiesel production, which will create more environmental benefits.

### 6. Conclusion
Emerging processes have been developed in recent years such as microwave-assisted, ultrasonic-assisted, supercritical transesterification and liquid plasma discharged-assisted biodiesel synthesis processes. These methods were reported to have a variety number of conversion yields from 75 to 99%, especially the supercritical transesterification that is carried out in a catalyst-free process. However, the main problem to use

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**Table 8** Comparison of the commonly used methods for biodiesel purification

| Purification technique       | Sample work                              | Pros                                                                 | Cons                                                                                   |
|------------------------------|------------------------------------------|----------------------------------------------------------------------|----------------------------------------------------------------------------------------|
| Water (wet) washing          | Feedstock: curcas oil                    | Efficient removals of alcohol and glycerol                          | Require high volume of water for washing                                              |
|                              | Purification: wet washing                | Simple and straightforward process                                   | Long time needed for the process                                                      |
|                              | Ester yield: 98%                         | Can be used to efficiently separate biodiesel from catalysts such as ZnO, SiO₂, ZrO₂, and SrO | Require further drying for the product after washing                                   |
|                              | Ref: (Shirazi et al., 2013)              |                                                                      | Large amount of wastewater is generated during the washing process                    |
| Dry washing (ion-exchange and adsorption) | Feedstock: Waste cooking oil            | Relatively faster process                                           | Non-eco-friendly method                                                               |
|                              | Purification: ion-exchange               | Needing smaller space for the process                               | Might be increase the acidity of biodiesel due to the used of high acidity adsorbents or resins |
|                              | Ester yield: 95.8%                       | Less amount of waste is generated                                   | Not efficient in decalcification                                                      |
|                              | Ref: (Hajra et al., 2015)                | Might be run in continuous system                                   | Can be reduce the acyglycerol from the product                                        |
|                              |                                          | The purifying agents i.e., resins, adsorbents, zeolites, etc., can be regenerated and reused | Cannot completely remove methanol or glycerol in one stage of process                  |
|                              |                                          | Eco-friendly method                                                 | Needing further wet washing and drying                                               |
|                              |                                          |                                                                      | The complexation agent i.e., EDTA has a high toxicity                                |
|                              |                                          |                                                                      | A very time requiring process                                                        |
|                              |                                          |                                                                      | Generate wastewater similarly to the wet washing method                               |
| EDTA complexation            | Feedstock: curcas oil                    | Calcium removal ability is remarkable                               | The purified biodiesel has a higher value of viscosity, making it unfitted the biodiesel standard |
|                              | Purification: complexation               | Ca-EDTA complex is water soluble                                    | Require long time with maintained operating temperature, pressure, and agitation.    |
|                              | Ester yield: 98%                         | The Ca-EDTA complex can be removed using centrifugation             | Priorly, the calcium content in the crude biodiesel should be measured                |
|                              | Ref: (Zhu et al., 2006)                  |                                                                      | Additional purification is required to remove soap, alcohol, and glycerol            |
| Precipitation                | Feedstock: curcas oil                    | Citric acid can be utilized to precipitate the calcium ions in the crude biodiesel (yield >95%) | The membrane should be periodically checked, cleaned, or changed                     |
|                              | Purification: citric acid precipitation  | High product yield                                                 | Gradual flux reduction over time due to natural fouling on the membrane               |
|                              | Ester yield: 98%                         | The residue/precipitate can be simply separate using a conventional filtration |                                                                        |
|                              | Ref: (Vieira et al., 2017)               | The precipitate Ca-citrate can be regenerated back into citric acid by the addition of strong acid |                                                                        |
| Membrane filtration         | Feedstock: Waste cooking oil             | Simple and flexible in operation                                     | Less effective in ion removal                                                        |
|                              | Purification: membrane filtration        | Require low energy                                                 | The membrane should be periodically checked, cleaned, or changed                     |
|                              | Ester yield: 5.6%                        | High product flux and selectivity                                   | Gradual flux reduction over time due to natural fouling on the membrane               |
|                              | Ref: (Bansod et al., 2021)               | Can be engineered for the special uses                              |                                                                        |
|                              |                                          | Relatively easier to separate higher size such as, soap and glycerol. |                                                                        |
|                              |                                          | Ease to scale-up                                                    |                                                                        |
these advanced processes is the high operating cost and a higher risk compared to the conventional process. Furthermore, the types of catalysts play an essential part in biodiesel synthesis because catalysts with different sources, phases, chemicals, and sizes have been applied to achieve better biodiesel quality and yield. Homogeneous and heterogeneous catalysts can achieve at least 80% to 95% biodiesel conversion. Nano-catalysts are the nanoparticle form of heterogeneous catalyst that showed a better catalytic performance due to their large active surface area. The enzymatic catalysts are eco-friendly with a variety of conversions from 70 to 99%. Moreover, the effectiveness of the separation process in biodiesel purification depends on the type of catalyst applied in the synthesis process. In certain cases, a simple wet washing can lead to the biodiesel product meeting the specification standard. Some efforts using several dry methods such as adsorption, cation- and anion-exchange resins, precipitation, complexation, and membrane-based separation have shown a great performance in calcium ions removal. However, some drawbacks of those methods include the complex process from upstream until downstream that needed to be simplified. Therefore, further research on this topic is necessary, especially on the reusability of the catalysts and purifying agents. This development will not only contribute to the intensification of biodiesel production and purification but also maximize the economic impact and minimize the environmental hazard of biodiesel.

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Author Contributions
Silviana: Conceptualization, methodology, writing—original draft., Didi Dwi Anggoro: Validation, resources. Hadiyanto: Writing—review and editing, supervision, Cantika Aulia. Salsabila: Writing—review and editing, and validation., Kevin Aprilio: Formal analysis, and resources, Anisa Widia Utami: Formal analysis, and project administration, Afriza N’imatus Sa’adah: Formal analysis, and resources., Febio Dalanta: Visualization, Literature review, Validation, and writing—original draft - review and editing. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest
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