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Recent Trends in Biodiesel Production Techniques: A Review

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***Abstract*— Biodiesel has emerged as a renewable and environmentally friendly alternative to traditional fossil fuels, attracting considerable interest for its ability to meet growing energy needs while mitigating environmental impacts. This review focuses on contemporary advancements in biodiesel production techniques, highlighting innovative methods aimed at improving efficiency, cost-effectiveness, and sustainability. Non-edible oils like pine oil and soapnut oil have gained prominence as viable feedstocks, offering the advantage of avoiding competition with food supplies. Cutting-edge catalytic systems, including heterogeneous catalysts, nano-catalysts, and enzyme-based approaches, have brought significant improvements to the transesterification process by ensuring higher yields and greater stability. Novel technologies, such as ultrasound-assisted and microwave-assisted transesterification, are recognized for their capacity to reduce both reaction duration and energy usage. Optimization tools like Response Surface Methodology (RSM), combined with the use of co-solvents and additives, play a key role in enhancing biodiesel production quality and efficiency. The review further explores challenges related to feedstock availability, production costs, and scalability, while proposing solutions such as genetically engineered feedstocks and the integration of biodiesel production into biorefineries. By emphasizing recent technological innovations, this study highlights the transformative potential of modern biodiesel production techniques to support a sustainable and environmentally conscious energy future.*

I. INTRODUCTION

The rapid depletion of fossil fuel reserves and escalating global energy demand have pushed researchers to explore renewable and sustainable energy sources. Among these, biodiesel has emerged as a highly promising alternative to conventional diesel fuels, owing to its biodegradability, renewability, and capacity to reduce greenhouse gas emissions [1]. Biodiesel is produced through the transesterification of vegetable oils, animal fats, or non-edible oils, resulting in fatty acid methyl esters (FAME), which are compatible with diesel engines without significant modifications [2]. Biodiesel addresses two critical global challenges, environmental sustainability and energy security.

Its ability to reduce harmful emissions, including carbon dioxide (CO_2), sulfur oxides (SO_x), and particulates, makes it a cleaner-burning fuel compared to petroleum diesel. Additionally, biodiesel production utilizes renewable feedstocks, reducing reliance on finite fossil resources [3]. These attributes have made biodiesel a focal point of energy research and policy-making worldwide. Feedstocks for biodiesel production can be classified into three categories, edible oils, non-edible oils, and waste oils. Edible oils, such as soybean, palm, and sunflower oil, have been widely used for biodiesel production [4]. However, the reliance on edible oils has raised ethical concerns regarding food security and price volatility. Non-edible oils, including jatropha, karanja, neem, pine oil, and soapnut oil, have emerged as alternative feedstocks, addressing these challenges while utilizing marginal lands unsuitable for food crops [5]. Moreover, waste oils, such as used cooking oil and animal fats, offer an economical and environmentally friendly option for biodiesel production [6]. The transesterification process is central to biodiesel production, involving the reaction of triglycerides with alcohol (methanol or ethanol) in the presence of a catalyst. Recent advancements have focused on improving the efficiency, yield, and sustainability of this process. The introduction of heterogeneous catalysts, nano-catalysts, and enzyme-based catalysts has significantly enhanced reaction kinetics and product quality [7]. Additionally, emerging technologies, such as ultrasound-assisted transesterification and microwave-assisted transesterification, have revolutionized biodiesel production by reducing reaction time and energy consumption. These methods have demonstrated higher process efficiency, particularly when combined with advanced catalytic systems [8]. The selection of suitable feedstocks has been a critical focus in advancing biodiesel production. Recent studies highlight the utilization of microalgae, such as Scenedesmus species, for their high lipid content and rapid growth rates, offering a sustainable alternative to edible oils. However, challenges like nutrient supply, harvesting, and processing costs must be addressed to realize their full potential [9].

Additionally, exploring unconventional feedstocks, such as agricultural residues and animal fats, provides cost-effective and environmentally friendly options. Catalysts play a crucial role in the transesterification process. Heterogeneous catalysts are gaining attention due to their reusability and reduced environmental impact compared to traditional homogeneous catalysts. Recent advancements include the development of calcium oxide-based nano-catalysts, which enhance reaction efficiency and reduce energy consumption. These catalysts offer a pathway to achieving higher yields with lower environmental footprints [10]. Emerging technologies like microwave-assisted transesterification are transforming biodiesel production. This method significantly reduces reaction times and energy requirements, particularly for feedstocks with high free fatty acid content. Ultrasound-assisted transesterification is another innovation that improves reaction kinetics and biodiesel yields by enhancing the mixing of reactants. Furthermore, mobile biodiesel production units have been introduced for small-scale applications, enabling on-site processing and reducing logistical costs [11]. Sustainability has become a cornerstone in biodiesel advancements. The integration of waste feedstocks, such as used cooking oils and animal fats, addresses waste management challenges while reducing production costs [12]. For example, large-scale initiatives in Brazil utilize animal fats for biodiesel production, contributing to a circular economy and lowering greenhouse gas emissions. Despite the advancements, the biodiesel industry faces several challenges. Feedstock availability remains a major bottleneck, with the need to balance agricultural land use for food and energy production [13]. Production costs are also high, primarily due to feedstock expenses and the energy-intensive nature of the transesterification process. Scalability and integration with existing fuel infrastructure pose additional hurdles for large-scale biodiesel adoption [14]. Ongoing research aims to overcome these challenges by exploring genetically modified feedstocks with higher oil yields and reduced cultivation requirements. Integrating biodiesel production with biorefineries can further improve economic viability by producing value-added co-products [15]. Moreover, policy-driven incentives, including subsidies and carbon credits, can enhance biodiesel's competitiveness in the energy market. This review provides a comprehensive analysis of recent trends and advancements in biodiesel production techniques. It highlights innovative approaches, such as the use of non-edible oils, advanced catalysts, and emerging technologies, while addressing existing challenges and proposing future directions for sustainable biodiesel production [16].

II. LITERATURE REVIEW

Biodiesel, derived from renewable biological resources, has emerged as a promising alternative to conventional diesel fuel due to its environmental benefits and biodegradability. The production techniques for biodiesel have evolved significantly, aiming for higher efficiency and sustainability. Traditional methods, such as transesterification using chemical catalysts, dominate the industry. However, advancements in enzymatic catalysis, microwave-assisted synthesis, and nano-catalyst applications have led to increased yields and reduced reaction times [17]. The choice of technique often depends on the feedstock used, as different oils exhibit unique chemical compositions. Studies have explored the potential of using non-edible oils like pine oil and soapnut oil due to their abundance and minimal competition with food resources. Additionally, hybrid approaches combining traditional and advanced methods have shown superior performance in achieving high-purity biodiesel with fewer by-products [18]. Government policies and regulations, such as those set by ASTM D6751 and EN 14214 standards, play a crucial role in shaping the methodologies for biodiesel production. These frameworks ensure that biodiesel meets performance and emission standards, making it a viable alternative to fossil fuels [19].

The transesterification process is the most widely used method for biodiesel production. It involves the reaction of triglycerides in oils or fats with an alcohol, typically methanol or ethanol, in the presence of a catalyst to produce fatty acid methyl esters (biodiesel) and glycerol as a by-product. This process is favored due to its simplicity, cost-effectiveness, and high conversion rates [20]. Homogeneous catalysts, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), are commonly used in transesterification due to their high efficiency. However, these catalysts lead to soap formation when water or free fatty acids are present, posing challenges in downstream separation processes [21]. To overcome these limitations, heterogeneous catalysts have been developed, offering advantages such as reusability and simplified purification of biodiesel. Enzymatic catalysts, though less widely used due to high costs, have gained attention for their eco-friendly nature and ability to operate under mild conditions. Advanced methods, such as microwave-assisted and ultrasonic-assisted transesterification, have further improved reaction kinetics, reducing energy consumption and increasing biodiesel yields [22]. The process parameters, including temperature, alcohol-to-oil molar ratio, catalyst concentration, and reaction time, significantly influence the efficiency of transesterification. Optimizing these parameters through statistical methods such as Response Surface Methodology (RSM) has proven effective in achieving maximum biodiesel yield [23]. Nano-catalysts have significantly advanced biodiesel production due to their high surface area and enhanced reactivity. Research indicates that nano-catalysts such as Al_2O_3, CeO_2, and TiO_2 improve transesterification efficiency, reduce reaction time, and operate under milder conditions [24].

The choice of feedstock significantly affects the biodiesel production process's economic and environmental aspects. Feedstocks are generally categorized into three types: edible oils (such as soybean and sunflower oil), non-edible oils (such as soapnut and pine oil), and waste oils (such as used cooking oils). Non-edible oils are preferred for large-scale production due to their availability, cost-effectiveness, and minimal competition with food resources [25]. Soapnut oil, extracted from *Sapindus mukorossi*, is a promising feedstock due to its high oil content and unique fatty acid profile. Research highlights its potential for biodiesel production with improved cetane number and oxidation stability. Similarly, pine oil has been studied for its low sulfur content and high combustion efficiency, making it a suitable alternative to conventional feedstocks [26]. Studies emphasize the importance of evaluating feedstock properties such as viscosity, density, and free fatty acid content. These parameters directly influence the transesterification process and the quality of the biodiesel produced. Moreover, exploring feedstocks like soapnut and pine oil can mitigate environmental concerns associated with deforestation and unsustainable agricultural practices [27]. Optimization of the biodiesel production process is critical to achieving high yields and cost-efficiency. Parameters such as reaction temperature, alcohol-to-oil molar ratio, catalyst concentration, and reaction time significantly influence the transesterification process. Response Surface Methodology (RSM) and Central Composite Design (CCD) are widely used statistical tools to optimize these parameters [28]. For instance, RSM enables the identification of interaction effects between variables and determines the optimum operating conditions for maximum biodiesel yield. Studies on soapnut oil biodiesel have demonstrated that optimization using RSM can achieve yields above 90% with reduced catalyst usage and reaction times [29]. CCD, a robust optimization technique, has been employed to study biodiesel production from pine oil. Research has shown that this approach minimizes experimental efforts while accurately predicting optimal conditions for high-quality biodiesel production. Furthermore, the inclusion of additives, such as nano-catalysts, in the optimization framework has enhanced the efficiency of the process [30].

III. RECENT TRENDS IN BIODIESEL PRODUCTION TECHNIQUES

A. Advancements in Catalytic Technologies

The catalytic process in biodiesel production has undergone significant advancements to enhance efficiency, cost-effectiveness, and environmental sustainability. Catalysts play a vital role in determining reaction rates, yields, and product quality in the transesterification process. Recent trends focus on the development of advanced catalytic technologies to address the challenges associated with traditional methods, such as high energy requirements, waste generation, and low reaction efficiency. Nano-catalysts have revolutionized biodiesel production due to their exceptional properties, including high surface area, enhanced reactivity, and reusability. Nano-catalysts like titanium dioxide (TiO_2), aluminum oxide (Al_2O_3), and cerium oxide (CeO_2) have been widely investigated for their ability to improve reaction kinetics and achieve higher biodiesel yields under milder conditions. These catalysts reduce the activation energy required for transesterification and enable faster reaction rates, making the process more energy-efficient [31]. Additionally, nano-catalysts are environmentally friendly as they minimize waste generation and can be easily separated and reused in subsequent cycles [32]. Studies have demonstrated that TiO_2 nanoparticles significantly enhance biodiesel yield from non-edible oils, including soapnut oil, while operating at lower temperatures and pressures. The use of CeO_2 nano-catalysts in combination with microwave-assisted transesterification has further reduced reaction times, indicating their potential for large-scale industrial applications [33].

Heterogeneous catalysts have emerged as a superior alternative to homogeneous catalysts due to their ease of separation, reusability, and reduced environmental impact. Solid acid and base catalysts, such as calcium oxide (CaO) and magnesium oxide (MgO), have gained prominence in biodiesel production, especially for feedstocks with high free fatty acid (FFA) content. Unlike homogeneous catalysts, which lead to soap formation and require extensive purification, heterogeneous catalysts offer a simplified process with higher biodiesel purity [34]. Recent advancements in heterogeneous catalysis include the development of bifunctional catalysts that combine acid and base sites. These catalysts facilitate simultaneous esterification and transesterification, making them highly efficient for low-quality feedstocks. For instance, the use of bifunctional catalysts in soapnut oil biodiesel production has shown improved yield and reduced by-products [35]. Enzymatic catalysts, particularly lipases, have garnered attention for their eco-friendly nature and ability to operate under mild reaction conditions. Unlike chemical catalysts, lipases are highly specific and do not produce undesirable by-products. However, their high cost and sensitivity to reaction conditions have limited their widespread adoption. To address these challenges, researchers have focused on immobilization techniques that enhance the stability and reusability of lipase enzymes [36]. Lipase-based transesterification has been successfully employed for biodiesel production from high FFA oils, such as waste cooking oil and soapnut oil. Immobilized lipases not only improve process economics but also enable continuous biodiesel production, making them suitable for industrial applications [37]. Dual catalysis systems, which combine the benefits of homogeneous and heterogeneous catalysts, represent a novel approach to improving biodiesel production efficiency. These systems utilize the strengths of both catalyst types, such as the high activity of homogeneous catalysts and the reusability of heterogeneous catalysts. Research on dual catalysis has demonstrated increased reaction rates and higher biodiesel yields, even with challenging feedstocks like soapnut oil [38]. In addition, the integration of nano-catalysts with dual catalytic systems has further enhanced the catalytic performance. For example, combining Al_2O_3 nano-catalysts with a solid acid-base catalyst has achieved remarkable results in biodiesel yield and process efficiency.

B. Non-Edible and Waste Feedstocks

The growing demand for biodiesel production has highlighted the need for sustainable and cost-effective feedstocks. While edible oils such as soybean and palm oil have been traditionally used, their competition with food resources poses a significant challenge. Consequently, the focus has shifted towards non-edible oils and waste feedstocks as viable alternatives. These options provide economic and environmental benefits by utilizing underexploited resources and reducing waste. Non-edible oils, including soapnut oil (*Sapindus mukorossi*) and pine oil, have gained prominence in recent years due to their abundance and non-competitiveness with food crops. Soapnut oil, with a high saponin content, has been recognized for its potential as a biodiesel feedstock. Research shows that soapnut oil biodiesel exhibits excellent fuel properties, such as high cetane number and low sulfur content, making it suitable for combustion engines [39]. Similarly, pine oil, derived from the distillation of pine resins, has been identified as an effective feedstock due to its low viscosity and high volatility. Pine oil blends have demonstrated improved performance and emission characteristics in diesel engines, positioning it as a promising candidate for large-scale biodiesel production [40]. Waste cooking oils and animal fats are gaining popularity as sustainable feedstocks for biodiesel production. These waste materials not only reduce feedstock costs but also address environmental concerns associated with their disposal. Studies indicate that biodiesel derived from waste cooking oils exhibits comparable properties to biodiesel from conventional feedstocks, such as similar energy content and combustion efficiency [41]. A comparative analysis of non-edible oils and waste oils reveals that both options have their unique advantages. Non-edible oils provide a steady supply chain for biodiesel production, while waste oils offer significant cost savings and environmental benefits. The selection of feedstocks often depends on regional availability and economic considerations [42].

C. Emerging Process Techniques

Innovative process techniques in biodiesel production have emerged to address the limitations of conventional methods, such as high energy requirements, long reaction times, and environmental concerns. These advancements leverage modern technologies to improve efficiency, reduce costs, and enhance the sustainability of biodiesel production. Microwave-assisted transesterification has gained attention as a highly efficient technique for biodiesel production. Unlike conventional heating, microwaves deliver energy directly to the reactants, ensuring uniform heating and faster reaction rates. This method significantly reduces reaction time and energy consumption while achieving high biodiesel yields [43]. Studies have demonstrated that microwave-assisted transesterification of non-edible oils, such as soapnut oil, results in yields exceeding 95% within minutes, making it a viable option for industrial-scale production [44].

Ultrasonic waves create cavitation effects in the reaction mixture, generating localized high temperatures and pressures that enhance the transesterification process. Ultrasonic-assisted methods are particularly effective for feedstocks with high free fatty acid (FFA) content, as they improve catalyst dispersion and mass transfer [45]. Research indicates that ultrasonic-assisted transesterification reduces the need for excess alcohol and catalyst, making it a cost-effective alternative for biodiesel production from waste oils [46]. Supercritical fluid technology involves the use of alcohols like methanol or ethanol at supercritical conditions (high temperature and pressure) as both reactant and catalyst. This technique eliminates the need for chemical catalysts, thereby avoiding issues related to catalyst recovery and by-product formation. Although energy-intensive, advancements in reactor design and process optimization have made this method increasingly attractive for large-scale operations. Supercritical methods have been successfully applied to a wide range of feedstocks, including non-edible and waste oils, yielding high-purity biodiesel [47]. Hydrodynamic cavitation utilizes pressure changes in the fluid to create vapor bubbles, which collapse and generate localized high temperatures. This process enhances the mixing of reactants and accelerates the transesterification reaction. Hydrodynamic cavitation has been shown to achieve comparable yields to ultrasonic methods while consuming less energy [48]. Its scalability and cost-effectiveness make it a promising technology for industrial biodiesel production. Hybrid approaches that combine two or more advanced techniques are emerging as the next frontier in biodiesel production. For instance, integrating microwave and ultrasonic technologies has demonstrated synergistic effects, further improving reaction efficiency and biodiesel yield. These hybrid methods optimize resource utilization and minimize environmental impact, aligning with the goals of sustainable biodiesel production [49].

D. Transesterification Process

The transesterification process is the fundamental chemical reaction behind biodiesel production. It involves converting triglycerides, which are esters of glycerol and fatty acids, into biodiesel (fatty acid methyl esters) and glycerol. This process significantly reduces the viscosity of raw oils, making them compatible for use in diesel engines.

The overall reaction can be expressed as:

Triglycerides + 3 Alcohol → 3 Biodiesel (Fatty Acid Esters) + Glycerol

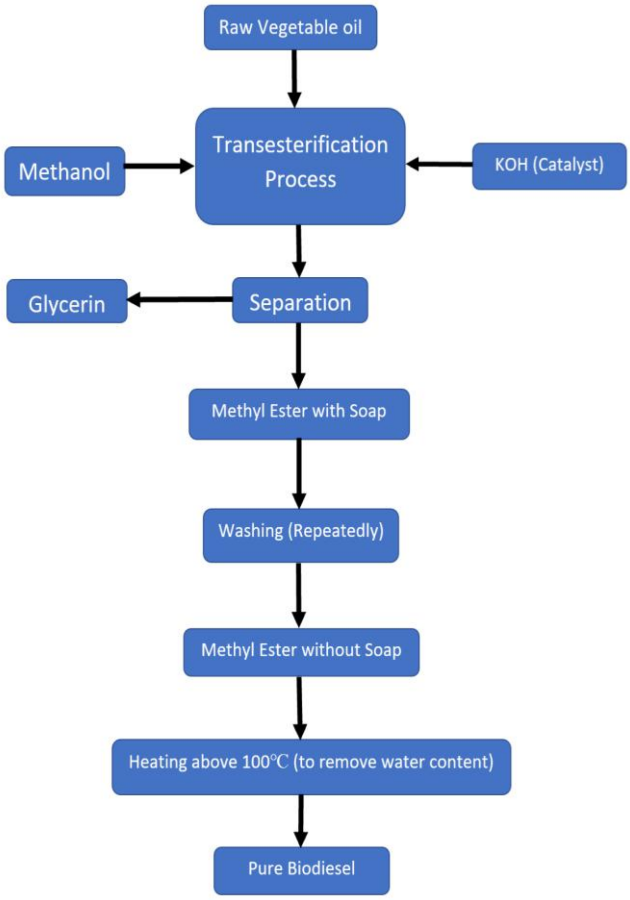

Fig. 1: *Schematic diagram of transesterification process*

This reversible reaction typically requires an excess of alcohol (commonly methanol) to drive the equilibrium toward the production of biodiesel and achieve high yields. Catalysts play a crucial role in the transesterification process, with the most commonly used types being homogeneous, heterogeneous, and biological catalysts. Homogeneous catalysts like sodium hydroxide (NaOH), potassium hydroxide (KOH), and sulfuric acid (H_2SO_4) dissolve entirely in the reaction medium, resulting in high reaction rates. The detailed schematic process of transesterification process as shown in figure 1. Alkali Catalysts (NaOH, KOH) are highly efficient for feedstocks with low free fatty acid (FFA) content. However, they are sensitive to water content, which can cause soap formation, reducing yield and complicating purification. Acid Catalysts (H_2SO_4, HCl) are suitable for feedstocks with high FFA content due to their tolerance to impurities. However, they are slower and require more energy compared to alkali catalysts. Solid catalysts such as calcium oxide (CaO) and magnesium oxide (MgO) offer several advantages, including reusability, ease of separation, and environmental friendliness. These catalysts eliminate the need for extensive purification steps, reducing overall production costs. Lipases, which are biological enzymes, function under mild conditions and exhibit high specificity to the transesterification reaction. However, their widespread application is limited by high costs and sensitivity to impurities. Immobilized lipases have shown potential for industrial applications, as they can be reused and provide improved economic feasibility.

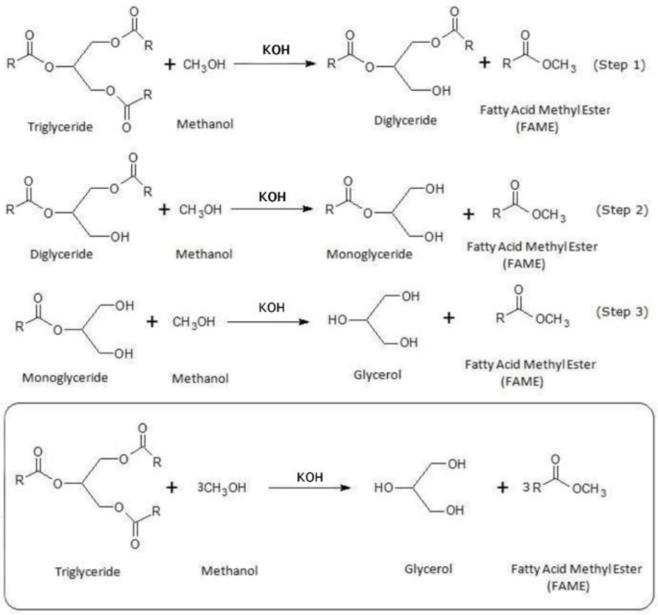

Fig. 2: Hydrocarbon chain reaction

1) Hydrocarbon Chain Reaction in Transesterification

The hydrocarbon chain reaction during transesterification involves the stepwise conversion of triglycerides into diglycerides, monoglycerides, and finally glycerol, while releasing fatty acid esters (biodiesel) at each step. A visual representation of this process is shown in Fig. 2, which details the reaction pathways: Triglycerides react with methanol (CH_3OH) in the presence of KOH, producing diglycerides and a fatty acid ester. Diglycerides then react with additional methanol, forming monoglycerides and another fatty acid ester. Monoglycerides undergo the final reaction with methanol, yielding glycerol and the third fatty acid ester.

IV. VARIABLES AFFECTING TRANSESTERIFICATION REACTION

The transesterification process is influenced by several critical variables, which directly impact biodiesel yield, quality, and process efficiency. Understanding and optimizing these parameters are essential for achieving economical and sustainable biodiesel production.

A. Effect of free fatty acid and moisture

Free fatty acids (FFAs) and moisture in feedstocks pose significant challenges to the transesterification process, affecting biodiesel yield and quality. FFAs react with alkaline catalysts to form soap, which not only reduces biodiesel yield but also complicates the separation of biodiesel from glycerol [50]. Moisture exacerbates the problem by hydrolyzing triglycerides into FFAs, further reducing the efficiency of the reaction. The combined presence of FFAs and moisture can render conventional alkaline transesterification ineffective. Pre-treatment techniques are critical for overcoming these challenges. Acid esterification is one of the most commonly employed methods for reducing FFA content, converting FFAs into esters, which are less reactive with alkaline catalysts [51]. Adsorption methods, such as using silica gel and molecular sieves, effectively reduce moisture content in feedstocks, improving reaction efficiency. Advanced processes like enzymatic pre-treatment and ultrasonic-assisted drying have also demonstrated significant potential in reducing both FFA and moisture levels [52]. High FFA feedstocks, such as waste cooking oil and animal fats, often require multiple pre-treatment steps to achieve acceptable FFA and moisture levels before transesterification. Techniques like combined esterification and bleaching have been proposed to address these issues efficiently. Furthermore, recent research highlights the use of heterogeneous acid catalysts, which are less sensitive to FFAs and can simultaneously catalyze esterification and transesterification reactions [53]. The optimization of these pre-treatment techniques is critical for improving biodiesel yield and reducing process costs. Studies using advanced modeling techniques, such as response surface methodology (RSM), have demonstrated the potential for optimizing these processes to handle high FFA feedstocks effectively [54].

B. Catalyst type and concentration

Catalyst selection and concentration are critical factors in the transesterification process, directly influencing reaction efficiency, biodiesel yield, and cost-effectiveness. Homogeneous catalysts, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), are extensively used in commercial biodiesel production due to their high reactivity, availability, and cost-efficiency. However, these catalysts are highly sensitive to free fatty acids (FFAs) in the feedstock, leading to soap formation that complicates product separation and reduces biodiesel yield [55]. Heterogeneous catalysts, including calcium oxide (CaO), zinc oxide (ZnO), and zeolites, have garnered increasing attention for their reusability, ease of separation from reaction mixtures, and low environmental impact [56]. These catalysts are particularly suitable for feedstocks with high FFA levels, as they minimize soap formation. Additionally, they can simultaneously catalyze esterification and transesterification reactions, making them advantageous for low-quality feedstocks. Enzymatic catalysts, primarily lipases, offer an eco-friendly alternative. They are highly tolerant to FFA-rich feedstocks, eliminating the need for pre-treatment processes [57]. However, the slower reaction rates, higher costs, and shorter lifespan of enzymes compared to chemical catalysts present significant challenges for large-scale applications. Recent advancements in immobilization techniques for lipases have enhanced their reusability and reduced overall production costs [58]. Catalyst concentration also plays a vital role in determining reaction efficiency. While low catalyst concentrations may lead to incomplete conversion, excessively high concentrations can cause soap formation and emulsification, increasing purification costs. Optimization of catalyst type and concentration is, therefore, essential for achieving high biodiesel yield and quality while minimizing production costs and environmental impact [59].

C. Molar ratio of alcohol to oil and type of alcohol

The molar ratio of alcohol to oil is a critical factor that significantly impacts the transesterification process, influencing both biodiesel yield and reaction efficiency. An optimal molar ratio ensures complete conversion of triglycerides into fatty acid methyl esters (FAMEs). A higher alcohol-to-oil molar ratio shifts the equilibrium towards product formation, increasing biodiesel yield, but it also elevates recovery costs due to the excess alcohol that must be removed and recycled [80]. Methanol is the most widely used alcohol in biodiesel production due to its low cost, high reactivity, and availability. It is particularly suited for base-catalyzed transesterification, forming a homogeneous mixture with triglycerides and catalysts [60]. Ethanol, though less commonly used, is considered a viable alternative, especially in regions where it is more accessible and cost-effective. Ethanol produces biodiesel with slightly improved cold flow properties but poses challenges due to its partial immiscibility with certain feedstocks and its higher propensity for forming water during the reaction. Other alcohols, such as butanol and isopropanol, are under investigation for their potential to enhance the biodiesel production process. Butanol, in particular, offers advantages such as reduced soap formation, better miscibility with oils, and the production of biodiesel with superior properties [61]. However, the cost and availability of these alcohols currently limit their widespread application. The selection of the alcohol type and its molar ratio depends on the feedstock, catalyst, and desired biodiesel properties. Studies suggest that a molar ratio of 6:1 for methanol and 9:1 for ethanol generally yields optimal results. Further research into advanced alcohols and innovative techniques, such as the use of co-solvents, continues to refine the efficiency and sustainability of the process [62].

D. Effect of reaction time and temperature

Reaction time and temperature are critical factors that significantly influence the efficiency and yield of the transesterification process. Reaction temperature affects the kinetic energy of molecules, enhancing molecular collisions and promoting faster reaction rates. Higher temperatures generally accelerate the conversion of triglycerides to biodiesel, reducing reaction time and improving efficiency [63]. Optimal temperatures for biodiesel production are typically close to the boiling point of the alcohol used, such as 60–65°C for methanol. However, excessively high temperatures can lead to undesirable side reactions, such as thermal degradation of biodiesel or increased soap formation, particularly when free fatty acids (FFAs) are present in the feedstock [64]. Such side reactions can reduce product yield and increase the complexity of the separation process. Similarly, while longer reaction times ensure complete conversion of triglycerides into biodiesel, excessively prolonged durations can lead to increased operational costs and energy consumption, as well as the potential for emulsification in alkaline-catalyzed reactions. The determination of optimal reaction time and temperature depends on the type of catalyst, feedstock, and alcohol used. Studies suggest that for most homogeneous alkaline-catalyzed reactions, a reaction time of 1–2 hours at a temperature of 60°C yields high conversion rates [65]. Heterogeneous catalysts often require slightly longer reaction times and higher temperatures due to their lower activity compared to homogeneous catalysts. Enzymatic catalysts operate efficiently at lower temperatures (30–

40°C) but require longer reaction times, making them less cost-effective for large-scale applications. Innovative techniques such as microwave-assisted and ultrasonic-assisted transesterification have shown promise in reducing reaction times while maintaining high biodiesel yields. These methods leverage energy-efficient heating and agitation mechanisms to enhance reaction kinetics, making the process more economical and environmentally friendly [66].

V. ANALYTICAL METHODS

The analysis of biodiesel and its feedstocks involves various sophisticated analytical techniques. These methods provide critical insights into the composition, properties, and performance characteristics of biodiesel, aiding in quality assurance and process optimization. This chapter explores key analytical methods used in biodiesel research.

A. Gas chromatography – Mass spectrometry (GC-MS)

Gas Chromatography–Mass Spectrometry (GC-MS) is an indispensable analytical technique in biodiesel production, widely used to identify and quantify fatty acid methyl esters (FAMEs), the primary constituents of biodiesel. This method combines the high-resolution separation capability of gas chromatography with the precise molecular identification offered by mass spectrometry, enabling highly sensitive and accurate analyses [67]. The process involves injecting a biodiesel sample into a chromatographic column, where the FAMEs are separated based on their boiling points and volatility. The separated components are subsequently ionized in the mass spectrometer, producing unique mass spectra that allow for the identification and quantification of individual compounds. GC-MS is particularly effective in detecting impurities, such as unreacted triglycerides, monoglycerides, diglycerides, and residual alcohols, ensuring compliance with stringent biodiesel quality standards, including ASTM D6751 and EN 14214 [68]. Recent advancements in GC-MS techniques have significantly enhanced its utility and efficiency. Innovations such as headspace GC-MS simplify the analysis of volatile compounds, while tandem MS (MS/MS) improves the resolution and sensitivity for complex mixtures. The technique is also employed in studying biodiesel degradation during storage, providing insights into the formation of oxidation products and polymerized compounds [69]. Moreover, GC-MS is utilized for compositional analysis of feedstocks and for evaluating the effect of additives on biodiesel properties. Techniques like pyrolysis-GC-MS have been used to analyze thermally degraded biodiesel samples, offering valuable information about its thermal stability. The development of portable GC-MS systems has further expanded its application in on-site biodiesel quality assessment [70].

B. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a vital analytical tool in biodiesel research, used to identify functional groups in biodiesel molecules through their infrared absorption spectra. This technique is instrumental in confirming the conversion of triglycerides into fatty acid methyl esters (FAMEs) by detecting the characteristic ester functional groups while monitoring the reduction of hydroxyl and carbonyl groups [71]. FTIR operates as a non-destructive, rapid, and cost-effective method, making it suitable for real-time monitoring of the transesterification process. Its applications extend beyond conversion confirmation to include the assessment of biodiesel's oxidative stability and degradation during storage. By measuring the formation of oxidation products such as aldehydes, ketones, and acids, FTIR aids in understanding biodiesel's long-term performance. Advanced FTIR techniques, such as Attenuated Total Reflectance (ATR-FTIR), have enhanced the analysis of biodiesel by improving sensitivity and eliminating complex sample preparation steps [72]. This approach is particularly advantageous for studying the molecular interactions of biodiesel blends and additives. Recent developments in two-dimensional correlation spectroscopy (2D-FTIR) have further expanded its applicability, allowing for the detailed analysis of biodiesel's thermal and oxidative behavior [114]. FTIR is also employed in monitoring biodiesel standards compliance, including ASTM D6751 and EN 14214, by assessing the presence of impurities and unreacted feedstock components. Studies have shown that FTIR combined with chemometric techniques such as Principal Component Analysis (PCA) and Partial Least Squares Regression (PLSR) offers enhanced accuracy in predicting biodiesel properties [73].

C. Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) is a widely utilized analytical technique in biodiesel research for evaluating changes in the mass of a sample as a function of temperature or time. This method is instrumental in studying the thermal stability, decomposition characteristics, and combustion behavior of biodiesel and its feedstocks. TGA provides critical data on the energy content and residue formation, aiding in understanding biodiesel's performance under varying thermal conditions [74]. During TGA analysis, the biodiesel sample is subjected to a controlled heating rate, and mass loss is recorded. This information reveals the temperature ranges at which volatile compounds are released and non-volatile residues decompose. Such insights are valuable for optimizing biodiesel formulations and improving its thermal stability [74]. TGA has also been used to evaluate the effects of additives on biodiesel combustion properties and to study the thermal degradation of feedstocks such as vegetable oils and animal fats [75]. Advanced techniques like coupled TGA-FTIR and TGA-MS provide additional insights by identifying the chemical composition of volatile products released during the heating process. These methods enhance understanding of biodiesel oxidation mechanisms and the formation of degradation products, such as aldehydes and ketones [75]. TGA is also employed in comparing biodiesel blends, helping researchers determine the impact of blending ratios on thermal stability and energy content. Recent studies have applied TGA in assessing the thermal behavior of biodiesel derived from waste cooking oil, jatropha oil, and microalgae feedstocks, highlighting its versatility across different biodiesel types. With the integration of advanced computational models and machine learning algorithms, TGA data can now be used to predict biodiesel properties with greater accuracy, supporting efforts to improve biodiesel production and storage stability.

D. Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Analysis (EDAX)

Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Analysis (EDAX) is an advanced analytical technique extensively used in biodiesel research to investigate the surface morphology and elemental composition of biodiesel feedstocks, additives, and catalysts. SEM generates high-resolution images of the sample's surface, revealing structural details such as porosity, particle size, and surface defects [76]. EDAX complements SEM by providing quantitative and qualitative elemental analysis, enabling the identification of elemental distributions across the sample. This technique is particularly valuable for characterizing heterogeneous catalysts used in biodiesel production, such as calcium oxide, zinc oxide, and zeolites. SEM analysis reveals surface roughness, cracks, and other structural features, while EDAX helps evaluate the distribution of active sites and confirm the presence of catalytic elements. These insights are critical for understanding catalyst performance and improving catalyst designs for enhanced biodiesel yield and quality [75].

SEM with EDAX is also used to analyze the ash content and contaminants in biodiesel samples, which can arise from feedstock impurities or incomplete reactions. This information aids in optimizing purification steps and ensuring compliance with biodiesel standards like ASTM D6751 and EN 14214 [74]. Additionally, the technique has been applied to study the effects of additives, such as nano-catalysts, on the structural and compositional changes during the transesterification process. Recent advancements in SEM technology, such as environmental SEM (ESEM), allow for the analysis of moist or liquid samples without extensive preparation, making it more versatile for biodiesel applications. Coupled techniques like SEM-TGA and SEM-FTIR further expand its analytical capabilities, enabling simultaneous structural and compositional analysis under thermal conditions [76].

VI. IMPACT OF FEEDSTOCK QUALITY ON BIODIESEL PROPERTIES

The quality of feedstock, particularly its free fatty acid (FFA) content and fatty acid composition, plays a critical role in determining the properties of biodiesel. A high FFA content in feedstock often results in reduced biodiesel yields when employing conventional base-catalyzed transesterification due to soap formation, which complicates the separation process. Furthermore, the fatty acid composition influences key parameters such as cetane number, cold flow properties, flash point, and oxidative stability [77]. Feedstocks rich in saturated fatty acids, such as palm oil, exhibit higher cetane numbers due to the abundance of palmitic acid (C16:0) and stearic acid (C18:0). Conversely, oils containing unsaturated fatty acids, including soybean, sunflower, and grapeseed oils, are associated with lower cetane numbers.

Additionally, the cloud point of biodiesel synthesized from oils like soybean and corn is lower, typically near or below 0°C, owing to negligible amounts of saturated fatty acids. In contrast, tallow-based biodiesel has a higher cloud point due to the presence of a substantial fraction of saturated fatty acids [78]. Biodiesel generally has a high flash point, often exceeding 150°C, which is crucial for safe storage and handling. Oils with shorter carbon chain lengths exhibit relatively lower flash points. Oxidative stability is another vital property influenced by the degree of unsaturation; oils such as palm and olive oils, rich in saturated fatty acids, demonstrate enhanced oxidative stability. On the other hand, unsaturated oils are more prone to degradation over time [79]. Viscosity, a fundamental parameter, increases with the chain length of fatty acids and their degree of saturation. For instance, oils containing higher levels of saturated or trans-fatty acids, such as castor oil, exhibit elevated viscosity. Interestingly, configurations such as cis double bonds result in lower viscosity compared to trans double bonds. Branching and the presence of functional groups like hydroxyls further influence viscosity, albeit to a lesser extent [80-82]. Table 1 provides a summary of biodiesel properties derived from various feedstocks, highlighting the influence of fatty acid composition on cetane number, cloud point, flash point, and oxidative stability [83].

Table 1 Properties of biodiesel produced by various oils [70, 71, 72]

Feedstock	**Cetane Number**	**Cloud Point (°C)**	**Flash Point (°C)**	**Oxidation Stability (hr), 110°C**	**Kinematic Viscosity at 40°C (cSt)**	**Density (g/cm^3)**
WCO	56.2	5.3	161.7	5.0	4.75	880.6
Corn	53.0	-2.8	170.0	1.1	4.4	885.0
Soybean	49.0	1.0	178.0	2.1	4.039	884.0
Canola	54.8	-1.8	159.0	11.0	4.40	881.6
Jatropha	55.7	2.7	58.5	2.3	4.8	879.5
Coconut	61.0	0.0	110.0	35.5	2.726	807.3
Oil Palm	62.0	13.0	164.0	4.0	5.7	876.0
Cottonseed	53.3	1.2	165.4	1.8	4.70	879.0
Peanut	54.0	5.0	176.0	2.0	4.9	883.0
Rapeseed	54.4	-3.3	170.0	7.6	4.439	882.0
Sunflower	49.0	3.4	183.0	0.9	4.439	880.0
Rubber	54.1	-3.3	164.4	7.4	4.63	882.2
Castor	42.1	-13.4	160.9	1.1	15.250	899.0
Karanja	55.4	7.6	160.0	4.1	3.90	880.0
Safflower	51.8	0.9	172.0	1.3	4.53	882.9
Tallow	60.9	16.0	157.2	1.6	4.824	874.0
Olive Oil	57.0	-2.0	178.0	3.3	4.5	881.2
Almond Kernel	57.0	-	172.0	3.0	4.2	-
Linseed	51.3	-1.7	161.0	0.4	4.2	891.5
Sesame Seed	50.48	-6.0	170.0	-	4.2	867.3
Mahua Oil	56.9	-1.7	208.0	0.4	3.980	850.0

VII. CONCLUSION

This review comprehensively highlights the key aspects of biodiesel production, focusing on the influence of feedstock quality, fatty acid composition, and processing parameters on biodiesel properties. The selection of feedstock, primarily dictated by its fatty acid profile and free fatty acid content, significantly determines the fuel’s performance, including cetane number, cold flow properties, flash point, oxidation stability, and viscosity. Oils rich in saturated fatty acids, such as palm oil, are advantageous for cetane number and oxidative stability but present challenges in cold flow properties. Conversely, unsaturated oils, such as soybean and sunflower oils, offer superior cold flow behavior but are less stable under oxidative conditions. Advanced analytical techniques, including GC-MS, FTIR, TGA, and SEM-EDAX, have proven instrumental in evaluating biodiesel's chemical and physical properties, ensuring compliance with international standards. These methods provide critical insights into biodiesel composition, thermal stability, and the performance of catalysts and additives during production. Despite considerable advancements, challenges persist in optimizing biodiesel production processes, particularly when using high-FFA feedstocks or achieving a balance between cold flow properties and oxidative stability. Future research should focus on exploring innovative feedstocks, enhancing catalyst efficiency, and integrating advanced analytical tools to refine biodiesel quality further. Biodiesel remains a promising renewable energy resource, offering significant potential to reduce dependency on fossil fuels and mitigate environmental impact. However, sustained efforts in research and development are essential to overcome the limitations and ensure its economic viability and widespread adoption.

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