BIODIESEL AND GLYCERINE FROM WASTE COOKING OILS: DIFFERENT METHODS AND THEIR EFFECTIVENESS

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Abstract - *The growing demand for renewable energy and rising environmental concerns have fueled increased research into producing biodiesel from waste cooking oils (WCOs). This review offers an in-depth examination of methods for producing both biodiesel and glycerin from WCOs from 2020 to 2024. It evaluates various transesterification approaches - such as alkaline, acidic, enzymatic, and supercritical methods - by assessing yield, purity, and economic feasibility. The study also explores glycerin processing, a key byproduct, and its diverse applications. In addition, it highlights the challenges and future outlook in biodiesel production from WCOs, emphasizing the critical role of sustainable practices and technological advancements. By addressing these factors, the review underscores the potential for WCO-based biodiesel and glycerin to make meaningful contributions to renewable energy solutions and environmental sustainability by addressing these factors.*

Key Words: **WCOs, FAMEs, FFAs, Transesterification, NaOH, Methanol, Glycerine, Biodiesel.**

1. Introduction

Fossil fuels have long been the dominant source of energy worldwide. However, their adverse environmental impacts, such as the release of greenhouse gases and air pollutants, have led to growing concerns over their sustainability. These environmental challenges have prompted an accelerated global shift toward renewable energy sources, which are seen as essential to mitigating climate change and ensuring energy security in the future. Among these renewable alternatives, biodiesel has emerged as a promising substitute for conventional diesel fuel. Derived from vegetable oils and animal fats, biodiesel is not only renewable and biodegradable but also reduces dependence on fossil fuels. It has been widely regarded as an eco-friendly solution to reduce emissions and improve air quality (Knothe et al., 2005).

Waste cooking oils (WCOs), which are abundantly available and inexpensive, have gained considerable attention in recent years as a potential feedstock for biodiesel production. WCOs, which are typically discarded after use in households, restaurants, and food processing industries, are a significant source of waste in many regions. The reuse of these oils for biodiesel production offers a dual benefit: it reduces waste disposal challenges while simultaneously contributing to the production of a sustainable energy source. Compared to virgin vegetable oils, WCOs offer multiple advantages, including lower raw material costs and a reduced environmental footprint. The process of converting waste into energy further enhances the ecological benefits, making WCO-based biodiesel a more sustainable and cost-effective alternative to traditional biodiesel sources (Dias et al., 2011).

The production of biodiesel from WCOs typically involves a chemical reaction known as transesterification. This process involves the conversion of triglycerides present in the oils into fatty acid methyl esters (FAMEs), which are the primary component of biodiesel, and glycerine as a by-product. Transesterification has become the most commonly employed method for biodiesel production due to its efficiency and established protocols in the industry. This chemical reaction allows for the transformation of the raw WCO material into a high-quality biodiesel fuel suitable for various applications (Freedman et al., 1984). Over the past few years, a number of advanced transesterification methods and modifications have been introduced to improve biodiesel yields, reduce the need for costly reagents, and increase overall efficiency.

This review aims to comprehensively examine the methods used for biodiesel and glycerine production from WCOs between 2020 and 2024. The focus will be on evaluating the effectiveness of these methods in terms of biodiesel yield, fuel purity, and economic feasibility. By reviewing and comparing recent advancements, this paper provides an updated understanding of the most promising techniques and their potential for large-scale implementation. Through this analysis, the review will contribute to the ongoing development of biodiesel production strategies, addressing both environmental and economic concerns

2. Methods for Biodiesel Production from WCOs

Several methods have been utilized for biodiesel production from WCOs, each with its own advantages and disadvantages. This section delves into the most commonly employed techniques, focusing on their effectiveness during the period 2020 to 2024.

2.1 Alkaline Transesterification

Alkaline transesterification is one of the most commonly used methods for biodiesel production, utilizing sodium or potassium hydroxide as the catalyst. It is favored for its simplicity, high yield, and relatively low cost, which make it an attractive option for large-scale biodiesel production (Vicente et al., 2004). The method involves the reaction of triglycerides (fats or oils) with alcohol (usually methanol or ethanol), producing biodiesel (methyl or ethyl esters) and glycerol as a by-product. Despite its widespread use, this method has a key limitation: its sensitivity to free fatty acids (FFAs) present in waste cooking oils (WCOs).

Challenges with FFAs in Alkaline Transesterification The presence of FFAs in WCOs can significantly hinder the alkaline transesterification process. When FFAs are present in the oil, they react with the alkaline catalyst (such as sodium hydroxide) to form soaps (sodium salts of fatty acids). Soap formation leads to a reduction in the overall biodiesel yield, as some of the fatty acids are "locked up" in the soap rather than converting into biodiesel. Additionally, soap formation complicates the purification and separation processes during downstream processing, making it more difficult to separate the biodiesel from the glycerol and residual contaminants.

Strategies to Overcome FFA Sensitivity In response to this challenge, various strategies have been developed in recent years to address the issue of FFAs in WCOs while still utilizing the alkaline transesterification method. One of the most widely explored approaches, particularly during the period from 2024 to 2020, involves two-stage transesterification.

Two-Stage Transesterification Process: This process involves two distinct steps aimed at removing or neutralizing FFAs before proceeding with the alkaline transesterification reaction. In the first stage, FFAs are removed or converted into esters. Typically, this is done by esterification, where methanol and an acidic catalyst (e.g., sulfuric acid) are used. The acid catalyzed esterification effectively reduces the FFA content by converting the FFAs into biodiesel (methyl esters). In the second stage, the alkali-catalyzed transesterification process is carried out as usual, with methanol and a base catalyst (such as sodium hydroxide) to produce biodiesel. This two-stage process helps reduce soap formation, as the FFA content has already been minimized. Studies have shown that this dual approach leads to higher yields of biodiesel, as it minimizes the issues associated with soap formation and makes the downstream processing much simpler (Islam et al., 2020). The acid-catalyzed esterification step ensures that even oils with a high FFA content can still be converted into biodiesel efficiently.

Other Modifications and Innovations In addition to the two-stage process, various other modifications and innovations have been explored in recent years to further improve the effectiveness of alkaline transesterification, particularly in dealing with WCOs and other feedstocks with high FFA content. Some of these include:

i. Pre-treatment with Activated Carbon or Clay: Research has explored the use of adsorbents like activated carbon or clay to remove FFAs from the oil before transesterification. This helps lower the FFA concentration, thus reducing the likelihood of soap formation during the alkaline transesterification.

ii. Ultrasound and Microwave-Assisted Transesterification: These technologies have been shown to improve the efficiency of the transesterification reaction by increasing the mass transfer and reaction rates. These methods can be particularly effective when using WCOs, as they can reduce the required reaction time and energy input.

iii. Optimized Catalyst Systems: Some studies have focused on developing more effective catalyst systems that can tolerate higher levels of FFAs, reducing the need for extensive pre-treatment. For example, mixed metal oxide catalysts or enzymatic catalysts have been investigated for their ability to catalyze transesterification reactions even in the presence of FFAs.

vi. Supercritical Fluid Transesterification: Another advanced approach involves conducting the transesterification reaction under supercritical conditions (high temperature and pressure). This method has been shown to improve biodiesel yields from oils with high FFA content, but it is generally more energy-intensive and costly.

Effectiveness and Future Outlook (2024-2020) The period from 2024 to 2020 has seen significant progress in refining the alkaline transesterification process for WCOs with high FFA content. Research has continuously focused on improving the yield and quality of biodiesel while minimizing the negative impact of FFAs. By incorporating modifications like two-stage transesterification, the production of high-quality biodiesel from WCOs has become more feasible and economically viable. Looking forward, it is likely that further improvements in catalyst design, pre-treatment processes, and reaction conditions will continue to enhance the efficiency of alkaline transesterification. The development of more sustainable and cost-effective methods to deal with FFAs and other impurities in WCOs will be crucial in making biodiesel production from waste oils more widespread and economically viable.

2.2 Acidic Transesterification

Acidic transesterification is a catalytic process that uses strong acids (typically sulfuric acid or hydrochloric acid) to convert triglycerides in vegetable oils or waste cooking oils (WCOs) into biodiesel (methyl or ethyl esters). This method is particularly beneficial for feedstocks with high levels of free fatty acids (FFAs), which would otherwise interfere with the alkaline transesterification process. Acidic transesterification circumvents the issue of soap formation, which occurs in alkaline catalysis when FFAs react with the base to form soap, leading to decreased biodiesel yield and more complex downstream processing.

Challenges with High FFA Content: One of the primary challenges in biodiesel production from oils with high FFA content (such as WCOs) is the formation of soaps during alkaline transesterification. These soaps not only reduce biodiesel yield but also complicate the separation of biodiesel from glycerol and other contaminants, increasing operational costs. In contrast, acidic transesterification addresses this challenge by converting FFAs into biodiesel before the main transesterification step, thus preventing soap formation.

The process of acidic transesterification typically follows two-step procedure: The first step involves esterification of the FFAs present in the feedstock. Methanol or ethanol is reacted with the FFAs in the presence of a strong acid catalyst, such as sulfuric acid. The reaction produces methyl or ethyl esters and reduces the FFA content in the oil to a level that can be effectively processed through alkaline transesterification. This step is crucial for reducing soap formation and improving biodiesel yield in subsequent reactions. Following esterification, the oil undergoes alkaline transesterification, typically using sodium hydroxide (NaOH) or potassium hydroxide (KOH) as catalysts. In this step, methanol is used to convert the triglycerides into biodiesel and glycerol. The alkali catalyst reacts with the triglycerides, yielding biodiesel and producing glycerol as a by-product.

This two-step approach allows for the effective conversion of oils with high FFA content, ensuring a high-quality biodiesel product with minimal soap formation.

Innovations and Modifications: Several modifications have been introduced in recent years to optimize acidic transesterification, including:

i. Solid Acid Catalysts: The use of solid acid catalysts, such as heteropolyacids or ion-exchange resins, has gained attention due to their ability to catalyze esterification reactions without generating significant waste. These catalysts are reusable, reducing the overall cost and environmental impact of the process.(Guo et al., 2023)

ii. Enzyme-Assisted Acidic Transesterification: Enzymatic catalysts have been combined with acidic transesterification to create a hybrid approach. Enzymes such as lipases can catalyze the esterification of FFAs under mild conditions, enhancing the efficiency of the reaction and reducing energy consumption.

iii. Optimization of Reaction Conditions: Research has focused on improving reaction parameters, such as temperature, pressure, and methanol-to-oil ratio, to maximize biodiesel yield while minimizing environmental impact. The development of continuous flow reactors and the integration of co-solvents have also shown promise in reducing the overall reaction time.

Effectiveness and Future Outlook (2024-2020): From 2020 to 2024, significant improvements in the efficiency of acidic transesterification have been achieved, particularly through the use of solid acid catalysts and optimization of process conditions. These innovations have reduced the environmental impact of acidic transesterification and made it more commercially viable. Going forward, the development of more efficient and sustainable acid catalysts, as well as the integration of this process with other biodiesel production technologies, will likely drive further improvements in the cost-effectiveness and scalability of acidic transesterification. Additionally, as waste cooking oils become a more widely used feedstock, the demand for acidic transesterification will continue to rise, prompting further research into optimizing this method.

2.3 Enzymatic Transesterification

Enzymatic transesterification is an environmentally friendly and sustainable method for producing biodiesel. This method utilizes enzymes, most commonly lipases, as catalysts to convert triglycerides into biodiesel. The process is carried out under mild conditions (lower temperature and pressure), which leads to less waste production and reduced energy consumption compared to traditional chemical methods. Despite these benefits, enzymatic transesterification faces challenges, such as higher enzyme costs, lower reaction rates, and sensitivity to impurities in the feedstock.

Challenges with Enzyme Cost and Efficiency: The high cost of enzymes remains one of the significant drawbacks of enzymatic transesterification. Enzymes, particularly lipases, are expensive to produce and may need to be replaced after each batch, making the process less cost-effective than chemical methods. Additionally, enzymes can be sensitive to various impurities in feedstocks, including water, alcohol, and FFAs, which can reduce their catalytic activity. However, innovations in enzyme immobilization and reactor design have improved the cost-efficiency and robustness of enzymatic transesterification.

Innovations and Modifications: Several strategies have been explored in recent years to enhance the performance of enzymatic transesterification, including:

i. Enzyme Immobilization: Enzyme immobilization techniques, such as adsorption on solid supports (e.g., silica, activated carbon, magnetic nanoparticles), have significantly improved the stability, reusability, and catalytic efficiency of lipases. Immobilized enzymes can be reused multiple times, reducing the overall operational costs and making the process more economically viable.

ii. Co-Solvents and Co-Catalysts: The addition of co-solvents or co-catalysts has been studied to improve the solubility of feedstocks and methanol in the reaction medium. Co-solvents such as acetone, ethanol, and hexane can enhance the interaction between the enzymes and the substrates, improving reaction rates and biodiesel yield.

iii. Thermal and pH Optimization: Enzymes are often optimized for use at higher temperatures and more extreme pH conditions, which helps enhance their catalytic efficiency and stability. Lipases that function under non-ideal conditions allow for greater flexibility when processing oils with higher FFA content or varying viscosity.

Effectiveness and Future Outlook (2020-2024): Enzymatic transesterification has gained substantial attention due to its environmental benefits, especially in terms of reduced waste production and the use of mild reaction conditions. However, the relatively high cost of enzymes and the slower reaction rates have hindered its widespread adoption. From 2020 to 2024, significant progress has been made in overcoming these barriers. Advances in enzyme immobilization have led to more cost-effective solutions, and hybrid approaches using enzymatic catalysts in combination with chemical methods are becoming more common. The future of enzymatic transesterification will likely involve the development of low-cost, robust enzymes and optimized reactor systems that further reduce the overall cost of biodiesel production. Additionally, the integration of this method with other technologies (e.g., ultrasonic or microwave-assisted transesterification) could improve the efficiency and scalability of enzymatic biodiesel production.

2.4 Supercritical Methanol Transesterification

Supercritical methanol transesterification is a high-efficiency, non-catalytic method for biodiesel production. In this process, methanol is used in its supercritical state, where it exhibits both solvent and catalytic properties, allowing for faster transesterification of triglycerides into biodiesel. The process occurs under high temperature and pressure conditions, typically above the critical temperature (240°C) and pressure (80 atm) of methanol. This method is advantageous for its high biodiesel yield and ability to process feedstocks with high FFA content, making it ideal for waste oils such as WCOs.

Challenges with High Energy Consumption: The primary challenge associated with supercritical methanol transesterification is its high energy consumption. Maintaining supercritical conditions requires specialized equipment, such as high-pressure reactors, and substantial energy input. This makes the process energy-intensive and costly compared to conventional catalytic methods. The high capital and operational costs are significant barriers to the widespread adoption of supercritical methanol transesterification in large-scale biodiesel production. Despite these challenges, researchers are continuously exploring ways to optimize this process to improve its economic feasibility.

Innovations and Modifications: Recent research has focused on improving the efficiency and reducing the energy consumption of supercritical methanol transesterification. Innovations include:

i. Microwave-Assisted Supercritical Methanol Transesterification: Microwave heating has been employed to enhance the heating efficiency in supercritical methanol transesterification. The use of microwaves reduces reaction time and improves energy efficiency by providing rapid and uniform heating. This approach significantly cuts down energy consumption while maintaining high biodiesel yields.

ii. Use of Co-Solvents and Additives: The incorporation of co-solvents, such as ethanol or water, has been investigated to reduce the temperature and pressure required to reach the supercritical state of methanol. This reduces the energy demand and makes the process more economical. Additionally, the use of additives can help increase the solubility of triglycerides and methanol, enhancing the efficiency of the transesterification reaction.

iii. Continuous Flow Reactors: Moving from batch reactors to continuous flow reactors has been explored to improve the scalability of supercritical methanol transesterification. Continuous flow reactors allow for higher throughput and more efficient heat transfer, improving the overall efficiency of the process and reducing the downtime between batches.

Effectiveness and Future Outlook (2020-2024): From 2020 to 2024, supercritical methanol transesterification has seen promising developments, particularly in terms of process optimization and energy efficiency. The integration of microwave-assisted heating and continuous flow reactors has improved the economic feasibility of the process.

However, the high energy demand remains a major challenge that needs to be addressed for the method to be more widely adopted. Future research will likely focus on reducing energy consumption, enhancing reactor design, and exploring new catalyst-free methods to lower operational costs. As the technology matures, supercritical methanol transesterification has the potential to become a key method for large-scale biodiesel production, particularly from waste oils and low-quality feedstocks.

Each method offers distinct advantages tailored to different feedstocks and production needs: alkaline transesterification is ideal for low-FFA oils, acidic transesterification works well for high-FFA oils, enzymatic transesterification provides an environmentally friendly and high-quality output, and supercritical methanol transesterification achieves rapid reactions and high yields without catalysts. As research progresses, these methods continue to be refined to improve their efficiency, sustainability, and economic viability, contributing to the growing potential of biodiesel as a renewable energy source.

Aspect	Alkaline Transesterification	Acidic Transesterification	Enzymatic Transesterification	Supercritical Methanol Transesterification
Catalyst Type	Sodium or potassium hydroxide	Sulfuric acid, hydrochloric acid	Lipases (enzymes)	None (uses supercritical methanol)
Yield and Purity	High yield but affected by free fatty acid (FFA) content, causing soap formation	Effective for high FFA content oils, but slower and produces waste	High-quality biodiesel, environmentally friendly	Very high yields and reduced separation issues
Reaction Conditions	Moderate temperature and pressure, but sensitive to impurities	Requires harsh conditions and longer reaction times	Mild conditions, lower temperatures, and environmentally friendly	High temperature and pressure, energy-intensive
Cost and Scalability	Cost-effective, simple, but requires FFA pre-treatment	More expensive and less efficient for large scale	High cost due to enzyme use, with research improving enzyme reusability	High initial investment for specialized equipment, less cost-effective
Environmental Impact	Generates soap waste if not optimized	High environmental impact due to waste and energy use	Low waste production, environmentally friendly	Energy-intensive but no catalyst waste
Advancements (2024-2020)	Two-stage processes for better FFA handling and higher yields	Use of solid acid catalysts derived from waste to reduce impact	Immobilization of enzymes for reusability and cost reduction	Microwave-assisted methods to reduce energy use and increase speed

Table 1: Method ⇧ Comparison

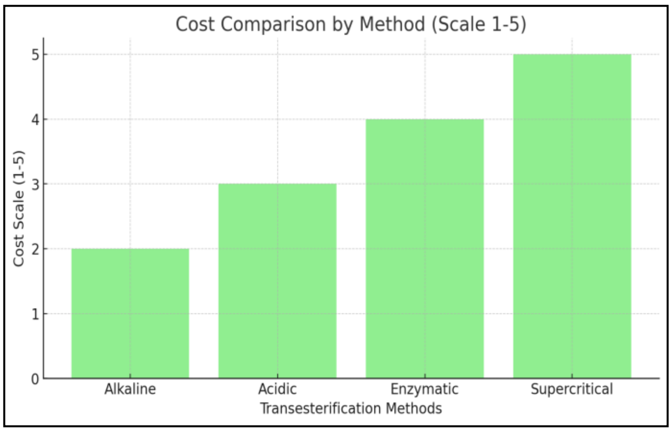

Fig 1: Cost comparison of process

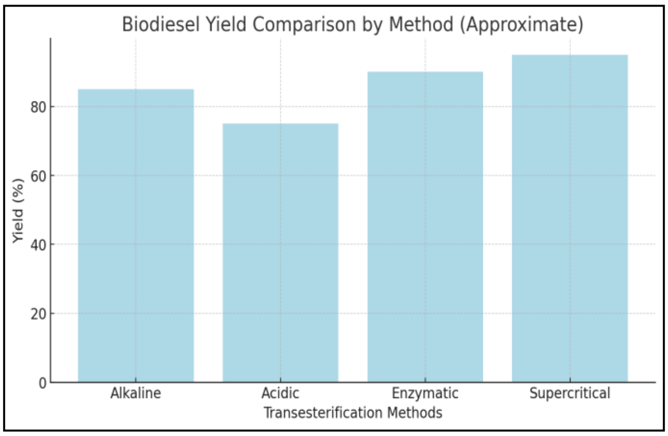

Fig 1 : Yield comparison of process

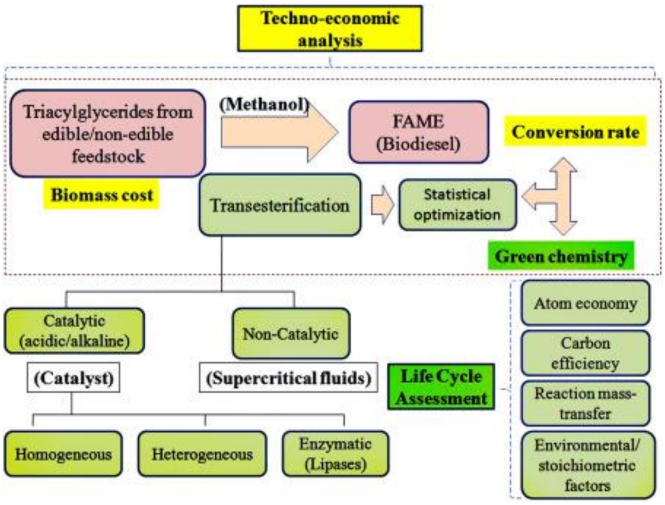

Fig 3: Schematic diagram of process

3. Glycerine Purification

Glycerine Recovery Methods in Biodiesel Production

The recovery of glycerin from biodiesel production is essential for ensuring the sustainability of biodiesel processes. Glycerine, a byproduct of transesterification, must be separated from biodiesel efficiently to be purified and reused. Common methods for glycerine recovery include decantation, filtration, and centrifugation, each with its own advantages and limitations depending on the mixture's characteristics and the required purity of glycerine.

i. Decantation: This is a simple and cost-effective method that relies on the density difference between glycerine (1.26 g/cm^3) and biodiesel (0.88–0.90 g/cm^3). Glycerine settles at the bottom of the mixture, making it easy to separate. However, decantation may not effectively remove smaller impurities like residual catalysts or methanol.

Aspect	Glycerin Recovery	Glycerin Purification
Recovery Methods	Decantation, filtration, centrifugation	Distillation, ion exchange, membrane separation
Advancements	Focused on efficient separation methods to increase yield	Development of energy-efficient purification methods
Economic Impact	Important for overall economic feasibility	High purity is necessary for certain applications

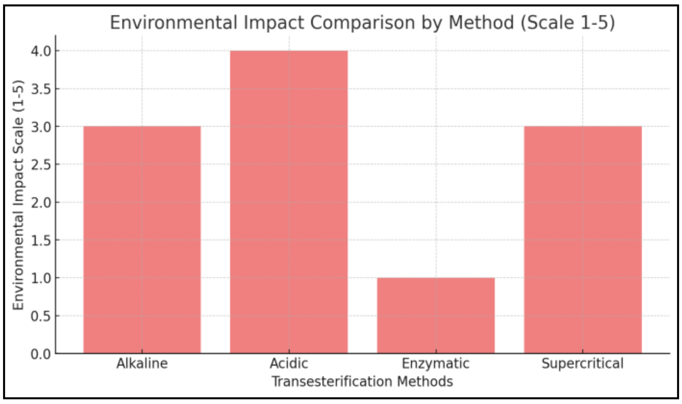

Fig 4: Environmental impact of process

ii. Filtration: Filtration removes particulate matter, such as fine solids and traces of catalysts, after decantation. It helps further purify the glycerin but is less effective at removing liquid impurities like methanol or other residual chemicals.

iii. Centrifugation: Centrifugation uses high-speed spinning to create centrifugal forces that separate glycerin from biodiesel more effectively, particularly in emulsified mixtures. This method can achieve higher purity than decantation and filtration but requires more energy and has higher operational costs.

The choice of method depends on:

a)Physical properties: If glycerine and biodiesel differ greatly in density or viscosity, decantation or filtration can be more effective.

b)Purity needs: For high-purity glycerine, advanced methods like centrifugation or a combination of techniques may be necessary.

c)Scale of operation: Large-scale biodiesel plants often rely on centrifugation or filtration for efficient glycerine recovery.

Applications of Glycerin:

The increasing production of biodiesel has led to a surge in the availability of glycerin. Consequently, researchers and industries are actively exploring new and diverse applications for glycerin to enhance its economic value.

a) Cosmetics and Personal Care: Glycerine's moisturizing properties make it a widely used ingredient in cosmetics and personal care products, such as lotions, creams, and soaps. It acts as a humectant, attracting and retaining moisture in the skin, leading to improved hydration and smoother skin texture (Singh & Sharma, 2012).

b) Pharmaceuticals: Glycerine is employed in pharmaceutical formulations as a solvent, humectant, and sweetener. It is used in various drug delivery systems, including syrups, tablets, and ointments. Its nontoxic nature and compatibility with a wide range of drugs make it a suitable excipient in pharmaceutical formulations (Patel et al., 2012).

c) Food Industry: Glycerine is a versatile ingredient in the food industry, serving as a sweetener, humectant, and stabilizer. It is used in various food products, including baked goods, confectionery, and beverages (Pagliaro & Rossi, 2010).

4. Challenges

Feedstock Availability and Quality: The consistency of WCO supply and its variable quality can pose challenges for biodiesel production. The presence of impurities, like FFAs and water, can affect the efficiency of the transesterification process. Therefore, efficient pretreatment methods for WCO are crucial.

Catalyst Optimization: Developing cost-effective and environmentally friendly catalysts is essential for improving the sustainability of biodiesel production. Research on heterogeneous catalysts derived from renewable sources is progressing, but further optimization is needed.

Glycerine Valorization: Despite the increasing applications of glycerine, its market value is still relatively low compared to the other biodiesel co-products. Exploring new and diverse applications for glycerin is crucial to maximize its economic potential.

Energy Consumption: The transesterification process requires energy for heating and stirring, impacting the overall energy efficiency of biodiesel production. Developing energy-efficient processes is vital for minimizing environmental impact.

Economic Viability: The economic feasibility of biodiesel production from WCOs is influenced by various factors, including feedstock cost, catalyst cost, and product demand. Ensuring the long-term economic viability of biodiesel production requires carefully considering these factors.

5. Future Prospects

Advancements in technologies like membrane separation, microwave-assisted processes, and supercritical fluid extraction can enhance the efficiency and sustainability of biodiesel production. Integrating biodiesel production with other industries, such as wastewater treatment and biogas production, can enhance resource utilization and reduce the overall environmental footprint.

Implementing the biorefinery concept, where various valuable products are extracted from WCOs, can maximize the economic and environmental benefits of this feedstock. Implementing supportive policies and incentives can encourage the adoption of biodiesel and stimulate further research and development in this field. (Zhang et al.,2023)

6. Conclusions

Biodiesel production from WCOs has emerged as a promising pathway for renewable energy generation and waste management. The period from 2020 to 2024 witnessed significant progress in understanding and optimizing the different methods for biodiesel and glycerin production, with a focus on enhancing efficiency, sustainability, and economic viability.

Alkaline transesterification remains the most popular method, but modifications like two stage transesterifications are being implemented to address challenges associated with FFA content. Enzymatic and supercritical transesterification are gaining traction due to their environmentally friendly nature and potential for high-quality biodiesel. Moreover, efforts are being made to optimize glycerin recovery and purification methods to maximize its economic value through diverse applications.

Despite the advancements, several challenges remain, including feedstock availability, catalyst optimization, and glycerin valorization. Addressing these challenges through continuous technological innovation, coupled with supportive policies and incentives, will be crucial for the long-term success of the biodiesel and glycerin industry from WCOs.

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