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REVIEW ARTICLE

A comprehensive review of biomass-derived heterogeneous catalysts for efficient biodiesel production

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Abstract: The transesterification process for biodiesel production relies on efficient catalysts to accelerate the chemical reactions involved. Choosing the appropriate catalyst is crucial and depends primarily on the free fatty acid content of the oil feedstock. Conventional biodiesel production processes typically employ homogeneous catalysts, which present several disadvantage, including toxicity, high flammability, corrosiveness, and significant effluent generation. Consequently, there is growing interest in biomass-derived heterogeneous catalysts and bio-waste, as these offer sustainable, recyclable, and environmentally friendly alternatives. These catalysts exhibit excellent stability and catalytic efficiency in both acidic and basic environments, as well as superior water resistance. This review provides an in-depth analysis of biomass-based heterogeneous catalysts, emphasizing their potential for sustainable biodiesel production. The primary focus is on the usage of biomass and bio-waste-derived catalysts for producing cost-effective biodiesel. This review offers an overview of present methods for synthesizing various types of catalysts, such as basic, acidic, bifunctional, and nanocatalysts, using a range of feedstocks. Furthermore, it explores the impact of different catalyst preparation techniques on biodiesel yield and evaluates the sustainability of these catalysts. This study also identifies gaps in the present literature on biomass-derived heterogeneous catalysts and other biocatalysts, offering suggestions for future research avenues.

Keywords: High catalytic activity; Homogeneous catalysts; Transesterification process; Biomass waste; Heterogeneous catalysts

1. Introduction

The use of solid catalysts, also known as heterogeneous catalysts, in biodiesel production has increased significantly in recent years. Concerns about global warming, growing environmental consciousness, diminishing fossil fuel reserves, and the volatility of global oil prices are the primary drivers of this heightened

interest. These challenges highlight the urgent need for sustainable alternative energy sources, such as biofuels and biodiesel.[1,2] At present, fossil fuels account for 80% of the world's primary energy consumption, with 58% of that energy utilized exclusively for transportation.[3,4]

There are several advantages to producing biodiesel through lipase catalysis, including easier product separation, reduced effluent generation, and fewer adverse reactions.[5,6] Free fatty acids (FFAs) and fatty acid alkyl esters may be processed more easily using immobilized lipase under moderate reaction conditions, unlike traditional homogeneous alkaline catalysis, which is ineffective at high FFA concentrations.[7,8] However, lipases can have several drawbacks, such as slower reaction rates and the potential for enzyme inactivation caused by high methanol content.[9]

The widespread availability of fossil fuels highlights the urgent need for alternative, green energy sources. Natural gas, syngas, hydrogen, and biofuels are notable, environmentally benign, and sustainable substitutes. The United States (US), Brazil, the European Union (EU), and several Asian countries are major participants in the global biofuel industry.[10,11] Renewable energy sources and biodiesel contribute to economic growth by generating employment, income, tax revenue, and expenditures on facilities and infrastructure.[12] Using alternative fuels, particularly in the transportation industry, can lessen reliance on crude oil supplies while enhancing the country's agricultural potential.

The advantages of alternative fuels outweigh the disadvantages of fossil fuels. These advantages include non-toxicity, biodegradability, high energy efficiency, and reductions in carbon dioxide, sulfur, fine particulates, and nitrogen oxide emissions.[13-15] The energy required to produce biodiesel, for instance, is thought to be between 40% and 90% less than the fossil fuels.[1] Additional biochemical advantages include high lubricity, low viscosity, a high cetane number, a high flash point, and reduced sulfur dioxide and carbon monoxide emissions.

Biodiesel is produced by transesterifying triglycerides from processed edible or non-edible oils using alcohols, such as methanol, ethanol, butanol, or amyl alcohol[16] and catalysts, such as acidic, alkaline, or biochemical catalysts, as shown in Figure 1. Methanol is commonly used due to its affordability, short chain structure, rapid rate of reaction, and ease of combining with the resulting media.[17] The catalyst assists in neutralizing the molecules of glycerol and fatty acid methyl esters (FAME) throughout the transesterification procedure, which occurs at temperatures between 60°C and 80°C. The resulting products are subsequently isolated for purification. Acidic substances, bases, biological catalysts, or catalysts made from biomass and bio-waste can all accelerate the transformation of fatty acids during this procedure.[18,19]

The manufacturing of biodiesel on a commercial basis frequently uses homogeneous base catalysts, such as phosphoric acid, potassium hydroxide (KOH), and sodium hydroxide (NaOH). These catalysts offer several benefits, including high catalytic activity,[20] quick response rates, easy availability, low cost, and mild working conditions. Nevertheless, they are extremely vulnerable to water and FFAs. The reaction may therefore result in soap and copious volumes of effluent, raising the cost of manufacturing and operation.[21] In addition, these considerations reduce the environmental sustainability of homogeneous base catalysts.[22]

For processing high FFA raw materials, including animal fats, leftover cooking oil, and most basic vegetable oils, homogeneous acid catalysts, such as sulfuric acid (H_2SO_4), hydrochloric acid, and phosphoric acid are more efficient than homogeneous base catalysts. These catalysts have several advantages, including the capacity to catalyze parallel procedures for transesterification and esterification without producing soap by-products, along with resistance to water and FFA sensitivity.[23,24] However, a key obstacle to commercialization is their slower reaction rate (about 4,000 times slower) than base catalysts. In addition, their extremely acidic and corrosive nature complicates handling and may compromise the lifespan of the equipment.

Biodiesel and biofuel production is usually expensive, with raw material and processing costs significantly determining the price.[25] Consequently, it is crucial to

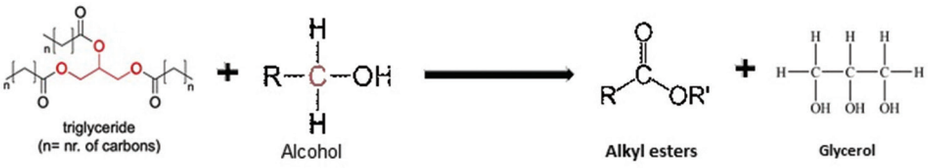

Figure 1. General method for preparing biodiesel

find novel approaches to lower processing and material expenses. In this context, heterogeneous catalysts present a promising alternative to conventional homogeneous catalysts. Since 2006, a pilot facility in France has been producing 160,000 tons of biodiesel annually despite the limited large-scale commercialization in the energy sector. Because heterogeneous catalysts are not consumed throughout the reaction, they reduce reaction durations and simplify product separation, making them essential in transesterification processes.[26]

Nowadays, several types of catalysts are available in the market, such as metal oxides, mixed oxides, and hydrotalcites.[27] Zeolites, carbon-based catalysts, ion exchange resins, and transition metal oxides are employed in acidic procedures. However, their three-phase reaction system, which results in diffusion constraints that obstruct the reaction process, is a major disadvantage of these heterogeneous catalysts.[28] In addition, this system decreases mass transfer effectiveness, which is crucial for optimizing reaction rates.[29] Other drawbacks include low active site counts, microporosity, leaching, toxicity, high expenses, non-renewability, and adverse environmental effects.[30]

2. Classification of catalysts

A catalyst speeds up a chemical process without changing its thermodynamic characteristics. By speeding up the reaction rate during the transesterification process, catalysts increase the yield of the end product. Most commonly, these catalysts fall into one of four categories: (i) homogeneous, (ii) heterogeneous, (iii) enzymatic, and (iv) non-catalytic procedures that operate at supercritical conditions.

2.1. Homogeneous acid catalysts

Homogeneous acid catalysts are frequently employed in biodiesel synthesis due to their affordability, accessibility, and fast reaction rates. Base-based and acid-based catalysts, with common examples, including H_2SO_4, NaOH, and KOH, can be used. According to research, the main mechanism by which these catalysts function involves a nucleophilic attack on the carbonyl group.[31] However, homogeneous catalysts have several limitations, such as higher costs, inefficient use, significant wastewater production, difficulties in end-product separation, and thermal instability, as they tend to break down at temperatures >150°C.[32] Table 1 summarizes the biodiesel manufacturing process using various solid acid catalysts, including the sources of raw materials, methods of preparation, parameters of the response, and relevant references.

Alkalized homogeneous catalysts further accelerate transesterification, which may achieve reaction rates up to 4,000 times faster than acid-based catalysts.[33] Common alkaline catalysts used in biodiesel production

Table 1. Solid acid catalysts for biodiesel production

Catalyst	Feedstocks	Continuous process manufacturing	Reaction operating conditions		References
			Molar ratio	Yield (%)	
Carbon-derived catalyst	*Calophyllum inophyllum* oil	Pyrolysis	30:1	90	35
Cesium phosphotungstate	Oleic acid-soybean mixture	Precipitation	20:1	90	36
$Mn_{3.5\times}Zr_{0.5y}Al_xO_3$	Waste cooking oil	Co-precipitation	14:1	75	37
Phosphotungstic acid-poly (glycidyl methacrylate)-magnetic nanoparticles	Greases	Direct mixing	33:1	96	38
Anion/cation-exchanged resin	Pure triolein	Neutralization	10:1	96	39
SiO_2-SO_3H/cobalt ferrite	Rambutan oil	Co-precipitation	20:1	95	40
M-phenylenediamine-SO_3H-ionic liquid	Jatropha oil	Co-polymerization	50:1	90	41
Aluminum chloride hexahydrate ($AlCl_3 \cdot 6H_2O$)	Brown grease	Hydrothermal	10:1	86	42
Organically modified resin-[C_4HMTA][SO_3H]	Brown grease	Co-polymerization	40:1	95	43
Cesium hydrogen phosphotungstate ($Cs_{2.5}H_{0.5}PW_{12}O_{40}$)	Sesame oil	Precipitation	40:1	90	44

include sodium methoxide, KOH, NaOH, carbonates, sodium peroxide, potassium methoxide, and sodium ethoxide. Among these, sodium methoxide and potassium methoxide are particularly efficient due to their ease of dissociation into CH_3O^- and Na^+ or K^+, respectively, resulting in larger yields of biodiesel.[34]

Homogeneous catalysts offer superior selectivity and activity due to their uniform dispersion in the reaction medium, ensuring precise control over reaction pathways and higher yields with minimal byproducts. They operate efficiently under mild conditions, reducing energy demands and enhancing safety, particularly for sensitive compounds. Their solubility enables straightforward characterization using spectroscopic methods, providing valuable mechanistic insights for optimization. In addition, the consistent reaction environment ensures uniform kinetics, enabling better control in complex syntheses.

Despite their various advantages, homogeneous catalysts also possess several disadvantages. Homogeneous catalysts face significant separation difficulties due to their shared phase with reactants, complicating recycling and risking product contamination. While solutions, such as multiphase systems exist, they often add process complexity. Thermal stability limitations (typically <200°C) restrict high-temperature applications, and many catalysts pose toxicity concerns with challenging disposal requirements. Immobilization attempts frequently suffer from leaching issues, causing deactivation and potential environmental contamination, which are key obstacles to industrial adoption.

Table 1 presents a comprehensive overview of solid acid catalysts utilized in biodiesel production from various feedstocks, highlighting their preparation methods, molar ratios, and biodiesel yields. For instance, a carbon-derived catalyst synthesized through pyrolysis from *Calophyllum inophyllum* oil achieved a biodiesel yield of 90%. Similarly, cesium phosphotungstate catalysts prepared through precipitation from oleic acid-soybean mixtures also attained a 90% yield. $Mn_{3.5x}Zr_{0.5y}Al_xO_3$catalysts synthesized by co-precipitation from waste cooking oil yielded 75% biodiesel, indicating effective utilization of waste oils. Phosphotungstic acid-poly(glycidyl methacrylate)-magnetic nanoparticle catalysts prepared by direct mixing from greases achieved an impressive 96% yield, showcasing high efficiency. Anion/cation-exchanged resin catalysts derived from pure triolein through neutralization also reached a 96% yield, demonstrating versatility in feedstock utilization. SiO_2-SO_3H/cobalt ferrite catalysts synthesized by co-precipitation from rambutan oil achieved a 95% yield, highlighting their potential in biodiesel production. M-phenylenediamine-SO_3H-ionic liquid catalysts prepared through co-polymerization from jatropha oil attained a 90% yield, emphasizing their effectiveness in high-acid-value oils. Aluminum chloride hexahydrate ($AlCl_3 \cdot 6H_2O$) catalysts synthesized through hydrothermal methods from brown grease yielded 86% biodiesel, indicating their suitability for waste grease conversion. Organically modified resin-[C_4HMTA][SO_3H] catalysts prepared by co-polymerization from brown grease achieved a 95% yield, demonstrating high catalytic activity. Cesium hydrogen phosphotungstate ($Cs_{2.5}H_{0.5}PW_{12}O_{40}$) catalysts synthesized through precipitation from sesame oil attained a 90% yield, showcasing their efficiency in biodiesel production. Significant advancements in solid acid catalyst development offer sustainable and efficient pathways for biodiesel production from diverse feedstocks. The high yields achieved across various catalysts demonstrate their potential to replace conventional homogeneous catalysts, providing an eco-friendly alternative with enhanced recyclability and reduced environmental impact.

2.2. Solid base heterogeneous catalysts

Increased catalytic activity in heterogeneous solid base catalysts is attributed to their large effective surface area and abundance of basic sites. These catalysts usually consist of transition metal oxides, alkaline earth metals, or metallic oxides that may be doped, mixed, or single-component.[45] The basic sites in most solid base metal oxide catalysts are formed by the Lewis acid and Bronsted base sites of metal ions. Typical single metal oxide catalysts include barium oxide (BaO), strontium oxide (SrO), calcium oxide (CaO), and magnesium oxide (MgO). These solid base catalysts can produce biodiesel with similar yields in shorter response times and temperatures below 55°C.[46]

However, when utilized with feedstocks containing high FFA (≥2%) content, solid base catalysts have a significant drawback: they tend to generate soaps, which makes them less appropriate for such feedstocks.[47] CaO is widely used in biodiesel synthesis due to its low cost and strong reactivity.[48] MgO and other single metal oxides, on the other hand, are less soluble in alcohol and less efficient as catalysts. To increase their catalytic durability, the temperature of calcination can be raised to 873 K.[49] Studies have shown that CaO has more basic sites than MgO, resulting in higher catalytic activity. However, at milder operating temperatures, alkaline

earth metal oxides are more efficient catalysts for biodiesel production of biodiesel using feedstocks with low FFA.[50]

It was recently proposed that doping these catalysts using metal oxides will increase their catalytic potential and stability. Such doping can increase the pore size, durability, and surface area of the catalyst material.[51-53] As an example, it has been demonstrated that doping CaO with lithium greatly increases yield, with a 23% w/w lithium-doped CaO catalyst achieving over 100% of the total yield within 20 min of reaction time.[54]

2.3. Solid acid-base heterogeneous catalysts

Lewis acidity makes heterogeneous solid acid-base catalysts highly effective.[55] In addition to being recyclable, these catalysts exhibit minimal deactivation during transesterification. Examples include mixed oxides, such as silica-alumina,[56] ion exchange resins,[57] sulfonated polystyrene,[58] heteropolyacids,[59] sulfated zirconia,[60] tungstated zirconia,[61] zeolites,[62] and zeotype materials. Zeolites and zeotype materials are naturally occurring crystalline aluminosilicates in which oxygen atoms link to produce uniformly sized pores arranged in three dimensions.[63]

Zeolites act as sieves, allowing molecules of the same size to enter their pores while excluding larger ones. In addition, this pore structure facilitates ion exchange, enhancing catalytic activity by supplying negative ions.[64] Furthermore, the high electric fields generated by cations at active sites within the zeolite's pores improve adsorption.[65]

Recent advancements in biodiesel production have made it significantly more cost-effective through the use of solid acid catalysts, increasing their competitiveness as an alternative to petroleum-based fuels. Compared to conventional base catalysts and mineral acids, heterogeneous solid catalysts containing both Lewis-type (like sulfated mixed oxides) and Brønsted-type (like sulfonic acids) sites have several advantages.[66] These catalysts allow simultaneous transesterification and esterification reactions. Furthermore, heterogeneous solid acids are well-suited for low-grade feedstocks because they are less susceptible to moisture and have high levels of FFAs. Using less expensive feedstocks reduces the need for acid pre-treatment, thereby lowering manufacturing costs.[67] In addition, eliminating the acid pre-treatment phase reduces the risk of corrosion from acid feedstocks.

Other benefits of heterogeneous solid acid catalysts include ease of regeneration, high efficiency, recyclability, decreased deactivation, lower contamination, and simplified product separation.[38,68]

However, methanol dehydration during zeolite-catalyzed transesterification may result in the production of unwanted by-products, such as dimethyl ether, as reported in several studies.[69] The relationship between acid strength and hydrophobicity, which is controlled by the SiO_2/Al_2O_3 ratio, significantly affects their efficiency. The ratio decreases as the acidity increases; weaker acid corresponds to a higher ratio. Nonetheless, other studies have found that zeolites with high aluminum concentrations perform poorly in these reactions.[70]

2.4. Green catalysts (biocatalysts and bio-waste heterogeneous catalysts)

The role that biocatalysts play in producing biodiesel has grown as the demand for biodiesel and biofuels continues to develop globally at an estimated 5% annual growth rate.[71] Industrial waste materials provide a sustainable way to make inexpensive heterogeneous solid base catalysts that are environmentally friendly.[72,73] Egg shells, animal bones, fish scales, oysters, mussels, and cockle shells, as well as plant waste ash, are examples of calcium-rich materials that have been discovered as possible catalyst materials.[74] The amount of calcium that is recovered using waste products to create CaO, a versatile catalyst for heterogeneous processes, has been highlighted by research.[75,76]

Despite the fact that enzymes have many advantages, their efficacy may be constrained by their water sensitivity. Enzymatic activity during transesterification can be inhibited by the presence of polar molecules, such as phospholipids, glycerol, methanol, and water.[77] Researchers have implemented tactics, such as the gradual injection of methanol and other organic solvents to help alleviate this problem. The enzymes that act as biocatalysts when oils generated from inedible feedstocks are transesterified are listed in Table 2, together with information about their reaction circumstances and relevant references.

Table 2 summarizes the use of various enzymes in the transesterification of inedible oils to produce biodiesel, highlighting their reaction conditions and yields. Lipozyme *Thermomyces lanuginosus* immobilized, an immobilized lipase, was employed to transesterify castor oil with methanol at a 3:1 molar ratio, 45°C for 24 h, achieving a 60% yield. *Burkholderia cepacia* lipase catalyzed the transesterification of *Jatropha curcas* oil with ethanol at a 10:1 molar ratio, 35°C for 24 h, resulting in a 100% yield. *Candida parapsilosis* lipase was used to transesterify *J. curcas* oil with methanol at a 2:1 molar ratio, 30°C for 8 h, achieving an

Table 2. Enzymes for inedible oils transesterification

Catalyst	Feedstock	Reaction operating conditions					References
		Solvent	Molar ratio	Temperature (°C)	Time (h)	Yield (%)	
Lipozyme *Thermomyces lanuginosus* immobilized	Castor oil	Methanol	3:1	45	24	60	78
Burkholderia cepacia	*Jatropha curcas*	Ethanol	10:1	35	24	100	79
Candida parapsilosis lipase	*Jatropha curcas*	Methanol	2:1	30	8	80.50	80
Candida parapsilosis lipase	*Pistacia chinensis*	Methanol	5:1	37	60	90	81
Candida parapsilosis lipase	Bungeseed oil	Methanol	5:1	37	60	91	38
Candida parapsilosis lipase	*Calophyllum inophyllum*	Methanol	12:1	35	25	92	82

80.5% yield. In another study, *Pistacia chinensis* Bunge seed oil was transesterified with methanol at a 5:1 molar ratio, 37°C for 60 h, yielding 90% biodiesel. Similarly, *C. inophyllum* oil underwent transesterification with methanol at a 12:1 molar ratio, 35°C for 25 h, resulting in a 92% yield. These studies demonstrate the potential of using enzymes as biocatalysts for biodiesel production from inedible oils, offering an environmentally friendly alternative to traditional chemical catalysts. The yields achieved vary depending on the enzyme used, feedstock, molar ratio, temperature, and reaction time, indicating the need for optimization to maximize efficiency. The high yields observed in some cases, such as with *B. cepacia* lipase, suggest that enzymatic transesterification can be a viable method for producing biodiesel from non-edible oils.

Since enzymes work well in a variety of processes, they are frequently regarded as the best substitutes for chemical catalysts.[83] Naturally occurring lipases are useful for producing biodiesel because they can effectively perform esterification and transesterification reactions. Enzymes aid in chemical processes without being consumed or changed.[84,85] Two types of enzymes often employed in the transesterification process for the production of biodiesel are extracellular and intracellular lipases.[86] Lipases that are intracellular remain within the cells or are affixed to the cell walls, whereas extracellular lipases are recovered from microbial broths and refined for use as catalysts. Compared to traditional catalysts, enzymes have a number of benefits, such as quicker catalyst separation, lower energy consumption, greater glycerol purity, less soap generation during reactions, simpler production procedures, and the capacity to reuse immobilized enzymes.[87]

Nonetheless, these enzymes possess several disadvantages, including decreased reaction rates, increased expenses for lipase manufacture, and a reduction in catalytic activity as a result of enzyme inhibition.[88] Many bacterial species, such as *Pseudomonas fluorescens*, *Pseudomonas cepacia*, *Rhizomucor miehei*, *Rhizopus oryzae*, *Candida rugosa*, *T. lanuginosus*, and *Candida antarctica*, have been exploited as catalysts. Lipozyme *T. lanuginosus* immobilized, Lipozyme *R. miehei* immobilized, Novozym 435, and Ps-C are some of the most researched immobilized lipases.[89-92]

Throughout transesterification, catalysts are influenced by several parameters, as shown in Figure 1. These factors are divided into fundamental parameters and sub-parameters in the figure. To provide a visual depiction of how these variables impact enzyme activity throughout the transesterification reaction, Figure 1 illustrates the connections and interactions between them.

One of the many difficulties encountered in biodiesel production is the instability of free enzymes under different transesterification circumstances. In addition to complicating enzyme recycling, the instability makes it challenging to separate the reaction mixture.[93] Scientists have focused on immobilizing enzymes onto solid carriers as a solution to these challenges. By keeping the enzymes from becoming denatured, these carriers improve their chemical and thermal stability.[94] However, potential issues include decreased catalytic activity, enzyme dissociation, and structural alterations due to interactions with the support matrix. In addition, the price of the support materials may also be a constraint.[95]

Numerous lipase immobilization techniques have been investigated to enhance the generation of biodiesel. By binding enzymes to inert materials, immobilization enables them to withstand variations in temperature, moisture content, and pH. Common methods include cross-linking, trapping, encapsulation, covalent bonding, and adsorption.[96] These techniques attempt to

increase lipase stability for biodiesel production, albeit they are not covered in depth here.[97,98] Furthermore, the problems with other heterogeneous catalysts, such as high energy consumption, high manufacturing costs, and complicated operating procedures, are lessened by the enzymes.[99]

2.5. Nanocatalysts as heterogeneous catalysts

The increased efficiency of nanocatalysts over traditional catalysts has drawn a lot of interest in biodiesel production. The two types of nanoparticles employed for enzyme immobilization are magnetic and non-magnetic. Materials that are not magnetic include silica, polystyrene, chitosan, and polylactic acid. These materials are further separated into synthetic renewable resources, which are used to make chemicals and biopolymers.[100] Silica is a frequently used framework for immobilizing enzymes due to its outstanding mechanical qualities, high thermal and chemical resistance, low cost, non-toxicity, and good adaptability. The adsorption capabilities of silica are also enhanced by its large surface area and porosity, which lessen diffusion limitations.[101] In addition to noble metals, such as gold and silver, organic nanoparticles, such as metal oxides (such as zirconia, titania, and alumina) also function well as nanocatalysts. These substances are well-known for their superior adsorption properties, high stability, and strong mechanical properties. Under varying reaction conditions, the nano-metals in this category support distinct types of enzymes while remaining inert during the process.[102,103]

The inability to separate non-magnetic nanoparticles from the reaction product, a process that typically requires high-speed centrifugation, poses a significant challenge for their reuse, despite their excellent dispersion in reaction solutions. One way to overcome this limitation is by attaching magnetic oxides, such as magnetite to enzyme molecules, which facilitates catalyst recovery.[104,105] Magnetite iron oxide is beneficial due to its large surface area, biological compatibility, non-toxic nature, and hydroxyl groups that enable strong covalent bonds with enzymes. Carbon nanotubes,[106-108] nanofibers,[109] and nanocomposites[110] are other significant nanoparticles that have been researched.

Nanocatalysts provide many advantages, including superior reusability, large surface area, higher stability, greater catalytic activity, improved resistance to saponification, and effective surface-to-volume proportions.[38] The properties increase enzyme loading and reduce diffusion constraints. In an evaluation of biodiesel synthesis, sodium titanate nanotubes, for instance, demonstrated a surface area of 200 m^2/g and a pore volume of 0.61 cm^3/g. This led to a 98% biodiesel output with a methanol-to-oil ratio of 20:1[111] and a catalyst loading of 1%. The use of lipases in conjunction with nanocatalysts has also been investigated in several hybrid studies.[112-115]

Numerous techniques, such as co-precipitation, impregnation, gas condensation, chemical vapor deposition, electrochemical deposition, vacuum deposition, evaporation, microwave combustion, conventional hydrothermal processes, microwave-assisted hydrothermal and solvothermal methods, sol-gel techniques, and self-propagating high-temperature synthesis, can be used to create nanocatalysts.[38,116] However, before these methods can be employed to produce biodiesel, they must be properly characterized as catalysts. An overview of the various nanocatalysts utilized in the production of biodiesel is given in Table 3, along with information on their input sources and reaction parameters.

Table 3 presents a comprehensive overview of various nanocatalysts employed in biodiesel production from diverse feedstocks, highlighting their reaction conditions and yields. For instance, Cs/Al/Fe_2O_4catalysts utilized methanol at a 14:1 molar ratio, 58°C for 2 h, achieving biodiesel yields ranging from 88% to 95% when applied to sunflower oil. Similarly, Ca/γ/Al_2O_3 catalysts processed corn oil with methanol at a 12:1 molar ratio, 65°C for 5 h, resulting in yields between 34.64% and 87.89%. Cr/Ca/γ-Al_2O_3 catalysts used methanol at an 18:1 molar ratio, 65°C for 3 h, achieving yields from 78.29% to 92.79% when applied to cooking oil. MgO/$MgAl_2O_3$ catalysts, both untreated and plasma-treated, processed sunflower oil with methanol at a 12:1 molar ratio, 110°C for 3 h, with a yield between 79.30% and 96.50%. KOH/Fe_3O_4@Al_2O_3 catalysts employed methanol at a 12:1 molar ratio, 110°C for 4 h, achieving yields from 88.40% to 98.80% with canola oil. CaO/$CuFe_2O_4$ catalysts utilized methanol at a 15:1 molar ratio, 70°C for 4 h, resulting in a 94.52% yield with chicken fat as the feedstock. 25%MoO_3/B-ZSM-5 catalysts processed oleic acid with methanol at a 20:1 molar ratio, 160°C for 6 h, achieving yields between 93% and 98%. The significant advancements in nanocatalyst development for biodiesel production offer efficient and sustainable alternatives to traditional methods. The variation in yields across different catalysts and feedstocks highlights the importance of optimizing reaction conditions to maximize biodiesel production efficiency. The use of nanocatalysts not only enhances the catalytic activity but also facilitates the

Table 3. Various nanocatalysts for producing biodiesel from various fuel sources

Catalyst	Feedstock	Reaction operating conditions					References
		Solvent	Molar ratio	Temperature (°C)	Reaction time (h)	Yield (%)	
$Cs/Al/Fe_2O_4$	Sunflower oil	Methanol	14:1	58	2	95 – 88	117
$Ca/\gamma/Al_2O_3$	Corn oil	Methanol	12:1	65	5	87.89 – 34.64	118
γ-Al_2O_3/KI	Palm oil	Methanol	14:1	60	4	98 – 79	119
$MgO/MgAl_2O_3$ (untreated and treated with plasma)	Sunflower oil	Methanol	12:1	110	3	79.30 – 91.10	120
$MgO/MgAl_2O_3$ (untreated and treated with plasma)	Sunflower oil	Methanol	12:1	110	3	95.70 – 96.50	121
$Cr/Ca/\gamma$-Al_2O_3	Cooking oil	Methanol	18:1	65	3	92.79 – 78.29	122
$Mg/MgFe_2O_4$	Sunflower oil	Methanol	12:1	110	4	91.20 – 82.40	123
$KOH/Fe_3O_4@Al_2O_3$	Canola oil	Methanol	12:1	110	4	98.80 – 88.40	124
$CaO/CuFe_2O_4$	Chicken fat	Methanol	15:1	70	4	94.52	125
$25\%MoO_3$/B-ZSM-5	Oleic acid	Methanol	20:1	160	6	98 – 93	126

reuse of catalysts, contributing to the sustainability of the biodiesel production process.

One of the most popular methods for describing nanocatalysts is X-ray diffraction, which is used to ascertain the catalyst's composition and crystallinity. Analyzing the shape of nanocatalysts is made easier with the use of scanning electron microscopy. Thermal stability and breakdown characteristics of catalyst samples are assessed using thermogravimetric analysis, while surface functional groups are identified using Fourier transform infrared spectroscopy.[51]

One of the benefits of heterogeneous nanocatalysts is their ease of separation from the reaction mixture, as they exist in a different phase compared to the reactants and products. This simplifies product purification and enables continuous processing, while also allowing for catalyst reuse with minimal activity loss, significantly lowering operational costs. In addition, heterogeneous nanocatalysts can deliver high conversion efficiencies in many reactions, often enabling processes to proceed under milder temperature and pressure conditions compared to homogeneous systems. This not only reduces energy requirements but also enhances process safety. Furthermore, many heterogeneous catalysts are cost-effective to manufacture, and their robust nature permits extended reuse without substantial degradation. These attributes, durability, reusability, and operational efficiency, make them particularly advantageous for large-scale industrial applications.

However, these catalysts also have several disadvantages. The performance of a heterogeneous catalyst is highly dependent on its surface area, as a low surface area limits the availability of active sites, reducing catalytic efficiency, particularly in reactions demanding high activity. Mass transfer limitations can also hinder performance, especially when using large catalyst particles or operating at low fluid velocities, as they restrict reactant access to active sites and impede product removal, slowing overall reaction kinetics. In addition, certain reaction components, such as water or acidic compounds, can induce undesirable side reactions (e.g., soap formation in esterification), compromising process efficiency.

3. Biomass waste-based catalysts and their function in biodiesel production

Biomass wastes that include elements, such as carbon, silicon, calcium, magnesium, potassium, and phosphorus have been investigated for the development of inexpensive and ecologically friendly catalysts (Figure 2). This section explores the production of solid catalysts from a variety of biomass waste sources, including ashes, discarded shells, bones, and carbon-based materials from agricultural leftovers, and assesses how well they work in the transesterification process. The best catalysts for transesterification processes are highlighted by comparing the catalytic activity of catalysts made from various biomass types.

3.1. Shell wastes

When transesterifying low FFA feedstocks, CaO is a very efficient basic heterogeneous catalyst that is frequently utilized because of its low solubility, low

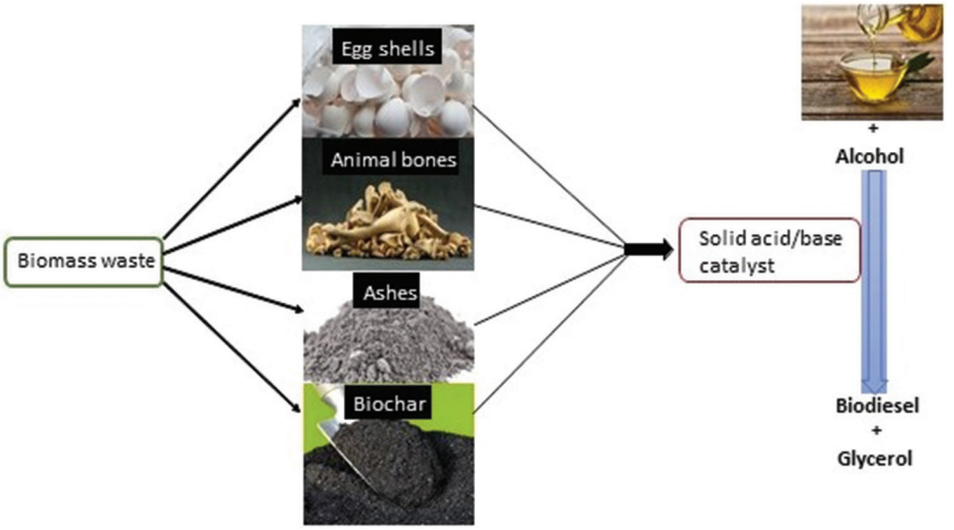

Figure 2. The manufacture of solid catalysts for the transesterification process using various biomass waste sources

cost, and high basicity.[127] The non-renewable mineral limestone is typically used to make CaO, which may be costly to generate. Utilizing discarded shells, which are high in calcium, might lessen the reliance on limestone for manufacturing CaO. Waste shells are produced in significant quantities worldwide due to the extensive consumption of mollusks and chicken eggs. A high-temperature calcination process may transform over 95% of calcium carbonate ($CaCO_3$) in these shells into CaO. During calcination, $CaCO_3$ decomposes from the outer surface inward, increasing the material's porosity and activating the resulting CaO catalyst.[128]

Calcium oxide catalysts made from shells are becoming more popular for their ability to catalyze transesterification processes due to their affordability and environmental advantages. Calcined egg shells were investigated as catalysts for the transesterification of fresh soybean oil versus waste cooking oil at ambient temperature, yielding 97 wt% and 98% FAME yields, respectively. In addition, the catalyst only showed a 10% reduction in activity after a year, and continued to exhibit catalytic activity for up to 3 months without any notable losses.[129] Similarly, Hu *et al*.,[130] discovered that CaO made from mud crab shells had catalytic activity comparable to that of commercially sold CaO.

However, there has been some variance in the catalytic activity of CaO produced from various shell species. A comparison of CaO made from egg shells, golden apple snails, and *Meretrix meretrix* shells in the process of transesterifying palmolein oil found that all of the catalysts functioned well, attaining over 90% FAME in 2 h. The driving force made from egg shells showed the maximum activity, followed by those made from *M. meretrix* shells and golden apple snails. These fluctuations in catalytic activity may be caused by variations in surface area and calcium content.[131] Therefore, it can be said that the surface area and calcium content of shell-derived CaO catalysts have a significant impact on their catalytic activity. The efficacies of several shell-derived catalysts in transesterification are compared in Table 4.

Natural CaO surfaces have been shown to deactivate when subjected to moisture and ambient carbon dioxide, forming $CaCO_3$ and calcium hydroxide, which lowers the substance's catalytic activity.[132,133] To mitigate this issue, ammonium carbonate is used in high-temperature calcination to eliminate these harmful species and improve the basicity of the catalyst.[134] Furthermore, a number of strategies have been investigated to further enhance CaO's catalytic activity, including chemical treatments, doping with alkali metals, hydration-dehydration cycles, and nanoscale particle reduction.[135,136] For instance, Jairam *et al*.[137] treated discarded quail shells with a 0.005 M hydrochloric acid solution and then calcined them for 2 h at temperatures higher than 800°C to create a CaO catalyst. By removing the thick outer layer of the cuticle, this technique resulted in a porous palisade layer that was rich in strong basic sites. During five transesterification cycles, the resultant catalyst consistently produced a 98% yield of FAME, similar to potassium methoxide (a potent homogeneous base) in terms of catalytic activity.

By increasing the surface area and the quantity of basic or acidic sites, impregnation is another efficient

Table 4. Comparative analysis of shell-derived transesterification catalysts from different biomass sources

Origin	Catalyst	Oil	Parameters of reaction				Production percentage	References
			Methanol to oil ratio	Catalyst load (%)	Time	Temp (°C)		
Scallop shell	CaO	Waste cooking oil	6:1	5	2 h	65	86	151
Mussel shell	CaO	Soybean oil	24:1	12	8 h	60	94.1	152
Eggshell	CaO	Waste frying oil	12:1	5	1 h	65	94.5	153
Eggshell	Lithium-doped CaO	*Mesua ferrera* Linn	10:1	5	4 h	65	94	154
Eggshell	M-CaO[a]	Eucalyptus oil	6:1	5	2.5 h	65	93.8	155
Turbonilla striatula	CaO	Mustard oil	9:1	3	3 h	65	93.3	156
Tourbo jourdani	CaO	Palm oil	3:1	10	7 h	80	>99	157
Eggshell	CaO	Soybean	10:1	7	3 h	57.5	93	158
Mud crab	CaO		0.5:1 (wt/wt.)	5 wt	150 min	65	98.8	159
Cockle	CaO	Palm olein	0.54:1 (wt/wt.)	4.9	180 min	65	99.4	160

Note: [a]M in M-CaO refers to zinc and copper.
Abbreviation: CaO: Calcium oxide.

way to improve catalytic efficiency. CaO from shells can be impregnated with other basic elements to increase its catalytic activity because it is naturally basic. For example, Jairam *et al*.[137] increased the surface area of calcined oyster shells from 0.19 to 6 m^2/g by impregnating them with potassium iodide. Under ideal transesterification conditions, the potassium iodide-impregnated catalyst obtained 85% conversion.[138] In an associated work, Joshi *et al*.[139] investigated the catalytic efficacy of creating a catalyst by impregnating leftover chicken eggshells containing CaO with zinc and copper to transesterify eucalyptus oil. Under ideal circumstances, pure CaO, zinc-doped CaO, and copper-doped CaO produced biodiesel yields of 68%, 93%, and 85%, respectively. The doped catalysts increased basicity and surface area and were credited with better performance.

The transesterification of low-quality materials with elevated levels of FFAs has drawn attention toward mixed metal oxides comprising transition metals.[140,141] For example, CaO derived from discarded eggshells, when supported by tungsten and molybdenum oxide, efficiently converted waste cooking oil into biodiesel.[142] Over the course of five reuse cycles, the catalyst maintained a yield of around 90% of methyl ester, having reached 96.2% in the first cycle. In addition, activated CaO produced from discarded crab shells was infused onto Na-ZSM-5 zeolite and used to transesterify neem oil at 75°C with only 15% CaO loading, resulting in a biodiesel output of over 95%.[143]

Nano-sized catalysts have demonstrated better catalytic performance than traditional heterogeneous catalysts due to their larger pore volume and increased surface area. Hydrothermal and sonochemical procedures are common ways to create nano-sized catalysts. Hoora *et al*.[144] created CaO nanocatalysts through sonication and hydration-dehydration cycles on calcined eggs, oysters, and clam waste shells. The sonicated CaO catalyst demonstrated the smallest particle size and greatest activity among these catalysts, showing considerably increased catalytic activity when methanol was used to transesterify soybean oil. Niju *et al*.[145] also produced CaO nanocatalysts using sea gastropod shells (*Chicoreus brunneus*), and when they transesterified rice bran oil at a catalyst concentration of 0.4 wt%, they obtained a 93% FAME yield. Similarly, Sun *et al*.[146] used the calcination-hydration-dehydration procedure to create a nano-CaO catalyst from leftover eggshells. In 180 min, they were able to obtain a 93.44% FAME yield from *Chlorella pyrenoidosa*. In a different investigation, CaO Nano catalysts made from leftover eggshells were doped with zinc and applied to waste

frying oil transesterification. Under ideal circumstances, this process produced a 96.74% biodiesel conversion (Johari *et al*.,).[147] To create a high-performance CaO-based catalyst that helps produce biodiesel, calcined shrimp shells in a fluidized bed reactor.

Despite their advantages over homogeneous catalysts, heterogeneous catalysts need longer reaction periods. Process intensification approaches, including co-solvent technologies, microwave heating, and ultrasound, have been investigated as ways of overcoming this issue. For example, microwave-assisted alcoholysis of used cooking oil with CaO obtained from used oyster shells produced an 87% biodiesel yield under optimal conditions.[148] The addition of co-solvents has been shown to accelerate reaction rates. For instance, CaO made from calcined snail shells generated 98% FAME in 2 h when acetone was added as a co-solvent.[149] Furthermore, ultrasound-assisted transesterification of used frying oil with eggshell-derived CaO significantly shortened the reaction time, producing 98.62% biodiesel in 39.84 min under ideal conditions.[150]

Table 4 presents a comparative analysis of biodiesel production using transesterification catalysts derived from various shell-based biomass sources. CaO, a solid base catalyst, has been synthesized from different shells and utilized in the transesterification of various oils. For instance, CaO derived from scallop shells catalyzed the transesterification of waste cooking oil, achieving a biodiesel yield of 86% under a 6:1 methanol-to-oil molar ratio, 2-h reaction time, and 65°C temperature. Similarly, mussel shell-derived CaO facilitated the transesterification of soybean oil, resulting in a 94.1% yield with a 24:1 methanol-to-oil ratio, 8-h reaction time, and 60°C temperature. Eggshell-derived CaO has also been extensively studied; for example, it achieved a 94.5% yield from waste frying oil under a 12:1 methanol-to-oil ratio, 1-h reaction time, and 65°C temperature. In addition, lithium-doped CaO from eggshells catalyzed the transesterification of *Mesua ferrea Linn* oil, yielding 94% biodiesel with a 10:1 methanol-to-oil ratio and a 4-h reaction time at 65°C. M-CaO catalysts, incorporating metals, such as Zn and Cu, derived from eggshells, were employed in the transesterification of eucalyptus oil, achieving a 93.8% yield with a 6:1 methanol-to-oil ratio and 2.5-h reaction time at 65°C. Furthermore, *Turbonilla striatula* shell-derived CaO catalyzed the transesterification of mustard oil, yielding 93.3% biodiesel under a 9:1 methanol-to-oil ratio, 3-h reaction time, and 65°C temperature. *Tourbo jourdani* shell-derived CaO achieved over 99% yield from palm oil with a 3:1 methanol-to-oil ratio, a 7-h reaction time, and 80°C temperature. Eggshell-derived CaO also facilitated the transesterification of soybean oil, yielding 93% biodiesel with a 10:1 methanol-to-oil ratio, 3-h reaction time, and 57.5°C temperature. Notably, mud crab shell-derived CaO achieved a 98.8% yield from waste oil with a 0.5:1 weight-to-weight ratio, 5% catalyst loading, 150 min, and 65°C reaction time. Cockle shell-derived CaO achieved a 99.4% yield from palm olein with a 0.54:1 weight-to-weight ratio, 4.9% catalyst loading, 180 min, and 65°C reaction time. These studies underscore the potential of utilizing various shell-derived CaO catalysts in biodiesel production, highlighting the importance of optimizing reaction parameters to achieve high yields.

3.2. Animal bones

Leftover animal bones are potentially a reasonably priced supply of CaO. Calcium phosphate is the main component of bones, making up over 40% of the bone's weight. Bones calcined at high temperatures yield beta-tricalcium phosphate and calcium oxide. Bones also include hydroxyapatite, a material that may be utilized as a catalyst or a booster for catalysts because of its high thermal stability, porosity, and wide surface area.[161,162] Given that different species have different elemental compositions, the catalytic qualities of hydroxyapatite can vary greatly depending on the source.[163] Thus, common species, including fish, pig, cow, and chicken bones, have been the main focus of research on bone-derived catalysts. Table 5 compares the efficiency of catalysts generated from different species in the transesterification process. Several studies have assessed the efficacy of biodiesel production using catalysts generated from bone.

Figure 3 illustrates how both the chemical and physical properties of heterogeneous catalysts significantly impact the transesterification process in biodiesel production. Chemically, catalysts with high basicity, such as CaO, effectively deprotonate methanol, facilitating the nucleophilic attack on triglycerides to form methyl esters and glycerol. The presence of active sites in these catalysts enhances the conversion efficiency. For instance, nanocrystalline CaO exhibits increased surface area and more active sites, leading to higher catalytic efficiency. Studies have shown that nanocrystalline CaO can achieve up to 99% conversion of soybean oil at room temperature with a 1:27 oil-to-methanol molar ratio. Physically, the surface area, particle size, and morphology of the catalyst influence its performance. Nanostructured catalysts offer increased surface area and more active

Table 5. Efficiencies of bone catalysts produced from different types of transesterification processes

Origin	Catalyst	Oil	Parameters of reaction				Production percentage	References
			Methanol to oil ratio	Catalyst load (%)	Time[a]	Temperature (°C)		
Waste ostrich bone	-----	Waste cooking oil	15:1	5	4	60	90.56	176
Quail waste head	-----	Canola oil, rapeseed oil, waste cooking oil	1:12	7	4	65	89.4 from canola oil, 91 from rapeseed oil, 91.7 from waste cooking oil	177
Waste animal bone	CaO	Palm oil	1:18	20	4	65	96.78	178
Fishbone	CaO	Waste cooking oil	15:1	5	4	---	89.33	179
Pork bone	NHAp	*Jatropha curcas*	18:1	4	5 min	-----	94	180
Fishbone	CaO	Palm oil	12:1	4.02	6.11	65	94.30	181
Cow bone	CaO	Waste frying oil	12:1	20	8	70	96	182
Aceh cow bone	CaO	Castor oil	12:1	6	4	65	58.7	183
Fishbone	CaO	Palm oil	12:1	10	4	65	77.2	184
Fishbone	CaO	Palm oil	18:1	10	2	65	90	185

Note: [a]Time is depicted as hours unless stated otherwise.
Abbreviations: CaO: Calcium oxide; NHAp: Pork bone-derived hydroxyapatite.

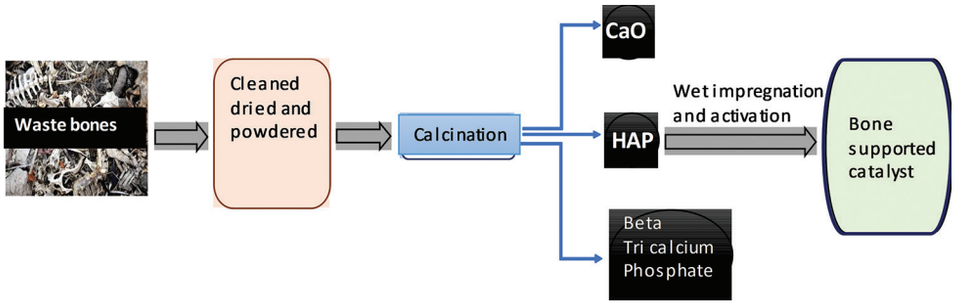

Figure 3. Bone-derived catalysts, synthesized from different types of bones, show significant efficiency in transesterification processes
Abbreviations: CaO: Calcium oxide; HAP: Hydroxyapatite.

sites, leading to higher catalytic efficiency. The choice of support material for the catalyst also plays a crucial role. Supports, such as mesoporous silica, alumina, or zeolites can enhance the surface area and stability of the catalyst. For instance, CaO/MgO supported on mesoporous silica has shown improved catalytic activity in transesterification reactions. The ability to regenerate and reuse catalysts is an important aspect of their performance. Catalysts, such as KOH supported on carbon-based materials have demonstrated high reusability, maintaining their catalytic activity over multiple cycles. Hence, both the chemical and physical properties of heterogeneous catalysts, including their basicity, surface area, active sites, and support materials, significantly impact the efficiency and sustainability of the transesterification process in biodiesel production.

The majority of the material found in fish bones that have been calcined below 600°C is hydroxyapatite. However, beta-tricalcium phosphate is formed when calcination is carried out at 800°C.[164,165] Bitire *et al*.[166] used fish bones from calcined herring subjected to calcination at 900°C for 4 h to create a beta-tricalcium phosphate catalyst. By transesterifying parsley seed oil at a 3 wt% catalyst concentration at 65°C and a

9:1 alcohol-to-oil molar ratio, this catalyst showed outstanding performance, producing a 93% biodiesel yield in 1.5 h. The catalytic qualities of bovine and chicken bones, in addition to fish bones, have been investigated in the process of producing biodiesel. The preparation of bone-derived catalysts is shown in Figure 3.

Ni *et al*.[167] obtained a 96.31% biodiesel yield using CaO derived from discarded chicken bones to transesterify waste cooking oil that was calcined at 800°C. According to them, lower calcination temperatures resulted in lower biodiesel yields.

The ideal calcination temperature must be established due to its significant impact on the catalyst's surface area and catalytic activity. Farooq *et al*.[168] examined the process of transesterifying soybean oil with catalysts made from leftover bovine bones that were calcined at temperatures between 350°C and 1,100°C. According to their findings, the ideal calcination temperature range was between 650°C and 950°C, while catalysts produced at lower calcination temperatures showed less catalytic activity. Surface area and catalytic performance were both decreased by higher temperatures. They found a clear association between surface area and calcination temperature after calcining leftover chicken bones at 800°C, 900°C, and 1,000°C for 4 h. Marzbali *et al*.[169] found that the highest performance was obtained by the catalyst that was calcined at 900°C. The findings revealed that extremely high temperatures were detrimental, even if catalytic activity rose with greater calcination temperatures. A biodiesel yield of 89.5% was produced in approximately 1.54 h under a 10:1 alcohol-to-oil molar ratio, 1.98% w/v catalyst concentration, and a reaction temperature of 65°C using a combination of fish and chicken bones that have been calcined at 1,000°C in identical weight ratios.

Nisar *et al*.,[170] used discarded quail beaks calcined at 900°C to create an extremely effective catalyst made on heterogeneous hydroxyapatite. According to their research, the catalyst's ability to effectively transesterify was caused by the increases in particle size and basic strength that occurred during calcination.

Although bone-derived catalysts are reasonably efficient in transesterification processes, their high molar ratio of methanol to oil, long reaction times, and large catalyst requirements make them unfeasible for large-scale biodiesel synthesis. Catalysts produced from bone have been modified to increase catalytic activity by improving their surface characteristics or fundamental strength. To transesterify pre-esterified *Jatropha* oil, Chen *et al*.[171] employed KOH supported on animal waste that has been calcined. Their study showed that biodiesel production increased with higher KOH loading, indicating that the catalyst containing KOH exhibited greater activity than the catalyst made from virgin calcined bone. Similarly, Olajide *et al*.,[172] reported enhanced catalytic activity in palm oil transesterification when calcined scrap pig bone was used as a catalyst impregnated with potassium carbonate, although some leaching of K^+ ions occurred during each cycle. Masango and Ngema[173] developed a bimetallic CaO-CeO_2 catalyst (1:1 ratio) supported on hydroxyapatite derived from calcined waste pig bones. This catalyst showed outstanding performance in transesterification, reaching a maximum biodiesel yield of 91.84%. Lattice distortion from Ca^{2+} and Ce^{2+} ions caused less catalyst leaching in future cycles. Waste canola oil was transesterified using a catalyst made from leftover chicken bones that included lithium, zinc, and a lithium/zinc hybrid combination. With an alcohol-to-oil molar ratio of 18:1, a catalyst concentration of 4wt%, and a temperature of 60°C, the lithium/zinc catalyst in a 2:2 ratio produced the best results, yielding 98% methyl esters in 3.5 h.[174]

In addition to modifying catalysts, other approaches should be explored to boost the efficiency of heterogeneous catalysts made from bone with an 18:1 molar ratio of methanol to oil, a 4 wt% concentration of catalysts, 800 W of microwave power, and a temperature of 65°C. Khan *et al*.,[175] used microwave heating to create a catalyst derived from the bones of Guinea fowls to produce biodiesel from *Annona squamosa* (custard apple seed) oil. They were able to convert 95.82% of FAME in 20 min.

Table 5 presents an analysis of the efficiency of bone-based catalysts derived from various animal origins for the transesterification process in biodiesel production. These catalysts, primarily CaO or hydroxyapatite, are synthesized by calcining animal bones at specific temperatures to enhance their catalytic properties. Calcined ostrich bone catalysts have been utilized in transesterifying waste cooking oil, achieving a biodiesel yield of 90.56% under a 15:1 methanol-to-oil molar ratio and 5% catalyst load for 4 h at 60°C. Similarly, quail waste head-derived catalysts have been employed for the transesterification of canola oil, rapeseed oil, and waste cooking oil, yielding 89.4%, 91%, and 91.7% biodiesel, respectively, under a 1:12 methanol-to-oil molar ratio and 7% catalyst load for 4 h at 65°C. Fish bone-derived CaO catalysts have demonstrated high efficiency, with yields ranging from 89.33% to 94.3% for waste cooking oil and palm oil under varying methanol-to-oil ratios, catalyst loadings, reaction times,

and temperatures. Pork bone-derived hydroxyapatite catalysts have been utilized for the transesterification of *J. curcas* oil, achieving a biodiesel yield of 94% with an 18:1 methanol-to-oil molar ratio and 4% catalyst load for 5 min in optimal temperature conditions. Cow bone-derived CaO catalysts have been employed for the transesterification of waste frying oil, yielding 96% biodiesel under a 12:1 methanol-to-oil molar ratio and 20% catalyst load for 8 h at 70°C. Aceh cow bone-derived CaO catalysts have been used for the transesterification of castor oil, yielding 58.7% biodiesel under a 12:1 methanol-to-oil molar ratio and 6% catalyst load for 4 h at 65°C. The potential of utilizing various animal bone-derived catalysts in biodiesel production highlights the importance of optimizing reaction parameters to achieve high yields. The choice of animal bone source, calcination temperature, and reaction conditions significantly influence the catalytic efficiency and biodiesel yield.

3.3. Ash biomass

A substantial quantity of agricultural waste produced annually across the world is burnt for fuel, producing a large amount of ash. The primary constituents of biomass, in order of abundance, are carbon, oxygen, hydrogen, nitrogen, calcium, potassium, silicon, magnesium, aluminum, sulfur, iron, phosphorus, chlorine, sodium, manganese, and titanium.[186] Burning biomass at high temperatures lowers its carbon and oxygen content, and calcining the resultant ash enhances its basicity as it contains CaO, MgO, and potassium oxide (K_2O), which are examples of alkali metal oxides. Ash derived from calcined biomass can therefore function as a basic catalyst.[187]

In India, wood has been widely used for millennia to produce heat, particularly in rural regions, which has resulted in a significant amount of wood ash, as shown in Figure 4. Due to the large amounts of plant nutrients in ash, such as lime and potash, it is frequently used as a fertilizer. Wood ash also possesses the capacity to act as a heterogeneous base catalyst due to its high alkalinity and inorganic composition. In an initial investigation, Eldiehy *et al*.[188] used a catalytic process to examine the transesterification of *Jatropha* oil from alkaline wood ash. A solid-state reaction involving double-carbonated potassium carbonate and $CaCO_3$catalyzed the activation of calcined wood ash, which was manufactured at temperatures between 500 and 1,200°C. This process resulted in the formation of activated wood ash catalysts that converted *Jatropha* oil to FAME at a rate of 97 – 99%. In their study, Kumar *et al*.[189] used an alkaline heterogeneous catalyst made from sugarcane leaf ash to transesterify *C. inophyllum* oil. The catalyst was made from the leftover ash after the silica was removed. Fourier transform infrared examination showed that MgO, CaO, and $CaCO_3$were present. Although a 22% decrease in yield was seen after 10 cycles, the catalyst attained a 97% FAME production under ideal circumstances, which included a 19:1 methanol-to-oil ratio and 5 wt% catalyst concentration at 64°C.[190]

Similar to this, walnut shell ash was investigated as a catalyst for sunflower oil transesterification, producing a 98% FAME production in 10 min. The high catalytic activity is attributed to the presence of potent basic sites, including CaO and K_2O.[191] As a heterogeneous catalyst, the transesterification of sunflower oil and *Sesamum indicum* waste from plant ash was also examined. Rich in carbonates, K_2O (29.64 wt%), and CaO (33.80 wt%), as well as sodium, magnesium, iron, manganese, zinc, silicon, strontium, and chlorine, this catalyst demonstrated exceptional catalytic activity, obtaining a 98.9% FAME yield in 40 min under ideal conditions, which included a 12:1 molar ratio of alcohol to oil and 7% concentration of catalysts at 65°C.[192]

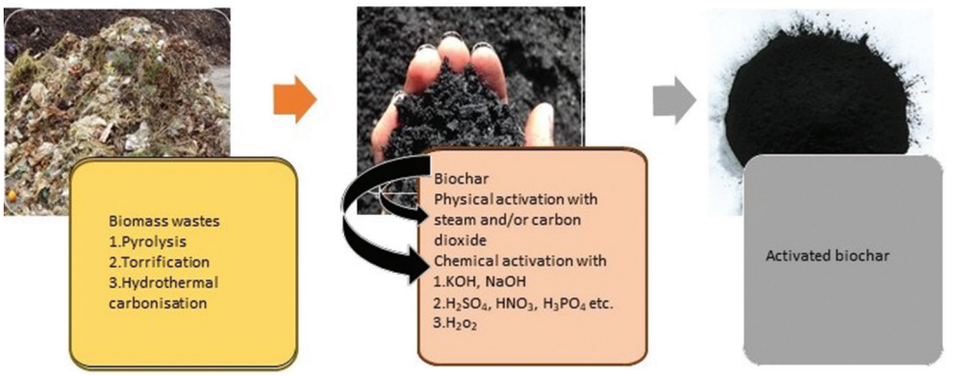

Figure 4. Various methods of converting biomass waste into activated biochar for the transesterification process

Another study examined *Brassica nigra* plant ash's catalytic activity for transesterifying different vegetable oils. High catalytic performance was established by the calcined plant waste ash catalyst, which produced biodiesel yields of up to 98.87%. According to Kordi *et al*.,[193] the catalyst's high concentrations of calcium (26.04 wt%) and potassium (56.13 wt%) as carbonates and oxides were responsible for its success. In an investigation on microwave-assisted transesterification of soybean oil, zirconium dioxide-supported bamboo leaf ash produced an ideal biodiesel yield of 92.75%.

Another interesting catalyst generated from biomass ash is rice husk ash (RHA). Approximately 200 kg of rice husk is generated for every ton of rice in rice-producing nations, such as China and India. When completely combusted, they yield about 50 kg of RHA. RHA could be a potential catalyst or pre-cursor for amorphous silica since it is mostly made up of silicon dioxide (87 – 99%).[194,195] Biodiesel generation was studied using an RHA-based basic sodium silicate catalyst created by applying NaOH to the calcined ash. The catalyst reached a 97% FAME yield in 30 min while operating at optimal conditions, which included a 12:1 molar ratio of methanol to oil and a 2.5 wt% loading of catalysts at 65°C.[196] According to Dhawane *et al*.,[197] a lithium-modified RHA catalyst with high activity and basic strength of more than 15 m^2/g converted 99.5% of the biodiesel at the molar ratio of methanol to oil of 24:1 and 4 wt% loading of catalysts at 65°C within 3 h. In addition, to produce heterogeneous catalysts that showed exceptional catalytic action in used frying oil transesterification, alkali metal hydroxides were impregnated onto silica from rice husks. The methyl esters generated by these catalysts ranged from 96.5% to 98.2% when the following parameters were met: 65°C, a 9:1 methanol-to-oil ratio, 3% catalyst loading, and an hour reaction period. High yields may be obtained for up to six consecutive cycles without the need for reactivation due to the catalysts' exceptional reusability.[198]

The calcined eggshell, which contains CaO, was also loaded onto RHA to generate a new heterogeneous base catalyst. The maximum catalytic activity was shown by this catalyst, which had 30% eggshell loading on RHA calcined at 800°C. After eight cycles, it maintained more than 80% efficiency and produced 91.5% biodiesel.[199] A comparison of catalysts made of biomass ash from various sources and how well they operate in the synthesis of biodiesel is tabulated in Table 6.

Table 6 presents a comparative analysis of various biomass-derived ash catalysts utilized in transesterification for biodiesel production. These catalysts, primarily composed of metal oxides, such as CaO, MgO, and K_2O, have demonstrated significant catalytic activity, offering a sustainable alternative to conventional homogeneous catalysts. Tamarind fruit shell ash, containing CaO, MgO, and K_2O, achieved a biodiesel yield of 96.2% from *Parinari curatellifolia* seed oil under a 9:1 methanol-to-oil ratio and 5% catalyst load at 60°C for 2 h. Similarly, pineapple leaf ash, rich in alkaline and alkaline earth metals, produced over 98% yield from soybean oil with a 40:1 methanol-to-oil ratio, 4% catalyst loading, and a 30-min reaction time at 60°C. RHA has been extensively studied for its catalytic properties. One study reported a biodiesel yield of 99.5% from soybean oil using rice husk ash as a catalyst under a 24:1 methanol-to-oil ratio, 4% catalyst loading, at 65°C for 3 h. Another study utilized sodium silicate derived from RHA, achieving a 97% yield from palm oil under a 12:1 methanol-to-oil ratio, 2.5% catalyst loading, at 65°C for 30 min. Sugarcane bagasse ash, when combined with CaO, has also shown promising results. A study demonstrated a 93.8% biodiesel yield from palm oil using silicon dioxide/CaO derived from sugarcane bagasse ash, under a 20:1 methanol-to-oil ratio, 6% catalyst loading, at 65°C for 3 h. Wood ash catalysts, both calcined and activated, have been effective in transesterifying *Jatropha* oil. The methyl ester conversion achieved ranged from 97% to 99%, depending on the specific catalyst preparation and reaction conditions. The potential of biomass-derived ash catalysts in biodiesel production, highlighting their efficiency, sustainability, and cost-effectiveness. The variability in yields across different studies emphasizes the importance of optimizing catalyst preparation methods and reaction conditions to maximize biodiesel production efficiency.

3.4. Carbon-based catalysts

Biochar, a carbon-rich compound made from biomass, is frequently utilized as a catalyst support or to make carbon-based heterogeneous catalysts. Carbon-based catalysts and rock derived from biomass are not typically categorized as biocatalysts. These are materials created from biomass (plant matter) through processes, such as pyrolysis or carbonization. They can be used as catalysts or catalyst supports in various reactions, including those related to biofuels, energy production, and chemical processes. Their benefits include a vast surface area, structural stability, remarkable mechanical and thermal qualities, and affordability.[212] Carbon-rich biomass sources, including wood, coconut shells, and

Table 6. Catalytic efficiencies of different types of biomass ashes in transesterification

Source	Catalyst	Oil	Methanol/ oil ratio	Catalyst load	Time	Temperature	Yield (%)	References
Tamarindus indica fruit shell ash	CaO, MgO, K_2O	*Parinari curatellifolia* seed oil	9:1	5	2 h	60°C	96.2	200
Pineapple leaves ash	Alkaline and alkaline earth metals	Soybean oil	40:1	4	30 min	60°C	>98	201
Acai seed ash	Metal oxides and carbonates	Soybean oil	18:1	12	1 h	100°C	98.5	202
Sugarcane bagasse ash/ CaO	Silicon dioxide/CaO	Palm oil	20:1	6 wt	3 h	65°C	93.8	203
Rice husk	------	Soybean oil	24:1	4 wt	3 h	65°C	99.5	204
Rice husk	Sodium silicate	Palm oil	12:1	2.5 wt	30 min	65°C	97	205
Waste of *Brassica nigra*	Carbonate, calcium oxide, and potassium oxide	Soybean oil	12:1	7 wt	25 min	65°C	98.8	206
Sesamum indicum	-----	Sunflower oil	12:1	7 wt	40 min	65°C	98.9	207
Walnut shell	----	Sunflower oil	12:1	5 wt	2 h	60°C	98	208
Rice husk ash	----	Refined palm oil	9:1	7 wt	4 h	65°C	91.58	209
Sugarcane leaves	----=	*Calophyllum inophyllum* oil	19:1	5wt	----	64°C	85 – 97	210
Wood ash	Calcined wood ash and activated wood ash	*Jatropha* oil	-----	1 – 3 mass fraction	30 – 210 min	65°C	97 – 99	211

Abbreviations: CaO: Calcium oxide; K_2O: Potassium oxide; MgO: Magnesium oxide.

sugarcane bagasse, are used to make biochar, as shown in Figure 5. Pyrolysis, torrefaction, hydrothermal carbonization, and gasification are the main processes used to produce biochar, with slow pyrolysis being the most popular technique due to its high biochar yield.[213]

Biochar is an ideal pre-cursor of catalysts that are carbon-based due to its high carbon content, broad surface area, exchange capability of cations, and stable structure. Functionalization is also made possible by its broad aromatic ring structure and high degree of cross-linking.[214] By affixing basic or acidic atoms to its surface, pyrolysis can be modified. The most popular sulfonating reagent is sulfuric acid, which is usually covalently bonded to the carbon surface to produce acid-functionalized activated carbon. There are two methods of sulfonation: reductive alkylation/ arylation[215] and direct sulfonation.[216] Like concentrated H_2SO_4, sulfonated activated carbon catalysts work well for producing biodiesel by esterifying FFAs or by esterifying and transesterifying at the same time.

According to Mardhiah *et al*.[216] and Feng *et al*.,[217] a sulfonated biochar catalyst derived from the leftover seed cake of *J. curcas* demonstrated better esterification catalytic activity than traditional sulfuric acid with shorter reaction times. In the fourth cycle, the catalyst achieved an 81.03% conversion yield while maintaining excellent stability over the cycles.[218] Using fuming and intense sulfuric acid, Kumar *et al*.[219] created two catalysts made from sulfonated biochar. Although the sulfuric acid-concentrated catalyst sulfonated exhibited moderate activity in transesterification, it was highly effective in esterifying FFAs in waste vegetable oil. Transesterification activity was greater in the fuming H_2SO_4-sulfonated catalyst. Another research yielded 95.6% biodiesel in 2 h with a 2% catalyst load and 9:1 methanol-to-oil molar percentage at 85°C using biochar made from pyrolyzed sawdust and sulfonated with strong H_2SO_4.[122] A 98% FAME conversion was obtained

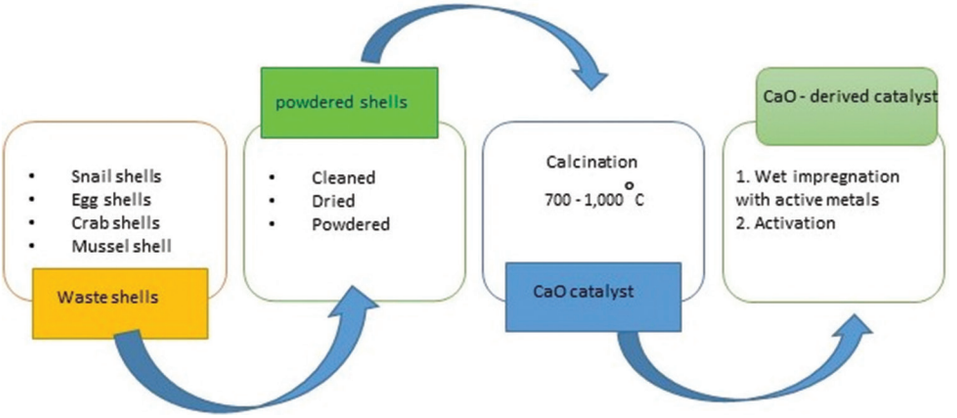

Figure 5. The method of turning waste shells into carbon-based catalysts
Abbreviation: Cao: Calcium oxide.

at a 25:1 molar ratio of alcohol to oil, 1.5% w/v loading of catalysts, and 65°C using extremely concentrated $H_2SO_4$to activates leftover cork charcoal, demonstrating excellent efficiency in transesterifying waste cooking oil.[220] A 98.1% biodiesel yield at a 20wt% catalyst concentration in 7 h was obtained by esterifying palm fatty acid distillate using charcoal from oil palm empty fruit bunches sulfonated with 4-benzenediazonium sulfonate.[221] Utilizing 4-chlorobenzenesulfonic acid as the sulfonating agent, Li *et al*.[222] created a hydrophobic trigger for benzene sulfonic acid made from biochar that outperformed Amberlyst-15 and sulfonated biochar in terms of catalytic efficiency and recyclability in FFA esterification. Zhou *et al*.,[223] used a microwave reactor for pyrolysis sulfonation in a unique way. This resulted in an increased use of sulfonic groups at 140°C, which enhanced the acidity and catalytic efficiency and produced 90% FAME in transesterification. It has also been possible to create base-functionalized carbon particles by impregnating the surfaces of biochar using pre-cursors that include calcium, potassium, or sodium. There have been transesterification reactions using these catalysts. A study investigated activated pomelo peel biochar loaded with potassium carbonate (activated at 600°C) as a catalyst for palm oil transesterification. The greatest catalytic performance was demonstrated by the catalyst that produced 98% FAME with 25 wt% potassium carbonate. However, due to the leaching of potassium ions during the first cycles, the catalyst's basic strength gradually declined.[224] The synthesis of biodiesel was also studied using a date palm-based biorefinery technique. To create a catalyst modified with CaO, date pits, which are rich in oil, were utilized as feedstock, and the waste residue was carbonized. Under ideal circumstances(molar ratio of methanol to oil of 12:1, 4.5 wt% catalyst loading, 70°C, 120 min), this catalyst produced a 98.2% FAME yield.[101] After modifications using alkaline earth metal oxides (CaO, MgO, barium oxide, and strontium oxide), residual date seed powder was employed to make activated carbon, which was then transformed into a bifunctional catalyst that contained basic and acidic surfaces. Due to the greater concentration of basic sites, the strontium oxide-supported catalyst demonstrated the best efficiency. It has been shown that during the transesterification of high FFA, low-quality oils, acidic sites promote the esterification of FFAs, which increases the output of biodiesel, while basic sites facilitate the transesterification of triglycerides.[225]

Although activated carbon-based catalysts have shown promising catalytic performance in the production of biodiesel using biomass, issues, such as prolonged reaction times and catalyst leaching still exist, restricting their reusability. To solve these problems and maximize the usefulness of these catalysts, more investigation is required.

3.5. The rocks

The generation of biodiesel has been investigated using rocks, such as dolomite, lime, and clays as possible catalysts. The naturally occurring rock dolomite is made up of alternating layers of magnesium carbonate and $CaCO_3$. These substances break down into magnesium MgO and CaO when heated to around 750°C.[226] CaO and MgO can be obtained from the thermal processing of $CaCO_3$ and dolomite.

Dolomite has garnered more interest in the past 10 years as a catalyst to produce biodiesel, specifically for the transesterification of C4-C8 oil from olives. Because of its affordability, it is commonly utilized in sectors,

such as landfilling and cement production. Dolomite's catalytic efficacy is increased when it is calcined, giving it a larger surface area than when it is uncalcined in nature. The application of calcined dolomite as a catalyst has been the subject of several studies.[227,228] The range of reported FAME transformation yields using dolomite is 92% to 99%. Furthermore, structural modifications to dolomite by hydrating and dehydrating have been investigated and shown to boost FAME transformation yields from93% to 97.4%.[229,230]

4. Methods of catalyst preparation

Heterogeneous catalysis in transesterification involves three separate phases: a solid catalyst and two immiscible liquids (methanol and oil). This makes the process complicated, and side reactions, such as the neutralization of FFAs with glycerol and methyl esters, may also occur throughout the process.[231-233]

Numerous techniques for creating solid catalysts for transesterification have been discussed in the literature. These consist of impregnation, heat treatment, physical mixing, and hydrothermal synthesis. The specific physical and chemical characteristics required for the final catalyst determine the selection of the synthesis method to be used.[234]

4.1. Catalyst selection

Metal oxides are Lewis acidic and Brønsted basic because they are composed of negatively charged anions and positively charged metal cations. These properties make metal oxides essential for the transesterification procedure, methanolysis of oils, which produces biodiesel. Methanol binds to metal oxides in large quantities, where the O-H link can be broken to produce hydrogen cations and methoxide anions.

Catalyst preparation techniques have been studied in the literature. In the synthesis of biodiesel, for instance, it has been discovered that adding a small quantity of water increases the catalytic action of CaO.[235] Oxygen on the catalytic surface releases hydrogen ions in the presence of water, forming hydroxide ions. These hydroxide ions then release hydrogen ions from methoxide anions, which serve as the crucial catalytic species in the transesterification process.[236] According to noteworthy research, methanol adsorption is a crucial rate-determining step in processes combining MgO and lanthanum(III) oxide. Higher basicity catalysts, such as barium oxide, CaO, and strontium oxide, have a rate-determining step that is connected to the surface reaction.[237]

More than 13 different metal oxides have been investigated in the production of biodiesel by transesterification. These oxides include calcium, barium, magnesium, and lanthanum oxides. The findings imply that catalysts derived from calcium exhibit higher catalytic activity within this procedure. Since CaO is readily available, inexpensive, and has a lower toxicity than other catalysts, it is particularly preferred for the production of biodiesel.[238,239]

4.2. Calcination application

One method that is frequently used to create catalysts from biomass is calcination. By applying heat without the presence of air or oxygen, biomass is broken down into smaller components.[240] Temperatures for the calcination process usually range from 300°C to 1,000°C, depending on the type of material used. Carbon dioxide gas is released as molecules, such as $CaCO_3$ break down into CaO during this procedure.

The solid base catalysts made from biomass, created using calcination, for the manufacture of biodiesel by transesterification are summarized in Table 7. The particular feedstocks utilized, the types of biomass employed, the reaction conditions, and the related references are all listed in the table. A key factor in defining the surface shape of the CaO catalyst is the calcination temperature. Many waste materials, such as shells, are originally non-porous; however, during calcination, pores form on their surfaces, which can significantly impact the final catalyst's surface area. The calcination temperature changes the distribution and intensity of active sites, impacting a catalyst's catalytic activity.[241] Numerous investigations, including those conducted by researchers, have examined the impact of calcination on catalyst production.[242-244]

Table 7 presents the elemental composition of various solid base catalysts derived from agricultural and organic wastes through calcination processes. The data reveal significant variations in alkali and alkaline earth metal contents, which are crucial for catalytic activity in transesterification reactions. Potassium emerges as the predominant component in most catalysts, particularly in *Musa* spp. peduncle (68.37%) and *Carica papaya* stem (56.71%), while calcium shows notable concentrations in *Tectona grandis* leaves (30.28%) and *S. indicum* (33.80%). The calcination conditions (500 – 900°C for 2 – 4 h) significantly influence the final catalyst composition, with higher temperatures generally enhancing metal oxide formation. Sweet potato leaves calcined at 900°C contain 65.45% potassium, whereas *Moringa* leaves

Table 7. Composition of various solid base catalysts derived from waste biomass

Source of catalyst	Calcination condition	Composition (%)								References
		Sodium	Potassium	Calcium	Magnesium	Aluminum	Silicon	Phosphorus	Carbon	Oxygen	
Musa spp. peduncle	700°C, 4h	-	68.37	7.09	4.66	-	-		-	19.88	245
Carica papaya stem	700°C, 4h	14.78	56.71	21.08	4.41	-	-	-	-	-	246
Sweet potato leaves	900°C, 3h	0.70	65.45	18.02	4.63	0.94	3.54	-	-	-	247
Ripe-unripe plantain ash	500°C, 4h	-	45.16	-	3.61	-	3.87	-	12.02	35.34	194
Tectona grandis leaves	700°C, 4h	1.67	53.25	30.28	4.77	-	10.03	-	-	-	248
Potato peel	700°C, 3h	1.85	36.54	2.71	2.02	-	3.06	-	9.18	41.55	249
Poovan banana pseudostem	700°C, 4h	0.4	20.2	7.4	4.52	0.25	3.79	1.91	-	-	250
Citrus sinensis peel	Burnt	-	8.95	5.01	1.30	-	-	-	-	37.20	251
Heteropanax fragrans	550°C, 2h	-	19.05	5.13	0.86	0.44	8.51	0.64	16.71	46.74	252
Pawpaw peel	600°C, 4h	0.00	23.89	2.86	1.00	-	0.00	3.04	29.16	36.72	216
Moringa leaves	500°C, 2h	-	9.87	10.09	5.92	-	-	1.19	12.19	59.57	247
Sesamum indicum	550°C, 2h	1.42	29.64	33.80	9.68	-	11.32	-	-	-	253
ACP-550	550°C, 2h	-	49.13	2.85	2.61	-	4.33	-	4.07	37.00	254

processed at 500°C retain more organic content (59.57% oxygen). The presence of multiple active components (potassium, calcium, and magnesium) in catalysts, such as *S. indicum* (potassium: 29.64%, calcium: 33.80%, and magnesium: 9.68%) suggests potential synergistic effects in catalytic applications. Carbon content varies substantially (1.91 – 29.16%) in *Citrus sinensis* peel and pawpaw peel, showing higher values, which may affect the surface area and porosity. The data underscores the importance of pre-cursor selection and processing conditions in tailoring catalyst composition for specific biodiesel production requirements. These waste-derived catalysts demonstrate the potential for sustainable catalyst development by valorizing agricultural byproducts.

4.3. Hydrothermal process

One popular technique for preparing catalysts is the hydrothermal process. To produce differences in crystal shape, the solution must be heated and mixed. As an example, zinc oxide nanorods with a mean width of 38 nm and a length of 230 nm are produced when a zinc nitrate solution is heated. Materials that are normally insoluble under normal circumstances can be dissolved and recovered using a process called hydrothermal synthesis.[255] Quick processing, excellent yields, affordability, and ecological responsibility are just a few of the benefits that make this approach popular.[256] Because of the increased surface contact between the solvent and the soluble particles; the final product of hydrothermal treatment closely resembles the original stoichiometric composition. Greater reaction temperatures promote mass transfer and molecular diffusion rates by decreasing the solvent's viscosity and increasing particle solubility.[257] This produces extremely crystalline nanoparticles that are very pure and have a limited size distribution, frequently obviating the need for further calcination. Particle size, shape, crystal structure, pressure, and reaction duration are just a few of the catalyst parameters that may be precisely controlled using this method.[258]

A number of researchers have effectively manufactured catalysts using the hydrothermal technique. Continuous hydrothermal synthesis, for instance, has been used to create nanoparticles modified by oleic and decanoic acids. This approach has produced positive results for other researchers.[259-262]

4.4. Impregnation process

Heterogeneous catalysts can be prepared through the impregnation process, which involves applying a solution to a catalyst support that contains metal.

The impregnation process is also known as capillary and dry impregnation. The solution is absorbed into the supporting material and then dried to remove the solvent from its substrate.

The two main categories of this technique are wet impregnation and dry impregnation. The size of the solution and the method of solid-solution interaction are the primary distinctions between the two. Wet impregnation uses an excess solution that is then dried after a pre-determined amount of time. In dry starting wetness impregnation, the quantity of solution utilized is at least as large as the amount of pore space of the active phase or sustaining solid.[263,264] A number of variables, such as temperature and the starting solution's concentration, might impact the effectiveness of the impregnation methods.[265,266] Wetting time, solution viscosity, and pre-cursor solubility are all impacted by temperature.[267,268] Studies have successfully used the impregnation approach, such as the creation of potassium fluoride/CaO nano-catalysts.[269,270] For instance, to transesterify soybean oil, a lithium/zinc oxide catalyst was made through impregnation and calcination. Catalytic activity and catalyst characteristics were shown to be correlated in the study, which also noted that the calcination temperature and lithium loading affected lithium/zinc oxide performance.

Benefits of the impregnation process include reduced expenses, quicker preparation, and improved control over the end-product catalyst properties.[271] However, achieving a homogeneous catalytic component dispersion on the support surface is a difficult task.

4.5. The sol-gel process

The sol-gel method serves as a versatile approach for both catalyst synthesis and feedstock preparation. This wet-chemical technique involves the transition of a colloidal suspension (sol) into a continuous three-dimensional network (gel), enabling the production of homogeneous, high-purity materials with controlled porosity. The process begins with nanoparticle dispersion in the liquid phase, followed by controlled agglomeration and gelation, making it particularly valuable for creating tailored catalytic materials and pre-cursor formulations. A popular technique for catalytic feedstocks having colloidal characteristics, such as silicon dioxide, the sol-gel method is comparable to the precipitation process.[272] The sol-gel procedure ensures a consistent distribution of components and allows for the fabrication of porous materials, in contrast to conventional catalyst preparation methods. Usually, inorganic pre-cursors or colloidal dispersions are used to create a sol and a gel. Due to its great versatility, the approach may work with a wide range of pre-cursors, most frequently alkoxides (M(OR)n)-.[269]

A solid-particle-based colloidal solution called the sol and a continuous liquid phase called the gelation phase are the two main stages of the sol-gel process.[273,274] Numerous benefits, including excellent yields, low temperatures of operation, and cost-effectiveness, are provided by the sol-gel technique.[275] This method has been used effectively in many research studies, such as those by Sharm *et al*.,[276] Ciesielczyk *et al*.,[277] Danks *et al*.,[278] Navas *et al*.,[279] and Rahman *et al*.,[280] Based on the following equations, the sol-gel process usually involves three primary routes: hydrolysis (Equation I), condensation (Equation II), and mixed condensation (Equation III).

$$-MOR+H_2O\rightarrow-MOH+ROH \quad \text{(I)}$$

$$-MOH+ROM-\rightarrow-MOM+ROH \quad \text{(II)}$$

$$-MOH+HOM-\rightarrow MOM+H_2O \quad \text{(III)}$$

4.6. Co-solvent preparation method

A co-solvent system consists of water mixed with a miscible or partially miscible organic solvent, creating a modified aqueous medium. This phenomenon, termed cosolvency, relies on the solvent's dual characteristics: hydrogen-bonding capacity (either as donor or acceptor) and the presence of hydrophobic domains. These properties enable co-solvents to modify solution thermodynamics and enhance the solubility of diverse compounds. To increase catalytic activity and accelerate the catalyzed processes, the co-solvent approach is a catalyst preparation methodology that overcomes limitations in mass transfer between oil and methanol.[281] In several transesterification processes, tetrahydrofuran is frequently employed as a co-solvent in co-solvent catalyst systems. The effectiveness of this strategy has been shown in a number of investigations, with the co-solvent technique producing FAME yields that range from 90% to 98.5% conversion.[282,283]

5. The impact of heterogeneous catalysts' chemical and physical characteristics on biodiesel production

The functionality of catalysts that are uniform, which usually appear in the liquid state, differs from that of catalysts that are heterogeneous and have a solid shape. Although homogeneous catalysts are readily accessible, reasonably priced, have quick processing times, and

rapid reaction rates for the manufacture of biodiesel, the use of H_2SO_4 raises the sulfur concentration, perhaps surpassing the 10 ppm biodiesel requirement.[284] However, using NaOH and KOH to produce biodiesel adds extra procedures, such as product separation, liquid catalyst recovery, and acid washing to neutralize the product. These additional processes result in more effluent, which raises production costs and prevents biofuels and biodiesel from becoming commercially available.[285]

The calorific value of biodiesel, a key indicator of its energy density, demonstrates notable variation across different production technologies, primarily due to differences in chemical conversion efficiency and byproduct formation. Alkaline-catalyzed transesterification, the most widely adopted commercial method, typically produces biodiesel with calorific values of 37 – 40 MJ/kg, as it effectively maintains the original fatty acid profile of the feedstock while achieving near-complete conversion. In contrast, enzymatic processes, despite their environmental advantages and mild operating conditions, often yield slightly reduced energy content (36 – 39 MJ/kg) because of incomplete separation of glycerol co-products and residual catalyst components. Supercritical methanol methods demonstrate superior performance (38 – 41 MJ/kg) through their ability to achieve nearly quantitative conversion of both triglycerides and FFAs without catalyst residues. The lower energy output from acid-catalyzed routes (35 – 38 MJ/kg) stems from ester bond hydrolysis and subsequent side reactions that alter the molecular structure of the biodiesel. Notably, hydro processing technologies achieve the highest energy densities (39 – 42 MJ/kg) by fundamentally transforming the biodiesel chemistry through oxygen removal and hydrocarbon saturation, producing molecules that more closely resemble conventional diesel. These calorific value differences, when considered alongside production costs and scalability, provide crucial guidance for selecting appropriate biodiesel manufacturing technologies based on specific application requirements.

Biodiesel's elevated kinematic viscosity (typically 4 – 6 mm^2/s at 40°C versus petroleum diesel's 2.5 – 3.5 mm^2/s) presents both challenges and considerations for fuel system design and operation. The increased viscosity directly affects fuel injection dynamics, resulting in larger droplet sizes (15 – 30% greater than conventional diesel) that impair air-fuel mixing and combustion efficiency, potentially increasing particulate emissions by 10 – 20%. This viscosity effect becomes particularly problematic in cold-weather operations, where biodiesel's viscosity can exceed 15 mm^2/s at temperatures below 0°C, dramatically raising the pumping work required and increasing the risk of fuel filter blockage due to gelling. Modern common-rail injection systems demonstrate better tolerance to biodiesel blends up to B20, as their higher injection pressures (2,000 – 3,000 bar) can partially compensate for the viscosity difference. However, extended use of pure biodiesel (B100) typically necessitates system modifications, including replacement of natural rubber components with Viton or other biodiesel-resistant elastomers, installation of fuel heaters for cold-climate operation, and potential recalibration of injection timing to account for altered combustion characteristics. These viscosity-related effects must be carefully evaluated when implementing biodiesel in existing fleets, as the required modifications can significantly impact the economic feasibility of biodiesel adoption in legacy equipment.

Numerous issues, such as the use of uniform catalysts in the utilization of heterogeneous catalysts, can optimize biodiesel manufacture.[29,286] Despite potentially causing longer response times, they help to minimize toxicity, corrosion, and energy consumption. Recent developments enable the simultaneous facilitation of transesterification and esterification through the utilization of heterogeneous catalysts in dual processes. This feature allows the synthesis of biodiesel without the need for an additional pre-treatment procedure to reduce the amount of FFAs.[287] In an effort to counteract the prominence of homogeneous catalysts, the increasing use of alternative and reused feedstocks, such as discarded greases or fats from animals with higher FFA levels, has brought attention to the benefits of heterogeneous catalysts. To prevent soap production, which can result in emulsification and other downstream problems during transesterification, homogeneous catalysts are limited to feedstocks with an FFA level of <0.5% w/w.

Figure 3 illustrates how the transesterification process can be significantly impacted by the properties of heterogeneous catalysts, both chemically and physically.[38,288] The reactor's configuration and the quality of the feedstock are additional elements that affect heterogeneous catalysis.[289,290] Since more oil results in a larger output of biodiesel, the quality of the feedstock has a direct impact on the yield. Regardless of the catalyst utilized, the reactor's design is also crucial in determining the price and volume of manufacturing. Numerous studies have examined the use of alkaline

earth oxides in transesterification processes, with particular attention to the effects of oxides of calcium, magnesium, strontium, and barium that have been treated at elevated temperatures for reactions between 500°C and 1,050°C.[291,292] In using methanol to transesterify vegetable oil under reflux circumstances, CaO has been identified as an efficient catalyst.[293-295] These investigations show that CaO's fundamental features enable it to speed up transesterification processes mainly through a nucleophilic mechanism.

Other compounds, such as calcium hydroxide, $CaCO_3$, MgO, and barium hydroxide have also been shown to exhibit comparable behavior, demonstrating the well-documented nucleophilicity of CaO, which increases its susceptibility to nucleophilic assaults. Higher calcination temperatures (270°C) have been demonstrated to activate more sites, especially for MgO. Research showed that strontium oxide catalysts, which were produced by thermal decomposition at 1,200°C, had lengthy catalytic lifetimes and unusually high FAME yields, with the ability to be reused for up to 10 cycles.[296] Due to its economic viability, strontium oxide is a promising catalyst that might lower the cost of producing biodiesel.

5.1. Impact of base-and acid-catalyzed reactions on methyl esters of fatty acid production

The abundance of acid and basic sites (or surfaces) of both types of catalysts has a major impact on their catalytic activity and selectivity. However, there are difficulties in determining these densities throughout the process of transesterification. Compared to acid catalysts, solid base catalysts are often recognized for their greater reactivity and quicker reaction rates.[297] Despite these benefits, base catalysts are extremely vulnerable to FFAs and water. Base catalysts need minimal FFA feedstocks to prevent catalyst deactivation and guarantee effective biodiesel production.[298] On the other hand, for catalysts made of solid acid to provide FAME yields that are acceptable, greater reaction temperatures and catalyst loadings are necessary, despite being able to tolerate high water and FFA concentrations.[299,300]

Methyl esters of fatty acids are produced through the transesterification of triglycerides, and the use of biomass-derived heterogeneous catalysts is a significant area of research aimed at enhancing the sustainability and cost-effectiveness of this process. The production of FAME is directly linked to biodiesel synthesis, as FAME constitutes the bulk of biodiesel's composition. This relationship underscores the importance of optimizing FAME production processes to enhance biodiesel yield and quality. Advancements in catalyst development, such as the use of biomass-derived heterogeneous catalysts, aim to improve the efficiency and sustainability of FAME production, thereby contributing to more cost-effective and environmentally friendly biodiesel production methods. In essence, the review likely explores how biomass-derived heterogeneous catalysts can improve the efficiency, sustainability, and economic viability of FAME production, which is the foundation of biodiesel synthesis. Both the chemical and physical characteristics of the distinct fatty acid esters in biodiesel dictate its qualities. Thus, the ultimate product is largely determined by the solvent employed, the feedstock's molecular structure, and its content. Significant research has been conducted over the past 20 years to find catalysts that produce certain FAME profiles and attributes.[301-304] Although catalysts' effects on yield have been well investigated, it is still not known how they affect the qualitative characteristics of biodiesel.[305,306] Consequently, the comprehension of catalyst activity under dynamic reaction settings remains a challenge for researchers.[307] In addition, the literature emphasizes various heterogeneous solid catalysts for acids and bases utilized in transesterification, such as salts supported on hydrotalcites, zeolite, alumina, alkali metals, hydroxides, and alkaline earth metal oxides. The transesterification of vegetable oils using solid base catalysts has been extensively studied, with various reaction parameters reported in the literature. As an example, high reactivity was demonstrated at low temperatures by potassium iodide, potassium fluoride, and potassium nitrate catalysts based on alumina, as salt reactions supported aluminum-oxygen-potassium groups or K_2O species to generate basic sites.[308] By ionizing sodium or potassium during transesterification, Na/NaOH/γ-Al_2O_3 and K/KOH/γ-Al_2O_3 catalysts also created strong base sites.[309] Nonetheless, leaching was shown to be a problem, suggesting chemical instability under the circumstances of the reaction.[310] However, calcium nitrate/aluminum oxide showed good efficiency of conversion and stability during transesterification, making it a viable catalyst for the manufacture of biodiesel in the future.[311]

5.2. Impact of solid catalysts on transesterification reactions

The interplay between acidic and basic sites determines a material's acidity. A solid acid catalyst must be capable of proton donation or transfer for it to interact with surface anions with regard to Brønsted acidity. Furthermore, when interacting with base molecules on

its outermost layer, a solid acid catalyst should take in an electron pair and create a coordinating bond. During transesterification, this bond formation encourages the development of electrophilic species, which have a major impact on the desorption and total reaction rates.[312] Strong acid sites, on the other hand, may cause a poor desorption rate, which would slow down the transesterification process.[313]

5.3. The temperature of the catalyst calcination impacts the production of methyl esters of fatty acids

Crucial catalytic qualities, including acidity, density, quantity of pores, surface areas, durability, and chemical and crystalline structures, are all influenced by the calcination temperature. By eliminating carbon dioxide and dissolved water molecules and altering the catalyst's bulk atoms and surface structure, increased calcination temperatures aid in the exposure of catalytic sites.[314]

According to research, the calcination effectiveness of catalysts is significantly influenced by temperature. As an illustration, calcining cesium zirconate/aluminum oxide for 4 h at 250 – 350°C raises FAME production from 62% to 90%.[315] Excessively high calcination temperatures, however, may have adverse effects by decreasing catalytic activity and FAME production. This decrease is explained by the dispersal of gases and the extraction of trapped water molecules through the catalyst arrangement through the catalyst's surface pores, which restricts pore availability.[316]

5.4. Reactor operation conditions and design impact

Optimizing the utilization of diverse catalysts for the production of biodiesel requires careful consideration of reactor architecture and conditions of operation. Most commercial biodiesel production now takes place in batch mode, making around 7,000 tons annually.[317,318] However, batch processing has drawbacks, such as separation difficulties, expensive capital expenditure, personnel expenses from start-stop operations, and problems with scalability (8,000 – 12,500 tons annually). To esterify FFAs, this has sparked an increasing interest in switching to continuous flow reactors,[319] such as reactive distillation,[320] pervaporation methods,[321] microchannel stream reactors,[322] and fixed-bed fluid reactors.[323]

Through constant water removal from the system, dynamic distillation streamlines production, reduces costs, and prolongs catalyst life by merging the chemical method that combines the processes of reaction and segregation. Effectiveness depends on the process's compatibility with the pressure and temperature requirements for distillation. The exact control over product composition provided by continuous reaction reactors, including plug flow reactors lowers separation costs and increases investment efficiency. However, the requirement for large length-to-diameter ratios for adequate mixing might cause slower reactions in plug flow reactors during FFA esterification and triglyceride transesterification.[324] By oscillating the reaction fluid over baffle plates, oscillatory baffled reactors can increase mixing and achieve plug flow, which can help with this problem.[325] The baffled oscillatory furnace oscillates at 4.5 Hz within baffles, demonstrating the mixing properties of the solid acid catalyst, propyl sulfonic acid-SBA-15.

5.5. Heterogeneous acid-base catalysts for biodiesel production

Heterogeneous catalysts are essential for optimizing the transesterification process as they lower production costs and minimize the formation of pollutants.[326] These catalysts provide a number of benefits, including simple product recovery, reusability, and the encouragement of economical, ecologically friendly procedures. They are easily extracted from reaction mixtures and undergo treatment processes that improve their lifetime, activity, and selectivity.[327]

In the process of creating heterogeneous catalysts, molecules that are active are positioned on the supporting material's exterior surface or within its pores using techniques, such as grafting and entrapment. In transesterification, solid supports, such as silica-alumina composites,[328] ion exchange resins,[329] mixed metal oxides,[330] transition metal oxides,[331] and metal oxides of alkaline and alkaline earth[306] are often utilized. Alkaline metal compounds supported on alumina or zeolite are often used in various chemical processes, including isomerization, aldol condensation, Knoevenagel condensation, Michael condensation, oxidation, and transesterification.[332] Beryllium oxide, MgO, CaO, strontium oxide, barium oxide, radium oxide, and MgO are among the alkaline earth metal oxides with good catalytic activity. Significantly, strontium oxide is very effective due to its strong basicity and capacity to remain effective for up to 10 cycles without dissolving in methanol.[333] When esterification and transesterification processes requiring high FFA feedstocks are involved, solid acid heterogeneous catalysts are very helpful. Some of the solid base catalysts used in transesterification are CaO, MgO, strontium oxide, potassium nitrate/aluminum oxide, potassium carbonate/aluminum oxide, potassium fluoride/aluminum oxide, lithium/CaO, and potassium fluoride/zinc oxide.[334]

Impregnation is one approach that is frequently used to enhance the performance of mixed metal oxides. Comparing impregnation to monometallic oxides, as was covered in Section 5, the catalyst's stability, surface area, and acid or basic strength are all improved. For instance, at a low temperature of 60°C, doping MgO with 9% lithium results in a FAME production of 93.9% and an oil-to-methanol ratio of 12:1.[335] The amount of water in crude oil and FFAs is also well tolerated by catalysts made of mixed metal oxides during transesterification. Calcium-containing perovskites (calcium titanate, calcium manganate, calcium ferrite, calcium zirconate, and CaO-cerium dioxide) are among the metal oxides that are known for their great stability and capacity to generate high FAME yields.[336]

5.6. Mixed-metal oxide derivatives for biodiesel production

When producing biodiesel, mixed-metal catalysts have shown strong catalytic activity, especially when increasing the effectiveness of active catalytic sites. By using this method, less catalyst is required to retain good catalytic performance and get high conversion rates.[337] When paired with sulfate metal oxides, mixed-metal catalysts show better structural and physical characteristics than their monometallic counterparts. The different sulfated oxides and mixed-metal catalysts utilized in the transesterification reaction, together with information on their feedstocks and references, are described. With normalized time yields continuously over 90%, Table 3 highlights the efficiency of catalysts made of mixed metals doped using sulfated oxides. The CaO-MgO combination stands out among the several oxides of mixed metals due to its great efficacy.[338]

Several studies have shown that adding MgO, CaO, or zinc to catalysts increases the incidence of saponification in addition to catalytic activity.[339] Sulfate ions, which may be added to transition metals, increase the acidity of the catalyst and produce both acidic and superacidic sites.[340] As sulfonation increases the number of sites for Lewis and Brønsted acids, it increases the surface acidity and produces electrophiles.[341] Sulfonation is also the recommended method for controlling metal oxide hydrophobicity, which is a crucial component in maximizing catalyst performance.

The benefits of doping solid acid heterogeneous catalysts with sulfated oxides have been emphasized in a number of investigations. Typical solid super acids include sulfate, zirconium dioxide, sulfate-titanium (IV) oxide, sulfate-tantalum oxide, and sulfate/niobium oxide.[342] An overview of sulfated metal oxides is added to create biodiesel, which also shows how these metal oxides interact with various feedstocks and reaction conditions. According to Table 3, these modified catalysts produce normalized time results ranging from 79% to 98%, showing that they are quite reactive. In some circumstances, catalysts treated with sulfonic acid can accomplish 100% FAME conversion. However, their catalytic activity may be reduced by high methanol concentrations.[343] Widely utilized in metal oxide catalytic processes, mesoporous SBA-15 is prized for its exceptional qualities, including big, consistent pore size, large contact area, corrosion resistance, and recycling.[344]

6. The efficiency of heterogeneous catalysts in biomass-derived biodiesel production

While some studies have examined biodiesel quality, a comprehensive analysis is beyond the scope of this article. The feedstock's fatty acid composition affects various quality characteristics, including density, viscosity, and cetane number. These elements determine the total quality of the fuel and how well the biodiesel separation and purification procedures work. These refinement stages and the FAME profile have an impact on parameters, such as the pour point and flash point. Heterogeneous catalyst efficacy is primarily assessed by biodiesel production and conversion conditions, which are impacted by energy efficiency variables, such as reaction time and temperature. In every case, extra methanol and the catalysts should be collected and re-utilized. In the process, heterogeneous catalysts usually exist as solid phases, in contrast to homogeneous catalysts, which are liquid-based. A major limitation of heterogeneous catalysts is the post-separation procedures, notwithstanding their benefits, which include reduced methanol use. The increased cost of these catalysts is still a drawback, albeit it can be decreased by employing pre-cursors produced from biomass.

Heterogeneous catalysts have an adjustable surface area, porosity, basicity, and acidity, which increase the variety of feedstocks that may be utilized for esterification and transesterification. In addition, these catalysts remove the need for extra pre-treatment to lower the FFA concentration, which makes them very helpful for processing oils with high acidity.

The biodiesel yields that range from 89% to 99% may be obtained using biomass-based CaO catalysts. However, when catalyst loadings were large (25%), Pawar *et al*.[345] found reduced conversion

yields. Reaction periods were 1.4 to 4 h, and reaction temperatures usually fell between 65°C and 70°C, much as in uniform basic catalysis. Although higher ratios of methanol to oil were occasionally employed, methanol recovery and reuse do not present any issues. Given their high conversion efficiency, eggshells are among the most often utilized catalyst pre-cursors. However, few researchers have thoroughly investigated the practical applications of these catalysts, and many studies do not establish a clear connection between the catalyst source and the feasibility of large-scale biodiesel production. Eggshells, for instance, may not be a viable pre-cursor for large-scale biodiesel manufacturing because of supply chain issues. To support a circular economy, a more sustainable strategy would be to use agro-industrial wastes as catalyst pre-cursors, such as *J. curcas* or palm oil leftovers.

Figure 4 shows encouraging outcomes for catalysts based on biomass ash and biochar. In contrast to CaO-based catalysts, biomass ash catalysts drastically shorten reaction durations and methanol-to-oil ratios. However, the yields of biodiesel produced by catalysts based on charcoal are often somewhat lower. Activated carbon-derived catalysts often outperform the other biomass pre-cursors examined, reducing the temperature, catalyst consumption, methanol-to-oil proportions, and the duration of reaction while preserving 92 – 98% biodiesel yields. Given that it is influenced by variables, including catalyst type, alcohol, temperature, catalyst quantities, and reaction duration, biodiesel yield is an essential metric for evaluating catalyst performance. The best results are obtained by homogeneous basic catalysis and ash-based catalysts, with the data showing no discernible difference in FAME production across the different catalysts. The least amount of catalyst is needed by homogeneous basic catalysis, followed by biochar-activated carbon catalysts. To obtain comparable FAME yields, other catalyst types typically need 6.1 – 7.5 wt% catalyst loading. Since they use biomass pre-cursors, biomass-based catalysts provide considerable economic savings and less environmental effect than HBC, but often require more material for comparable performance.

The type of catalyst has minimal effect on the quality of biodiesel produced using heterogeneous catalysts since catalysts primarily affect reaction kinetics. The quality of biodiesel can be impacted by downstream processes, such as separation and purification, which are impacted by catalysts. The biodiesel synthesis efficiency of heterogeneous catalysts generated from biomass is given in Table 8. The quality characteristics of biodiesel produced with the catalysts examined in this research often satisfy the needs of the industry. The type of catalyst, feedstock properties, and operating circumstances are some of the variables that affect these outcomes. A comparison of homogeneous and heterogeneous catalysis for the manufacture of biodiesel reveals that the economic feasibility of heterogeneous catalysis is mostly dependent on the number of cycles and the yield per cycle. Although heterogeneous catalysts sometimes have greater upfront costs, their total biodiesel production and cycle count make them a viable alternative. The subsequent section delves more into the impact of cycling efficiency on catalyst lifespan.

Table 8 provides a comprehensive overview of biodiesel synthesis efficiencies using heterogeneous catalysts derived from various biomass sources. These catalysts, often calcined at high temperatures, facilitate the transesterification of oils into FAME under optimized conditions. For instance, catalysts derived from seed cakes, such as sunflower, castor, jatropha, and palm oil cakes, with catalyst loadings ranging from 5% to 20%, achieved biodiesel yields between 84% and 94% under a 12:1 methanol-to-oil molar ratio at 60°C for 2 h. Similarly, palm stearin-derived catalysts, particularly those from palm oil shells, demonstrated a high yield of 95.3% under a 20:1 molar ratio at 60°C for 4 h. Animal-derived catalysts, including those from mussels, shrimps, snails, and clam shells, exhibited impressive yields, with some reaching up to 99%. For example, shrimp shell-derived catalysts achieved a yield of 89.1% under a 9:1 molar ratio at 65°C for 3 h. Eggshell-derived catalysts consistently produced yields between 95% and 98% across various feedstocks, showcasing their versatility and efficiency. Ash-based catalysts, such as those from coconut husk, banana peels, and *Azadirachta indica*, also demonstrated high catalytic activity. Coconut husk-derived catalysts achieved yields up to 99% under a 12:1 molar ratio at 45°C for 45 min. Banana peel-derived catalysts produced yields of 98%, highlighting the potential of agricultural waste as a sustainable catalyst source. Biochar-supported catalysts, including those from canola, rice husk, and palm fatty acid distillate, exhibited varying efficiencies. For instance, canola-derived catalysts achieved yields between 89% and 92% under an 18:1 molar ratio at 65°C for 3 h, while rice husk-derived catalysts reached a yield of 87.6% under a 20:1 molar ratio at 110°C for 15 h. These variations underscore the importance of optimizing reaction conditions for each catalyst type. The data presented in Table 8 underscores the significant impact of catalyst source, preparation

Table 8. Biodiesel synthesis efficiency of heterogeneous catalysts generated from biomass

Source	Catalyst	Loading (wt%)	Time (h)	Temperature (°C)	Molar-to-oil ratio	Yield (%)	References
Seed cake-based oleic acid	Sunflower seed oil cake	5 – 20	2	60	12:1	84 – 94	346
	Castor seed oil cake	5 – 20	2	60	12:1	84 – 94	347
	Jatropha seed oil cake	5 – 20	2	60	12:1	84 – 94	348
	Palm oil	5 – 20	2	60	12:1	84 – 94	349
Palm stearin	Palm oil shell	10.5	4	60	20:1	95.3	350
Frying oil	Sugarcane bagasse	1	5	66	18:1	94	351
	Palm oil shell	1 – 6	1–4	60 – 100	6:1 – 18:1	82 – 95	352
	Jatropha shell	-	2	60	20:1	87	353
Palm fatty acid distillate	Sugar beet pulp	-	5	85	5:1	92	354
Tallow	Mussel shell	5	1.5	70	12:1	90	157
Rapeseed	Biont shell	3	3	70	ns	97.5	355
	Shrimp shell	2.5	3	65	9:1	89.1	356
Frying oil	Eggshell	1.5	2	65	12:1	96	357
	Snail shell	2	8	65	6:1	87 – 99	154
	Clam shell	3	3	60	6:1	89 – 97	358
Soybean	Eggshell	3	3	65	9:1	95	359
Palm olein	Eggshell	10	2	60	12:1	95	360
Pongamia	Eggshell	2.5	2.5	65	8:1	94 – 97	361
Fish oil	Crab shell	2.5	1.5	65	10:1	96	362
Mustard	*Turatia striatula* shell	3	6	65	9:1	93.3	363
Palm oil	*Pomacea* sp. shell	4	4	60	7:1	95.6	364
	Animal bones	20	4	65	18:1	96.7	176
	Eggshell	1.5	2	65	12:1	98	365
	Green mussel shells	7.5	3	65	2:1	95.1	366
Frying oil	Chicken bones	3	3	80	3:1	96	367
Soybean	Oyster shell	25	5	65	6:1	70	368
Ash-based							
Thevetia peruviana	*Musa balbisiana* trunk	20	3	32	10:1	96	369
Jatropha curcas	*Musa balbisiana* underground stem	5	1	275	9:1	98	370
	Lemna perpusilla Torrey	5	1	300	9:1	89	371
	Acacia tree stem	5	3	65	12:1	98	372
	Coconut husk	7	45	45	12:1	99	373
Mesua ferrea	*Musa balbisiana* underground stem	5	1	275	9:1	95	374
Bauhinia monandra	Banana peels	2.75	1	65	7.6:1	98	375
Azadiracha indica	Cocoa pod husk	0.65	1	65	3:2.2	99	376

(Cont'd...)

Table 8. ***(Continued)***

Source	Catalyst	Loading (wt%)	Time (h)	Temperature (°C)	Molar-to-oil ratio	Yield (%)	References
Cerbera manghas	Coconut husk	10	3	60	6:1	88.6	377
Palm oil	Eggshell	7	4	65	9:1	91.5	378
Canola	Harwood	5	3	65	18:1	89 – 92	379
Wood	Canola	5	3	150	6:1	48	380
Frying oil	Rice husk	5	15	110	20:1	87.6	381
Palm fatty acid distillate	Palm waste	2.5	2	60	9:1	96.1	382
Oleic acid	Corn straw	7	4	60	7:1	98	383
Palm oil	Palm oil trunk	2	0.75	65	-	93	384
	Sugarcane bagasse	2	0.75	65	-	94	385

method, and reaction conditions on biodiesel yield. The use of biomass-derived heterogeneous catalysts not only enhances biodiesel production efficiency but also promotes the utilization of renewable and waste materials, contributing to sustainable energy solutions.

6.1. Heterogeneous solid catalysts derived from biomass

Alkali metals, metal oxides, and alkaline earth metals are examples of heterogeneous catalysts that are frequently employed in the manufacture of biodiesel. These catalysts are frequently accompanied by precursors that offer a large surface area.[386] Since solid acid catalysts provide several active sites with both Lewis and Brønsted acidity, they are preferred over liquid acid catalysts.[387] Table 8 summarizes a variety of feedstocks, catalyst preparation techniques, and solvents utilized in the manufacture of biodiesel, along with examples of mixed-metal solid acid catalysts. The performance of these catalysts is highlighted in Table 8, with special attention to the normalized time and biodiesel yields, as reported in different investigations.

Despite being extensively researched for biodiesel production, solid alkaline catalysts face several limitations, including slower reaction rates and the possibility of side reactions, which have prevented their wider use.[388,389] Over the past 10 years, substantial progress has been achieved in developing solid alkaline catalysts despite these obstacles. Among them, CaO, which can be obtained from both conventional and biomass sources, has gained popularity due to its extended operational life and strong catalytic activity in mild reaction settings. In contrast to other heterogeneous catalysts, CaO has been shown to exhibit a reduced rate of reactivity.[390]

For the manufacture of biodiesel, CaO has been employed as an alkaline solid catalyst in several experiments.[391] Research has shown that CaO is a viable choice for recyclable catalysts since it is extremely soluble in methanol.[392] In addition, other research has looked at biodiesel production using various feedstocks, including soybeans, and catalysts with mixed oxide heterogeneity, including calcium-doped silicates. Mesoporous calcium silicate[393] and pectin methyl esterase-templated calcium structures (PMCS1-9) are notable CaO catalysts that incorporate silicates as references.

6.2. Biodiesel production using zeolite-based catalysts

Zeolite catalysts are made up of crystalline aluminosilicate compounds with a microporous, three-dimensional structure composed of tetrahedra connected by oxygen atoms.[394] In accordance with criteria, such as molecular size, ion exchange characteristics, and internal acidity, these catalysts are selective, permitting only certain hydrocarbon molecules to pass through their pores while obstructing others.[64,395] Since the 1970s, zeolites have garnered substantial commercial attention, expanding on discoveries made in the 1950s. Their varied uses, including catalytic cracking, alkylation, and isomerization, have had a major impact on the petrochemical industry.[396]

Zeolites work well in esterification reactions with carboxylic acids. However, only large-pore zeolites work well in such reactions, and their reaction rates

are often sluggish. Numerous variables, such as site strength, surface hydrophobicity, pore size, channel system dimensionality, and aluminum concentration, all affect their catalytic activity.[397]

Some of the benefits of zeolites include high acidity, high surface area, superior ion exchange capacity, and both thermal and hydrothermal stability.[398,399] In addition, they make shape-selective reactions possible, in which the catalyst can only interact with specific reactants, products, and transition states. With customized crystal structures, different silicon/aluminum ratios, pore diameters, and proton exchange levels, zeolites may be produced for certain acid-base catalyzed processes. These selective processes are facilitated by zeolites' homogeneous crystalline channels.[400] The benefits have been investigated and validated in several studies.[401]

Making active areas more accessible in microporous zeolite structures has been the subject of recent studies. The "top-down" and "bottom-up" techniques have become the two primary methods for creating hierarchical structures in zeolites.[402] Dealumination is a crucial technique in the top-down approach, which entails synthetically altering microporous zeolites to produce a hierarchical structure.[403] The method creates a more stable type of zeolite that is perfect for catalytic cracking and optimizes the silicon/aluminum ratio.[404,405] In contrast, by employing template elements to manufacture both microporous and mesoporous domains, the bottom-up approach produces hierarchical zeolites. Since they are readily available and versatile, substances containing carbon, including carbon aerogels, carbon nanotubes, and carbon black, are frequently utilized as templates.[406,407]

During synthesis, different mesoporous molecules that are well-ordered and linked may be created utilizing the bottom-up method with carbon templates. However, the use of materials, such as graphene and graphene oxide as templates presents difficulties.[408]

A summary of biodiesel production using heterogeneous catalysts made from zeolite is presented here, with details on the catalyst weight and FAME production in regard to the reaction temperature, reaction conditions, feedstock sources, and normalized time yields. Zeolite-based catalysts have been reported to provide FAME outputs ranging from 79.30% to 98%, as Table 1 illustrates.

6.3. Heterogeneous biodiesel production through microbubble-mediated catalysis

A new approach in catalyst technology called microbubble-mediated catalysis expands on well-known processes, such as pressure swing reactive distillation and reactive distillation, both of which have been widely applied in earlier studies. Compared to conventional esterification techniques, which frequently have problems with kinetics and mass transfer, this method enhances reaction speeds and conversion efficiency.[409] Microbubble catalysis has been created to overcome these restrictions and improve the overall effectiveness of esterification.

By reducing buoyancy, the microbubble catalysis approach lengthens the residence period in the liquid phase.[410] Its use as a heterogeneous catalyst in reactive distillation processes has been the subject of several investigations.[411-413] The review does not delve deeply into this approach due to it being relatively new. However, when more research becomes available, it is anticipated that subsequent reviews will provide a more thorough analysis.

7. A comparative analysis of biodiesel production costs versus petroleum diesel

Table 9 provides a comprehensive overview of biodiesel, which offers a renewable alternative to petroleum diesel. However, its economic viability varies significantly depending on feedstock and production methods (Table 8). According to recent data (2023 – 2024), waste cooking oil biodiesel (2.50 – 3.50 US Dollar [USD]/gal) and animal fat biodiesel (2.70 – 3.60 USD/gal) are the most cost-competitive, nearing parity with petroleum diesel (3.00 – 4.20 USD/gal). In contrast, soybean (3.20 – 4.80 USD/gal) and rapeseed biodiesel (3.50 – 5.00 USD/gal) remain more expensive due to high feedstock costs, while algal biodiesel (8.00 – 15.00 USD/gal) is still prohibitively expensive due to high cultivation and extraction expenses. Palm biodiesel (2.80 – 3.80 USD/gal) is economically viable but faces sustainability concerns. Key cost drivers include feedstock availability (70 – 80% of production costs), processing efficiency, and policy incentives, such as the USs Renewable Fuel Standard and the EU's RED III subsidies, which can reduce biodiesel costs by 0.50 – 1.50 USD/gal. Despite higher production costs for some biodiesel types, their potential for lower greenhouse gas emissions and energy security benefits makes them attractive alternatives, especially as petroleum prices remain volatile due to geopolitical factors. Future cost reductions in algal biodiesel and expanded use of waste feedstocks could further improve biodiesel's competitiveness against conventional diesel.

Table 9. Comparison of biodiesel types versus petroleum diesel

Biodiesel type	Feedstock	Production cost (USD/gallon)	Petroleum diesel cost (USD/gallon)	Key cost drivers
Soybean biodiesel	Soybean oil	3.20 – 4.80	3.00 – 4.20 (2024 average)	Feedstock (80% of cost), processing
Rapeseed biodiesel	Rapeseed oil	3.50 – 5.00	Same as above	Higher oil yield than soybean
Palm biodiesel	Palm oil	2.80 – 3.80	Same as above	Low feedstock cost; sustainability issues
Waste cooking oil biodiesel	Used cooking oil	2.50 – 3.50	Same as above	Cheap feedstock; purification challenges
Algal biodiesel	Microalgae	8.00 – 15.00	Same as above	High cultivation/extraction costs
Animal fat biodiesel	Tallow, lard	2.70 – 3.60	Same as above	Limited supply; low feedstock cost
Petroleum diesel	Crude oil	3.00 – 4.20	Same as above	Geopolitical factors, refining costs

Abbreviation: USD: United States dollar.

8. Biomass-derived heterogeneous catalysts: a strengths, weaknesses, opportunities, and threats review

Solid catalysts face several issues, such as microporosity, leaching, toxicity, lack of active sites, and high prices.[414] These problems are caused by the traditional homogeneous catalysts' dependence on non-renewable, hazardous materials. To mitigate some of these issues, the three-phase heterogeneous system reduces reaction inhibition.[415] However, slower reaction rates can result from the immiscibility of solid catalyst phases, affecting mass transfer efficiency.[416] In the synthesis of biodiesel, low conversion yields are also caused by mass transfer restrictions for bigger molecules. Increased larger pore sizes, increased surface areas, enhanced hydrophobicity, and greater external catalytic regions are all necessary for solid acid catalysts to operate better.[417]

Facilitating the use of biomass feedstocks, especially those derived from waste, is crucial for enhancing catalyst sustainability and mitigating their ecological effects. To investigate waste-derived materials that might support sustainability in the manufacture of biodiesel, more research funding is essential. Regulations and tax breaks that promote the use of recycled materials are examples of policies that can propel the growth of a circular economy in which customers and manufacturers are both actively involved.

It is imperative to reduce reliance on fossil fuels due to the expanding global need for energy, especially in the transportation industry, which uses 40.5% of all energy.[418] Creating sustainable alternative energy sources, such as biodiesel made from the provision of biomass and bio-waste, is crucial in meeting this expanding need.

The worldwide production of biodiesel has increased, but the efficient utilization of available feedstock is still constrained by the dominance of homogeneous catalysts. Since these catalysts have a number of shortcomings, heterogeneous catalysts have emerged as a viable substitute. Advantages of heterogeneous catalysts include faster manufacturing, more catalyst reuse, fewer by-products (like soap) generation, and an environmentally friendly method. Nevertheless, they still fall short of the necessary FAME content requirements (>96.5%).[419] Future developments in catalyst modification, process optimization, and manufacturing platform innovations are required to improve reaction performance.[420]

Although heterogeneous catalysts have a lot of potential for producing biodiesel, little is known about their kinematics and reaction pathways, especially when transesterification and esterification are involved.[421] The existing corpus of studies is inadequate to make firm judgments on the mechanisms and kinetics of these catalysts.[422] Hence, further thorough research is required to counterbalance the present emphasis on the advantages and disadvantages of these catalysts.

The difficulties with homogeneous catalysts have been thoroughly studied, whereas heterogeneous catalysts remain relatively new. Important problems that need to be addressed include instability, poor reaction rates, and limited catalyst life spans. Solid base catalysts' heightened sensitivity to carbon dioxide, water, and FFAs results in catalyst deactivation through saponification.

Other difficulties include lipase inhibition during enzymatic biodiesel production in the presence of methanol, catalyst leaching, and product contamination from water hydrolysis of ionic groups. Higher reaction

temperatures are frequently required for nanocatalysts to function, which raises the energy and cost requirements. More economical and energy-efficient techniques are required to improve the recovery and repurposing of nanocatalysts, which can address present challenges in biodiesel production.

9. Prospects of biomass-derived catalysts

Novel approaches, such as solid-state fermentation with solid whole-cell biological catalysts, have raised significant interest. This technique permits the direct use of crude fermented solids as biocatalysts by supporting minimum microorganism growth on substrates, such as agro-industrial waste. Complex lipase purification and immobilization procedures are no longer required with this method.[423]

Hydro-esterification, which focuses on feedstocks with high levels of FFAs and water, is another exciting advancement. This process consists of two steps: first, glycerol and FFAs are produced by hydrolyzing mono- and triacylglycerols, and second, the FFAs are separated and esterified to create biodiesel.[424] Researchers have put forward methods, including solvent usage, gradual methanol introduction, or continual glycerol elimination using solvent extraction or dialysis, to overcome methanol inhibition in enzymatic processes. In addition, a viable solution for this problem is recombinant DNA technology.[425] Furthermore, to decrease catalyst inhibition and shorten reaction durations, continuous systems have been designed that utilize near-critical carbon dioxide as the reaction medium.[426]

The transformation of glycerol, an esterification by-product, into syngas represents an additional area of potential. Several techniques have been investigated for this purpose, such as supercritical water reforming, aqueous-phase transforming, and auto-thermal reforming.[427-430] Despite their continued high cost, the usage of lipase methyl and ethyl acetate is growing as alcohol replacements in the manufacture of biodiesel. By preventing the synthesis of glycerol, this method makes downstream separation and recovery easier and produces triacetin, a higher-value product, which lowers manufacturing costs.

10. Recovered and utilized catalysts

The ability of heterogeneous catalysts to be recovered and reused is one of their main benefits in the synthesis of biodiesel since it drastically reduces production costs. However, maintaining catalyst stability throughout several reaction cycles is essential, and methods for improving catalyst performance have been put forth. The experiments in Table 8 examine using catalysts in a number of different reaction phases (three to ten cycles), both with and without in-cycle reactivation treatments. During the fourth and fifth phases, some studies indicate that the production of biodiesel only slightly decreases, staying at 90%. However, in some situations, after the third cycle, there are notable drops in catalytic activity, which results in a yield loss of more than 10%. Karmakar *et al*.[431] for instance, found that yield had stabilized at about 80% from the third to the tenth cycle but had decreased by 8% after the second cycle. In addition, Reshad *et al*.[432] observed that yields in the first cycle fell short of 90%. With no change in performance from the first to the fourth cycles, the catalysts did, however, show encouraging potential for reuse.

A number of catalysts have been shown in several experiments to retain their reactivation procedures, which are not required for stability and catalytic performance. To produce biodiesel from *C. inophyllum* oil, Olatundun *et al*.,[433] for example, looked at the repurposing of a catalyst based on cocoa pod husk ash. Centrifugation was employed to separate the catalyst from the biodiesel and glycerol once the transesterification reaction was finished, and it was then reused without any further washing or heat/chemical treatment. Notably, biodiesel yields were above 98% for three consecutive cycles, suggesting that there was no discernible decrease in catalytic activity. In a similar vein, catalysts made from *Moringa oleifera* leaf ash demonstrated a biodiesel production of over 85% in the first cycle, but by the third cycle, the output had dropped by 30%, and the catalyst had darkened. This implies deactivation due to surface pollution, which probably reduces the quantity of active servers. It was proposed that high-temperature regeneration (over 500°C) was necessary after each cycle to restart the catalyst.

The repurposing of catalysts made from walnut shell ash, despite the use of heat during cycles, was examined by Foroutan *et al*.[434] They discovered that there was a significant drop in FAME content (from over 95% to about 30%) in the absence of reactivation. Following 2 h of recalculation at 800°C, the catalyst resumed its activity, and for four cycles, the reaction yield surpassed 90%. On the other hand, the recalcined catalyst underwent structural alterations and a significant decrease in specific surface area. According to them thermal reactivation also reduced the catalyst's surface area, basicity, and acidity, which resulted in

pore structure collapse and closed pores. The authors suggested researching catalyst reuse without heat treatment or looking at alternate activation techniques in light of these findings. A recovered catalyst's catalytic function and stability are intimately related. Catalytic effectiveness may be lowered by the leaching of catalyst components during reuse, which can decrease the number of active sites. A catalyst based on *S. indicum* plant ash showed notable potassium leaching, and during the third cycle, Nath *et al*.[254] also noted a loss of silicon and sodium. A progressive decrease in catalytic activity was facilitated by this leaching.[266] A significant deactivation issue for *Carica papaya* stem ash catalysts, according to Gohain *et al*.,[435] is potassium leaching because K_2O is very soluble in glycerol and methanol. Even upon reactivation, Tsai and Tsai[436] observed a notable reduction in catalytic activity (down to 22% conversion), which they ascribed to potential leaching. Leaching can reduce the catalyst's usable life, reduce its possibility for reuse, and contaminate biodiesel.[19] By recovering the catalyst and evaluating it in a different process, the scientists used a leaching test. With a conversion rate of 60.38%, they discovered that leached homogenous species were responsible for a portion of the conversion.

Rostamian *et al*.,[437] discovered no leaching in catalysts based on wood ash. However, by detecting very little soluble alkalinity in the methanol filtrate and monitoring the catalytic activity of the leached residue, they were able to verify the durability of the catalyst's active sites. This demonstrated the catalyst's continued heterogeneity and stability for repurposing. In addition, the authors discovered that high-temperature calcination enhanced catalyst stability and permitted several cycles of reuse by preventing potassium leaching.

Various reports have shown the performance of four catalytic pre-cursors produced from biomass. Although catalyst reuse works, a number of factors affect its effectiveness. Notably, FAME yield frequently decreases noticeably after the second or third cycle. To find the best time to either convert to a new catalyst or reactivate the present one for optimum efficiency and cost-effectiveness, catalyst reuse must be carefully considered in each situation. According to studies, frequent usage can cause ester, glycerol, and FFA molecules to aggregate on the catalyst surface, obstructing active sites and decreasing catalytic performance. Leaching can also decrease the conversion of biodiesel, which limits the possibility of reusing the catalyst. These elements emphasize how important it is to closely monitor catalyst functioning to ensure effective and long-lasting biodiesel production.

11. A critical examination of catalysts

Heterogeneous catalysts that utilize biomass provide many advantages over traditional chemical catalysts, including non-toxicity, biodegradability, and broad availability. However, the elemental composition (e.g., potassium, calcium, magnesium) and crystalline components (e.g., potassium carbonate, K_2O, CaO, MgO) of these biomass-derived catalysts affect their catalytic performance. CaO and K_2O are widely utilized as solid base catalysts in biodiesel production through transesterification. While these catalysts offer high activity and environmental benefits, they can lead to increased sediment formation during the reaction. CaO, in particular, exhibits strong basicity, which enhances its catalytic efficiency. However, this high basicity can also result in the leaching of calcium species into the reaction medium. These leached calcium ions can react with FFAs present in the feedstock, forming calcium soaps. These soaps precipitate as solid sediments, which can accumulate and cause operational issues, such as clogging of equipment and complications in the purification process. K_2O, when used as a dopant in catalysts, such as CaO-zinc oxide, can increase the surface area and basicity of the catalyst, potentially enhancing its catalytic activity. However, similar to CaO, K_2O can also contribute to sediment formation. The mechanism may differ slightly, but the outcome remains a potential increase in solid byproducts that can affect the biodiesel production process. To mitigate these issues, researchers have explored various strategies, such as supporting CaO on materials, such as alumina, manganese dioxide, and titanium dioxide. These supports can help reduce the leaching of calcium species and minimize soap formation, thereby improving the overall efficiency and sustainability of the biodiesel production process.

The reactivity of transesterification processes is largely determined by these substances and components. In particular, biomass catalysts have a strong basic character because of the high concentrations of alkali and alkaline earth components or their derivatives, which increases their efficiency in promoting transesterification reactions. Transesterification feedstock type affects the catalytic function of biomass-derived catalysts. Because soap generation might result from FFA concentration, basic catalysts work better with feedstocks that have a low FFA level (<3%).

Since acid-catalyzed transesterification may esterify FFAs into their corresponding methyl esters, it is unaffected by high FFA levels and can increase the output of biodiesel. In addition, this analysis highlights how catalyst size, surface area, basicity, and acidity all have a major influence on the output of biodiesel and transesterification. Increased surface area and tiny particles allow catalysts to better reach active sites, which improve the generation of biodiesel. It has not yet been determined that the catalyst surface area and biodiesel production are directly correlated. Since they react more quickly than acid catalysts, basic functionalized catalysts are very useful for the manufacture of biodiesel. Magnetic functionalized heterogeneous basic catalysts are also easy to separate from the reaction media, which increases the catalyst's reusability across a number of cycles.

12. Difficulties and forthcoming tasks

The heterogeneous catalysis transesterification method for producing biodiesel is very simple, but it necessitates a significant financial outlay for implementation. Because they can function with lower reactor capacities, ongoing reactors are typically chosen due to their increased efficiency. When it comes to heterogeneous catalysis, the most popular reactor types are:

(i) Biodiesel, methanol, and glycerol, which are collected in the permeate stream and separated in packed bed reactors
(ii) Reactors with trickle bed technology, in which oil is supplied through a tube and dispersed across the bed of catalysts
(iii) Reactors with membranes in the packed bed that allow goods and unreacted reactants to flow through and be gathered in different containers to give selectivity. Since it has a lower boiling point, methanol is repeatedly evaporated, distilled, and then recycled back into the process to lessen depletion
(iv) Resins that exchange ions
(v) Electro-catalytic reactors
(vi) Standard homogeneous batch reactors that have been improved with a magnetic field to aid in the recovery and separation of catalysts.

Reactor changes or a reactor overhaul would be necessary to integrate these technologies into the present biodiesel facilities, which rely on homogeneous catalysis. Before making any investment decisions, a thorough examination should be carried out, as the availability of various technologies and their suitability for different situations may differ. Although technologies, such as resins that exchange ions and electro-catalytic reactors work well for metal oxide catalysts, they are often not appropriate for biomass-based heterogeneous catalysts. Notwithstanding these obstacles, the obvious advantages of heterogeneous catalysis for sustainability and the environment suggest a bright future. However, additional studies and development into reasonable and effective technology are required to make these processes commercially feasible. Important topics requiring further investigation include:

(i) Improving catalyst yield and boosting the quantity of cycles for reuse
(ii) Creating novel catalysts, especially ones made from industrial waste or biomass
(iii) Increasing the selectivity, renewability, and lowering the deactivation rates of catalysts
(iv) Designing reactors and procedures that are economical
(v) Developing innovative upstream and downstream procedures to increase productivity.

13. Circular bioeconomy

The incorporation of a circular bioeconomy strategy in the conversion of oils to biodiesel by transesterification has been the focus of limited research, such as the study conducted by Tulashie *et al*.[438] A circular bioeconomy prioritizes the production of biodiesel in a sustainable and resource-efficient manner while minimizing waste, lowering dependence on scarce resources, and encouraging a regenerative, closed-loop framework. The following guidelines should be the main focus of future studies in this field:

(i) Employing non-food and renewable feedstocks: future research should focus on employing waste materials or by-products, including non-edible oils or agricultural residues, as feedstock to avoid competing with food production
(ii) Turning waste into valuable resources: gathering and turning used waste streams high in lipids, such as animal fats and cooking oils, creating biodiesel is a crucial step in the manufacture of biodiesel for an effective circular bioeconomy. This method will increase resource efficiency and assist in reducing waste
(iii) Encouraging systems with closed loops: waste products, such as glycerol, which are frequently created, should be treated throughout transesterification for the extraction of important chemicals or used in novel ways, such as making

chemicals, energy, or animal feed

(iv) Reducing waste through process integration: a circular bioeconomy promotes the integration of several processes to maximize resource use and decrease waste production, hence increasing system efficiency.

14. Executing life cycle assessment

Assessing the economic impact of the transesterification procedure, society, and environment requires incorporating life cycle assessment into the circular bio-economy framework. Using life cycle assessment, Malode *et al*.[439] recently examined advancements in the production of biofuels from biomass and their environmental impacts. Future studies should evaluate how well transesterification processes perform environmentally and look for ways to cut energy use, greenhouse gas emissions, and resource usage.

15. Conclusion

Further sustainable and affordable fuel is needed to enhance the manufacturing of biodiesel. Conventional catalysis, which usually uses uniform basic catalysts, generates a lot of wastewater and has purification issues, resulting in less consistent biodiesel quality. In particular, this review focused on heterogeneous catalysts, exploring their types, synthesis processes, and applications in the generation of biodiesel. Clearly, calcination temperature and time have a significant impact on catalyst performance. The intrinsic characteristics of biomass-derived heterogeneous catalysts promote sustainable methods and demonstrate encouraging triglyceride conversion outcomes. Although a number of reasonably priced catalysts produced from biomass have previously been investigated, more research is still required to uncover other organic entities.

Converting biomass waste into catalysts promotes sustainable catalyst manufacturing and offers an efficient waste disposal alternative. The utilization of waste materials and inedible oils in conjunction with inexpensive catalysts derived from renewable resources provides a more economical and ecologically sustainable means of producing biodiesel. A number of these catalysts have demonstrated catalytic activity that is similar to existing ones of commercially sold solid catalysts. However, long reaction times and high temperatures still remain challenges, as they result in higher energy usage. Enhancing catalyst efficiency requires more study, either through catalyst modification or the use of technology for process intensification, such as co-solvents, microwaves, or ultrasonication.

Efficient manufacturing of FAME from natural resources has been a primary area of scientific interest. The most efficient technique for producing biodiesel is transesterification. Heterogeneous catalysts provide several benefits over homogeneous ones, including the ability to operate reactors continuously, produce cleaner glycerol, and eliminate the need for alkaline catalyst replacement or neutralization. Due to these benefits, heterogeneous catalysts are a good substitute for FAME generation. Temperature, reactor type, oil kind, and the molar ratio of alcohol-to-oil influence the reactivity of solid catalysts. To get high biodiesel yields, these characteristics must be optimized. The possibility for recovery and their dependence on external-surface active sites make heterogeneous catalysts an attractive option.

The economic viability of biodiesel production varies significantly across technologies, with conventional alkaline-catalyzed transesterification being the most cost-effective (0.30 – 0.50 USD/L) but sensitive to feedstock quality, while enzymatic (0.80 – 1.50 USD/L) and supercritical (0.60 – 1.00 USD/L) methods offer greater feedstock flexibility at higher costs. Stability remains a critical challenge, as biodiesel's oxidative degradation, accelerated by unsaturated fatty acids, can lead to sediment formation, an increase in acidity, and fuel system deposits during storage. To mitigate these issues, antioxidants (e.g., butylated hydroxytoluene, tocopherols) and selective hydrogenation are employed, though they add 0.02 – 0.30 USD/L to production cost. Heterogeneous catalysts (e.g., CaO, K_2O) provide a middle ground (0.40 – 0.70 USD/L) with improved reusability and reduced soap formation, but their tendency to leach metal ions can still compromise long-term fuel stability. Optimizing production methods to balance cost and stability, such as using stabilized nanocatalysts or blended feedstock, is essential for enhancing biodiesel competitiveness against petroleum diesel while ensuring reliable engine performance.

The results imply that catalyst characteristics, particularly basicity and acidity, are essential for the synthesis of biodiesel. Acidity affects esterification activity, but the basicity of the catalyst directly correlates with transesterification efficiency, according to several studies. Better esterification activity results from higher catalyst acidity. Notwithstanding their promise, biomass-derived materials still face a number of obstacles that call for more studies, including their restricted supply, the high energy costs associated

with catalyst modification, the quick loss of catalytic performance brought on by interactions with carbon dioxide and water, and the leaching of CaO during reactions.

To satisfy industrial demands, the primary objective of this research is to call for an improvement in present methods for producing biodiesel and to develop novel ones. It is hard to make existing processes better and develop more effective alternatives, especially when it comes to reactor design, catalyst reusability, and alternative feedstock usage. By addressing many of the challenges related to homogeneous catalysis, biomass-derived heterogeneous catalysts provide a possible solution. By using waste or biomass as a catalyst source, waste materials may find new applications, and the price of solid catalysts that are sold commercially may be lowered. Catalysts' qualitative involvement under dynamic response settings still needs to be better understood, even if the majority of the work so far has focused on their quantitative effects. More investigation is needed to investigate catalyst behavior under these conditions.

Governmental incentives that encourage the use of recycled materials and waste-derived catalysts, such as tax breaks and laws, might boost the economy and provide employment. Further study of biomass-derived catalysts is also required to improve their catalytic efficacy for the generation of biodiesel and other chemical processes.

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Conflict of interest

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Availability of data

All the data analyzed have been presented in the paper. Data will be available upon request to the corresponding author.

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