



Recent Advances in Catalytic Systems for the Sustainable Synthesis of Ethyl Levulinate from Biomass

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Abstract. The esterification of levulinic acid to ethyl levulinate presents challenges in catalyst efficiency, reusability, and environmentally friendly process design, restricting commercial scalability. This study examines recent studies on diverse catalysts, including Deep Eutectic Solvents (DES), homogeneous and heterogeneous systems, and their effects on yield. DES is positioned as a more sustainable option, with yields as high as 99.8%, quicker reaction times, and a lower environmental effect. While heterogeneous catalysts require harsher conditions and have reusability difficulties, DES provides a greener and more efficient alternative to produce ethyl levulinate. Life cycle assessments (LCA) of DES procedures reveal reductions in energy usage and greenhouse gas emissions of up to 69.72%. Future research should focus on improving DES recovery and scalability for industrial applications. This effort supports the United Nations' Sustainable Development Goals (SDGs), namely SDG 7 (Affordable and Clean Energy), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action).

Keywords: Biofuel Additive, Esterification, Ethyl Levulinate, Green Catalysis, Process Intensification

(Received 2025-05-15, Revised 2025-06-19, Accepted 2025-06-21, Available Online by 2025-07-24)

1. Introduction

The growth in demand for renewable and green chemicals has prompted extensive research on chemicals derived from biomass. Recent advancements mention the green synthesis of chemicals derived from biomass such as reductive amination for nitrogen compounds [1], pyruvate-producing microbe metabolic engineering [2], the valorization of biomass using carbon-based nanocatalysts (CnCs) [3], and the development of biochar briquettes from biomass waste for sustainable energy production [4]. Levulinic acid, a platform chemical obtained from the acid hydrolysis of lignocellulosic biomass, has emerged as a significant precursor for many industrial uses among biomass-derived chemicals [5]. Ethyl levulinate is notable among its several derivatives for its versatility and potential applications in the fragrance [6], flavor [7], and biofuel sectors [8].

Esterification is a chemical reaction in which an acid reacts with an alcohol to produce an ester and

water. This process is essential for converting levulinic acid into ethyl levulinate. It enhances the value of levulinic acid and plays a role in advancing green chemistry. By relying on renewable resources and minimizing waste, this process supports sustainability and environmental goals [9]. The transformation of levulinic acid into ethyl levulinate is valuable because of the desirable features of ethyl levulinate, such as its ability to adhere well to metals, its low toxicity, and its biodegradability, making it a strong candidate for replacing petrochemical-based products [10]. Furthermore, ethyl levulinate has shown promise as a fuel additive in diesel, with potential benefits like improving fuel efficiency and lowering emissions [8,11].

Although ethyl levulinate presents promising applications, the esterification process of levulinic acid encounters multiple challenges that impede its scalability and sustainability in industrial contexts. Conventional homogeneous acid catalysts come with several drawbacks, such as causing equipment corrosion, difficulty in separation, and lack of reusability, all of which go against the principles of green chemistry [12]. Heterogeneous catalysts, although reusable, generally require harsh conditions to function effectively or encounter stability issues after being used multiple times, which can limit their performance in various reactions [13]. Achieving the optimal balance in reaction conditions to improve selectivity, yield, and energy efficiency is a complex task due to the interplay of factors such as temperature, the amount of catalyst, and reaction time [14,15]. These issues emphasize the necessity to design more efficient, green catalytic systems and reaction parameters to satisfy both the requirements of industry and the environment.

This review is distinguished from previous studies by its broader focus on the various types of catalysts employed in the esterification process of levulinic acid. Previous reviews have mainly concentrated on specific catalysts, especially heterogeneous ones [16]. However, recent studies and emerging trends have underscored the potential of alternative catalysts, including Deep Eutectic Solvents (DES) [17,18] and enzymes [19]. Alternative catalysts present significant environmental and efficiency advantages, generating optimism regarding the industry's future. Comparing the effectiveness of these catalysts in industrial-scale applications is essential. This review addresses a critical need within the industrial sector for catalysts that demonstrate high efficiency and reusability while adhering to green chemistry principles, thereby promoting sustainable and scalable production processes. This study seeks to address the existing literature gap by conducting a comprehensive comparison of emerging catalysts and evaluating their feasibility for large-scale industrial application.

2. Methods

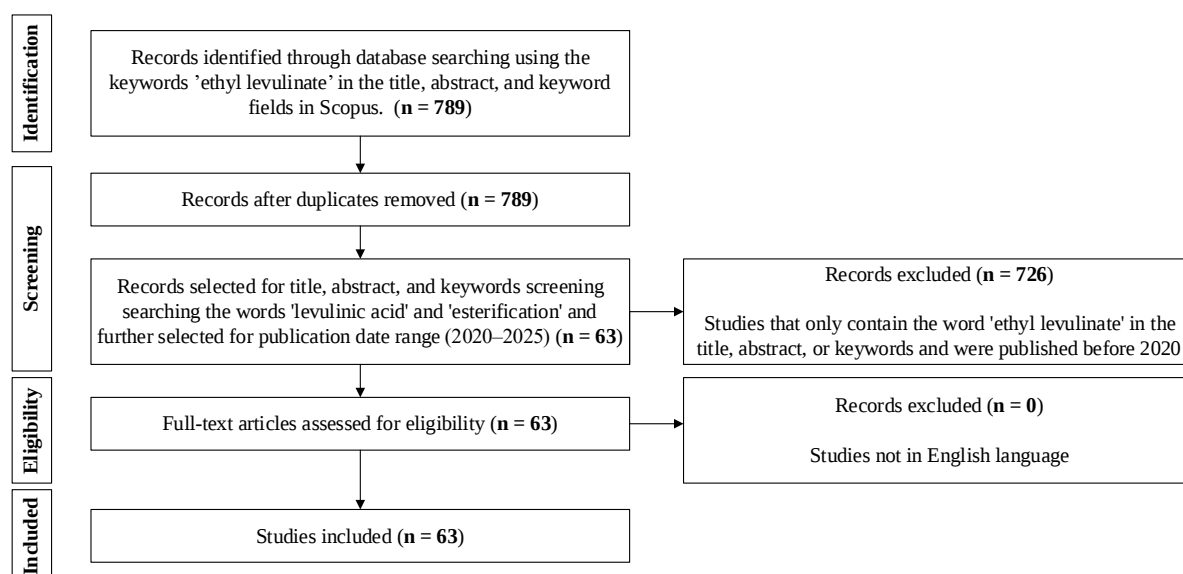


Figure 1. Research Methodology

This research adheres to the PRISMA methodology as outlined by [20], as shown in Figure 1. Initially, 789 records were identified through database searches in Scopus using the keywords 'ethyl levulinate' in the title, abstract, and keyword fields. The total count remained at 789 records after removing duplicates. In the screening phase, records were selected based on their title, abstract, and keyword screening, incorporating additional search terms such as 'levulinic acid' and 'esterification.' The selection process was further refined based on the publication date range of 2020–2025, resulting in the retention of 63 studies. These 63 full-text articles were subsequently assessed for eligibility. Ultimately, no studies were excluded based on language, and the final set of 63 studies was included for analysis.

Subsequently, the papers were analysed to identify significant characteristics, extracting essential aspects, including catalyst type, catalyst load, reaction duration, molar ratio, temperature, and yield from the research. The next phase involved comparing the data., in which the identified parameters were analysed across several research studies. A study of parameter influence was performed to assess the impact of each element on the overall reaction outcomes. In conclusion, the main findings of the research were summarized, along with recommendations for future inquiries and advancements in the field.

3. Results and Discussion

3.1. Recent Studies in the Esterification of Levulinic Acid into Ethyl Levulinate

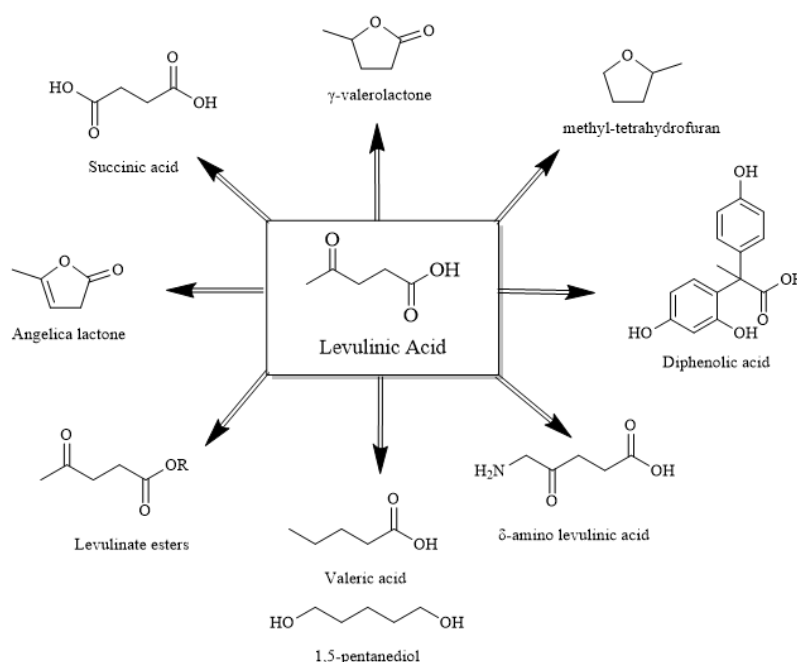


Figure 2. Transformation of Levulinic Acid

Levulinic acid is an economical and easily produced biomass-derived compound that can be converted into various compounds through catalytic reactions [21]. Chemically, levulinic acid has a structure consisting of a carbon chain with one carboxyl group and one carbonyl group, making it a keto acid useful in various chemical applications [22]. Figure 2 illustrates the various valuable compounds that can be produced from levulinic acid. Levulinic acid can be turned into γ -valerolactone, a biofuel additive and green solvent [23], and methyl-tetrahydrofuran, a solvent and fuel additive that improves fuel economy [24]. Diphenolic acid is used to make sustainable and biodegradable polymers [25], while angelica lactone is an important intermediary in pharmaceuticals and fine chemicals [26]. Levulinate esters, such as ethyl levulinate, are commonly utilised in the energy industry as biofuel additives [27]. Other derivatives, such as δ -amino levulinic acid, have potential in medicine and agriculture [28]. Furthermore, levulinic acid can be converted into succinic acid, an important step in chemical synthesis

[29], and hydrogenation of levulinic acid produces chemicals such as 1,5-pentanediol and valeric acid, which are employed in plastics, solvents, and medical applications [30].

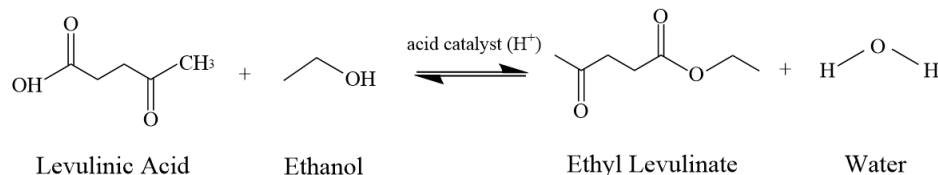


Figure 3. Esterification of Levulinic Acid to Ethyl Levulinate Reaction

One approach to produce ethyl levulinate is to esterify ethanol with levulinic acid, as shown in Figure 3. Ethyl levulinate is a carbon chain compound containing one ester and one carbonyl group and it falls into the class of keto ester compound [31]. It is a very useful compound which has many uses but mainly found in the biofuel industry and in the production of a large number of chemicals. Ethyl levulinate is a suitable biofuel additive that may enhance the burning characteristics of the fuel while not lowering the octane number [32]. Ethyl levulinate is also applicable in fragrance and flavoring industries due to its agreeable smell and solubility [14]. The great scientific and technological groundwork laid over the years on different catalytic methods proposed to produce ethyl levulinate emphasizes its industrial and environmental relevance, when used as an interesting compound not only for biofuel but also for chemical industries [33].

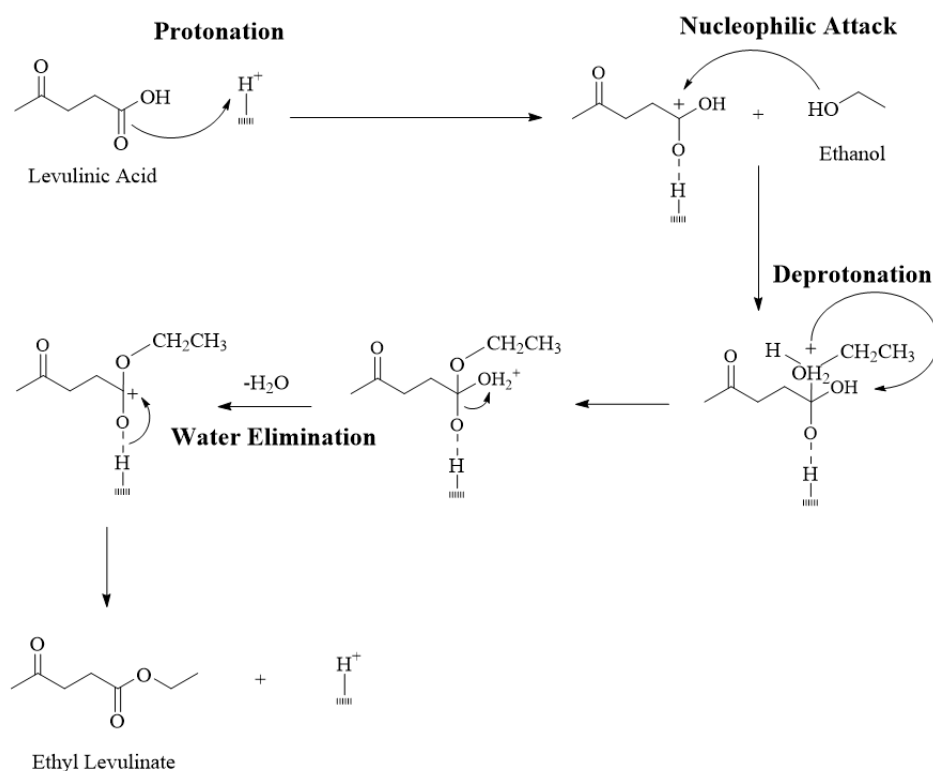


Figure 4. Esterification of Levulinic Acid to Ethyl Levulinate Reaction Mechanism

The esterification process of levulinic acid to ethyl levulinate involves three main steps, as illustrated in Figure 4. First, an acid catalyst such as sulfuric acid protonates the carboxyl group of levulinic acid. This protonation makes the carbon atom in the carboxyl group more electrophilic, thus more susceptible to nucleophilic attack. Next, ethanol attacks the protonated carbon atom in levulinic acid, forming a tetrahedral intermediate. In this step, ethanol acts as a nucleophile attacking the electrophile at the carbon

of the protonated carboxyl group. Finally, the tetrahedral intermediate undergoes deprotonation and elimination of water, resulting in ethyl levulinate as the final product with the release of a water molecule as a byproduct [34–36].

Table 1. Recent studies in the esterification of levulinic acid into ethyl levulinate

Catalyst	Catalyst Load	Reaction Time	Molar Ratio	Temperature	Yield (%)	Reference
Choline chloride and para-toluene sulfonic acid DES	5 wt%	1 h	1:1	80°C	99,8	[37]
MsOH	2.75 g	5.25 h	N.A.	90°C	92,2	[38]
SFCB-4	80 mg	6 h	1:16	60°C	97.6	[39]
AlCl ₃ ·6H ₂ O	5 mol%	2 h	1:01	75°C	98.48	[40]
SAPO-11	0.1 g	0,5 h	1:11	180°C	N.A.	[41]
MoPO supported on TiO ₂ -ZrO ₂	20 wt%	6 h	1:8	70°C	N.A.	[42]
ChCl/sulfanilic acid	2 g	3 h	1:7	80°C	90.6	[43]
UCC-S-Fe	10 wt%	4 h	1:10	80°C	99.5	[44]
ChCl/pTSA	5 wt%	1 h	1:5	80°C	N.A.	[45]
lipase@amino-grafted silica nanoflower	25 mg	8 h	1:10	40°C	99.5	[46]
Thermomyces lanuginosus lipase	5 wt%	12 h	1:4	45°C	90	[47]

Ethyl levulinate production can be achieved through various routes, including both non-catalytic and catalytic methods, as shown in Table 1. Microwave non-catalytic synthesis has been proved to be an efficient green method leading to high conversion efficiencies without the catalyst [48]. Catalytic processes include the use of various catalysts such as AlCl₃·6H₂O catalytic reaction favors the separation of the phases to facilitate the recovery of the product [49], and deep eutectic solvents are environmentally friendly and highly efficient [50]. Furthermore, alternative solid acid catalysts such as kitchen waste-derived materials have been investigated and proposed as green and low-cost alternatives [37]. Especially in the presence of a catalyst, high yield and selectivity to ethyl levulinate can be obtained by the esterification of levulinic with ethanol, which is an attractive method to prepare ethyl levulinate [33].

Table 1 summarises recent studies on the esterification of levulinic acid into ethyl levulinate, detailing the catalysts, operating conditions, and yields obtained. The studies provide significant insights into the influence of different catalysts, reaction conditions, and process parameters on the efficiency and scalability of ethyl levulinate production. An analysis of these factors is essential for understanding their broader implications on reaction outcomes. This section will analyse the primary factors affecting esterification efficiency, including catalyst type and concentration, reaction temperature, molar ratio of reactants, water content, and reaction time, and assess their impact on overall yield and process sustainability.

3.2. Factors Affecting Reaction Outcomes

3.2.1. Catalyst Type and Concentration

Homogeneous catalysts, such as MsOH and DES, achieve increased yields in esterification reactions owing to their exceptional catalytic activity and selectivity. The addition of MsOH with a catalyst load of 2.75 g results in a yield of 92.2% at 90°C after 5.25 hours [38]. In contrast, using DES at a 5 wt% catalyst load and a molar ratio of 1:1 achieves a yield of 99.8% after just 1 hour at 80°C [37]. Increasing

the concentration of these catalysts typically leads to enhanced reaction rates and yields, as it raises the availability of active sites [51]. However, their practicality for large-scale applications is limited by notable challenges in post-reaction separation and reusability [52]. While DES offers greater environmental benefits, it still faces obstacles related to scalability and the effective recovery of catalysts [53].

Heterogeneous catalysts like $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ and SFCB-4 offer easier separation and recycling, which improves their reusability and simplifies operational processes. For example, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ with a catalyst load of 5 mol% can reach a yield of 98.48% at 75°C after 2 hours [40], whereas SFCB-4 with a catalyst load of 80 mg results in a yield of 97.6% at 60°C after 6 hours [39]. These catalysts demonstrate greater cost efficiency over time because they can be employed repeatedly. However, they often require harsher reaction conditions, including higher temperatures, which may diminish their environmental advantages and raise energy costs [54]. Therefore, it is essential to identify the ideal concentration for heterogeneous catalysts based on the operational needs to achieve an effective balance in their performance.

Enzymatic catalysts, including lipases, offer mild reaction conditions, exceptional selectivity, and sustainability, in balance with the principles of green chemistry. The use of lipase catalysts at a concentration of 25 mg results in an impressive yield of 99.5% after 8 hours at 40°C [46]. Enhancing reaction rates can be achieved through an increase of enzyme concentration; nonetheless, this frequently leads to higher costs and challenges associated with enzyme recovery. While these materials are biodegradable and non-toxic, their reduced stability and the difficulties associated with recovering and reusing enzymes on an industrial scale make them less efficient for large-scale esterification processes [55]. As a result, enzymatic catalysts are ideal for reactions that require high purity and small scale; nonetheless, their use in industrial settings is limited due to operational difficulties and concerns regarding cost-effectiveness [56]. Table 2 presents the conclusion on each catalyst type, along with its efficiency and associated challenges.

Table 2. Overview of Catalyst Types, Efficiencies, and Challenges

Catalyst Type	Efficiency	Challenges
Homogeneous Catalyst	High yields are achieved with both DES (99.8%) and MsOH (92.2%) catalysts.	Require a large amount of catalyst, as well as pose challenges in post-reaction separation and reusability.
Heterogeneous Catalyst	High yield is also obtained for both $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (98.48%) and SFCB-4 (97.6%).	Require harsh conditions, such as higher temperatures, which diminish the environmental advantages and increase energy costs.
Enzyme	Enzymes require lower temperatures, but the yield is not as high as the previous catalysts (90%).	Enzymes are costly, and enzyme recovery is complicated since they are living organisms and require a long reaction time.

3.2.2. Temperature

The temperature has impact on the kinetics of reaction, the activation energy, and position of equilibrium. Higher temperatures generally raise reaction rates by furnishing the kinetic energy that reactant molecules need to surmount the barriers to reaction. Nonetheless, the use of high temperature could result in side reactions or decomposition of the labile compounds, decreasing yields and purity of the products. For example, . For example, in the esterification of acrylic acid with n-butanol, the equilibrium conversion increased from 67.55% to 77.81% as the temperature was raised from 50°C to 70°C [57]. Other works have also demonstrated that higher temperature favors higher degree of substitution in starch esterification, thus evidencing the importance of temperature in diverse esterification systems [58].

3.2.3. Molar Ratio of Reactants

The molar ratio of reactants is also an important factor that significantly affects the conversion and yield

of esterification reactions. The ideal molar ratio provides enough reactant for a reaction and a minimum quantity of unreacted starting materials. A study reported the optimal substrate molar ratio of 2:1 for esterification as an excess reactant could prevent the reaction by the increase in viscosity that can inhibit mass transfer and kinetics of a reaction in aggregate [59]. Relatedly, another study reported that the reactant molar ratios directly affect product distribution and conversion efficiencies in different esterification systems [60].

3.2.4. Water Content

The amount of water in the reaction system is a key factor in the esterification reaction, as water is not only a product but also a reactant in the hydrolytic equilibrium. The formation of esters can be reversible at high water concentrations and the esters are consequently converted back to fatty acids, which results in a decreased yield. Water content of deep eutectic solvents (DES) can significantly influence the reaction viscosity and the reactant mobilization revealing a reduction of viscosity that promotes optimal diffusion and reaction kinetics [61]. This observation was in line with another study, highlighted the importance of withdrawing the water products from the reaction while it progresses to the favour the reaction momentum and conversion to higher percentage [62].

3.2.5. Reaction Time

The reaction time is crucial to achieve a required yield in esterification reactions. There should be enough time to permit the reactants to mix properly and establish equilibrium. Increasing reaction time can lead to higher ester content but the benefits tend to diminish as the system reaches the equilibrium as a result of the water product generated blocking the further reaction progress [60]. Also, it has been reported that at certain conditions, reaction times can be optimized resulting in improved enzyme activity in the enzymatic esterification, which could have a significant effect on the respective conversion rates [63].

3.3. Future Perspective for Esterification of Levulinic Acid to Ethyl Levulinate

The esterification process of levulinic acid to ethyl levulinate remains one of the promising green routes for the synthesis of bio-based chemicals as a result of the increasing demand for bio-based feed stock in the chemical industry. Levulinic acid is largely prepared from biomass, with industrial production by major manufacturers such as Biofine Technology (U.S.), GF Biochemicals (Italy), and Chinese manufacturers; however, current production is not yet at industrial scale. The worldwide levulinic acid market size was USD 27.2 million in 2019 and is anticipated to progress at an 8.8% CAGR expected to reach USD 60.2 million by 2030, COVID-19 impact notwithstanding [64]. Although ethyl levulinate is a new entry into the market (a market size of 32.4 ton in 2014), the use of the compound is projected to increase to 49.1 ton by 2022 and US\$14 million in 2026, due to the development of the production technology and the increase of applications in fragrances, food and biofuel. Asia Pacific, contributing to 30% of global levulinic acid market share in 2014, is expected to dominate as a potential market due to China and India industrial growth. With increasing production and cost reduction driven by process optimization and economy of scale, wider acceptance of ethyl levulinate across sectors is likely, potentially cementing its position in the renewable chemicals arena [65].

Many industries promote for heterogeneous catalysts because of their reusability and cost-effectiveness [54]. However, Deep Eutectic Solvents (DES) are gaining recognition as a viable option for industrial applications. DES catalysts demonstrate considerable potential, attaining high yields of up to 99.8% within reduced reaction times, such as 1 hour at 80°C. Their elevated activity, combined with a reduced environmental impact, renders them a compelling alternative for large-scale esterification processes [37]. Advancements in process intensification, including continuous flow reactors and membrane systems, enhance heat and mass transfer efficiency, thereby reducing energy costs. The utilization of agricultural residues in levulinic acid production increases both sustainability and economic value. Sustainability metrics, such as life cycle impact and E-factor, are increasingly used to evaluate the environmental performance of processes [66], and DES catalysts show promise for reducing both energy consumption and greenhouse gas emissions. Life cycle assessment (LCA) studies have shown that DES-assisted processes, when optimized for recovery, can reduce fossil fuel consumption

by up to 69.72%, although DES synthesis and recovery processes remain key areas for improvement to lower energy input and global warming potential [67]. Optimizing reaction conditions and catalyst recovery enables the scaling of DES for industrial applications, in accordance with green chemistry principles and enhancing the bio-economy.

4. Conclusion

This review emphasizes the potential of Deep Eutectic Solvents (DES) in the esterification of levulinic acid to ethyl levulinate. Heterogeneous catalysts are widely utilized in industries due to their reusability and cost-effectiveness. However, deep eutectic solvents (DES) offer notable benefits, such as increased yields and diminished environmental impact, positioning them as an attractive choice for industrial applications. Process intensification techniques, including continuous flow reactors, along with sustainable feedstocks such as agricultural residues, significantly improve the viability of DES. Future research should concentrate on enhancing DES recovery techniques and refining reaction conditions for large-scale application. The industrial sector needs to invest in the expansion of DES applications and enhance cost-effectiveness to promote widespread adoption and achieve sustainability objectives. To facilitate the ongoing advancement of DES in industrial applications, the following innovations are suggested:

- Innovative techniques for the recovery of deep eutectic solvents (DES) are being developed to reduce energy consumption and enhance reusability, including the integration of membrane filtration with recrystallization.
- Designing continuous flow reactors aims to optimize heat and mass transfer, enhance energy efficiency, and minimize environmental impact.
- Increasing the utilization of agricultural residues as feedstocks for levulinic acid production to improve sustainability and economic value.

Acknowledgements

The research was financially supported by The Faculty of Engineering, Diponegoro University, Indonesia through Strategic Research Grant 2025.

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