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Synthesis and Application of Deep Eutectic Solvents for the Deacidification of Waste Cooking Oil: A Pathway to High-Quality Biodiesel

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ABSTRACT

This study investigates the synthesis and application of deep eutectic solvents (DESs) for deacidifying waste cooking oil (WCO) to produce high-quality biodiesel. The research addresses conventional biodiesel production's economic and environmental challenges by using WCO as a cost-effective feedstock and employing DESs as a greener alternative to traditional solvents. DESs are biodegradable, have low toxicity, and can be easily prepared by mixing a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA). The study synthesized DES using Choline Chloride (ChCl) as the HBA and ethylene glycol (EG) as the HBD at a constant temperature of 60° C and variable stirring rates of 150 and 200 rpm. Five different molar ratios of ChCl to EG were tested: 1:1, 1:2, 1:4, 1:6, and 1:8. The effectiveness of the DESs was evaluated based on their ability to remove free fatty acids (FFAs) from WCO, with DES 2 (1:2 molar ratio) and DES 3 (1:4 molar ratio) showing the highest adsorption efficiencies. The research also characterized the resulting biodiesel, comparing key properties such as cetane number, sulfur content, and flash point to established standards. The results show that increasing the DES concentration significantly enhances the removal of FFAs, with DES 2 achieving an 84% efficiency and DES 3 achieving an 83% efficiency at a 1:6 WCO: DES molar ratio. DES treatment consistently improved key fuel properties: the cetane number increased, while sulfur content and total acid number (TAN) were significantly reduced. DES 3 emerged as the most effective solvent, yielding the highest cetane number (56.3), lowest sulfur content (18 ppm), and lowest TAN (0.12 mgKOH/g) at the 1:6 molar ratio. While DES treatment reduced the fuel's flash point, the overall benefits of producing a cleaner, higher-quality fuel outweigh this concern. This research confirms that DESs provide a sustainable and practical pathway for producing high-quality biodiesel from waste cooking oil

Keywords: Biodegradable, Biodiesel, Deacidification, Transesterification, Waste Cooking Oil

ABSTRAK

Studi ini menyelidiki sintesis dan aplikasi pelarut eutektik dalam (DES) untuk mendeasidifikasi minyak goreng bekas (WCO) guna menghasilkan biodiesel berkualitas tinggi. Penelitian ini membahas tantangan ekonomi dan lingkungan dari produksi biodiesel konvensional dengan menggunakan WCO sebagai bahan baku yang hemat biaya dan menggunakan DES sebagai alternatif yang lebih ramah lingkungan dibandingkan pelarut tradisional. DES bersifat biodegradable, memiliki toksisitas rendah, dan dapat dengan mudah disiapkan dengan mencampurkan donor ikatan hidrogen (HBD) dan akseptor ikatan hidrogen (HBA). Studi ini mensintesis DES menggunakan Kolin Klorida (ChCl) sebagai HBA dan etilen glikol (EG) sebagai HBD pada suhu konstan 60°C dan kecepatan pengadukan variabel 150 dan 200 rpm. Lima rasio molar ChCl terhadap EG yang berbeda diuji: 1:1, 1:2, 1:4, 1:6, dan 1:8. Efektivitas DES dievaluasi berdasarkan kemampuannya untuk menghilangkan asam lemak bebas (FFA) dari WCO, dengan DES 2 (rasio molar 1:2) dan DES 3 (rasio molar 1:4) menunjukkan efisiensi adsorpsi tertinggi. Penelitian ini juga mengkarakterisasi biodiesel yang dihasilkan, membandingkan sifat-sifat utama seperti angka setana, kandungan sulfur, dan titik nyala dengan standar yang telah ditetapkan. Hasil menunjukkan bahwa peningkatan konsentrasi DES secara signifikan meningkatkan penghilangan FFA, dengan DES 2 mencapai efisiensi 84% dan DES 3 mencapai efisiensi 83% pada rasio molar WCO:DES 1:6. Perlakuan DES secara konsisten meningkatkan sifat-sifat bahan bakar utama: angka setana meningkat, sementara kandungan sulfur dan angka asam total (TAN) berkurang secara signifikan. DES 3 muncul sebagai pelarut yang paling efektif, menghasilkan angka setana tertinggi (56,3), kandungan sulfur terendah (18 ppm), dan TAN terendah (0,12 mgKOH/g) pada rasio molar 1:6. Meskipun perlakuan DES menurunkan titik nyala bahan bakar, manfaat keseluruhan dari produksi bahan bakar yang lebih bersih dan berkualitas lebih tinggi lebih besar daripada kekhawatiran ini. Penelitian ini menegaskan bahwa DES menyediakan jalur yang berkelanjutan dan praktis untuk menghasilkan biodiesel berkualitas tinggi dari minyak goreng bekas.

Kata kunci: Biodegradabel, Biodiesel, Deasidifikasi, Transesterifikasi, Minyak Goreng Bekas

I. INTRODUCTION

1. Background

The growing global energy crisis and environmental concerns over fossil fuels have spurred a strong interest in alternative, renewable energy sources. Among these, biodiesel, a clean-burning and biodegradable fuel, is considered a viable substitute for conventional diesel, primarily because it helps to reduce greenhouse gas emissions and reliance on fossil resources. (Sharaf-Addin and Al-Dhubaibi 2025). However, a significant challenge for the large-scale commercialisation of biodiesel is the high cost associated with using virgin vegetable oils as feedstock. This economic barrier has driven extensive research into more sustainable and cost-effective feedstocks, particularly waste cooking oil (WCO), which offers the dual benefit of reducing production costs and providing a solution for waste management (Buasri et al. 2023).The increasing demand for sustainable and environmentally friendly energy sources has led to significant interest in producing biodiesel from waste feedstocks, such as waste cooking oil (WCO. One of the primary challenges in utilising WCO for biodiesel production is the high content of free fatty acids (FFAs), which necessitates an effective deacidification process to ensure high-quality biodiesel. (Andermann et al. 2021). The use of WCO as a feedstock presents its own technical challenges, notably the presence of high concentrations of free fatty acids (FFA) and water, which can inhibit the efficiency of conventional transesterification reactions. To overcome these limitations, recent studies have explored several innovative approaches. One promising strategy involves the development of novel catalysts and reactor systems. Deep eutectic solvents offer the advantage of reusability and easy separation from the product, making the process more economical and environmentally friendly. (Würfel and Heinze 2025). Developing simple, cost-effective reactors, like the rotating tube reactor, is effective for continuous biodiesel production, providing a scalable solution for small communities. (Blaschke, Hasso, and Hacker 2024). Due to their unique properties and environmental benefits, deep eutectic solvents (DES) have emerged as a promising alternative for deacidifying waste cooking oil (WCO) in biodiesel production. Here are the key properties and their implications. Deep eutectic solvents (DESs) have emerged as a promising solution for deacidifying WCO due to their unique properties, including low toxicity, biodegradability, and ease of preparation. DESs are formed by mixing a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA), resulting in a solvent with a lower melting point than its individual components. These solvents have been successfully applied in various extraction and purification processes, demonstrating their potential as green alternatives to conventional solvents (Zheng et al. 2024).

2. Problem Formulation

How can the research entitled Synthesis and Application of Deep Eutectic Solvents for Deacidification of Used Cooking Oil: A Path to High-Quality Biodiesel be carried out properly on time and according to procedures?.

3. Research objectives

This research aims to obtain research results related to the "Synthesis and Application of Deep Eutectic Solvents for Deacidification of Used Cooking Oil: A Path to High-Quality Biodiesel" title.

4. Research Benefits

The benefit of this research is to apply the research results obtained from the title "Synthesis and Application of Deep Eutectic Solvents for Deacidification of Used Cooking Oil: A Path to High-Quality Biodiesel" to the public and to provide literature for future research...

II. RESEARCH METHOD

Materials

The waste cooking oil was sourced from local households and blended to ensure a uniform composition. The chemicals used in this study, including Choline Chloride (ChCl) (98%) and Ethylene Glycol (99%), were procured from Geojaya, Bogor, Indonesia, and Bratachem, Palembang, Indonesia, respectively. The Energy Engineering Laboratory at the Politeknik Negeri Sriwijaya provided all other necessary chemicals.

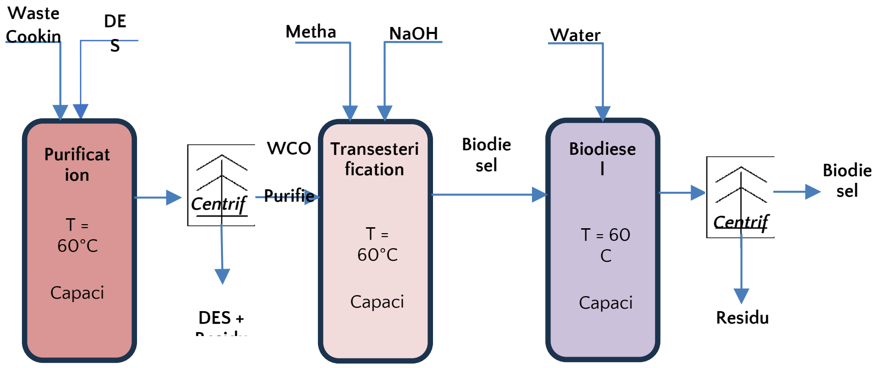

Fig 1. Flow diagram of the refining process for biodiesel production

Waste Cooking Oil Purification

In this study, a deep eutectic solvent (DES) was synthesised using Choline Chloride (ChCl) as the hydrogen bond acceptor (HBA) and ethylene glycol as the hydrogen bond donor (HBD). The synthesis was conducted at a constant reaction temperature of 60°C for one hour, with variable stirring rates of 150 and 200 rpm. The synthesised DES was prepared at different molar ratios of ChCl to ethylene glycol to investigate its effectiveness. The specific molar ratios tested were: DES 1 (1:1), DES 2 (1:2), DES 3 (1:4), DES 4 (1:6), and DES 5 (1:8).

$$\text{FFA removal \%} = \frac{FFA_i - FFA_a}{FFA_i} \times 100 \quad (1)$$ (Buasri et al. 2023)

FFA_i = FFA initial concentration

FFA_a = FFA after adsorption by DES

Transesterification

Transesterification tests were performed in a 100 mL glass reactor with a condenser and magnetic stirrer to generate biodiesel from WCO using NaOH and methanol. The glass reactor was immersed in a temperature-controlled oil bath to explore the influence of reaction temperature on biodiesel yield, and biodiesel generation was conducted under the designated coded conditions. To perform the biodiesel production process under the desired coded circumstances (e.g., Run 1), first, 20 g of the selected oil source was put in the glass reactor and heated for 20 minutes at 60 ◦C to reach equilibrium temperature. Then, 0.5 g of NaOH and a specified amount of methanol were added to the glass reactor, and the mixture was stirred using the magnetic stirrer at 60 ◦C for 180 minutes. Upon completion of the reaction, the methanol/biodiesel/glycerol mixture was placed in a separating funnel for 24 hours to allow glycerol (bottom layer) to separate from the methanol/biodiesel (top layer). The methanol/biodiesel mixture was transferred to a glass beaker with a magnetic stirrer and heated at 80 ◦C for 3 hours to recover excess biodiesel. Biodiesel yield and WCO were calculated:

$$\text{Yield } (\%) = \frac{\text{Weight of the Biodiesel}}{\text{Weight of the Raw Oil}} \times 100 \quad (2)$$ (Wahyuni et al. 2025)

Biodiesel Characterization

Using ASTM and EN standards, kinematic viscosity, density, flash point, cloud point, pour point, acid value, and moisture content were determined and quantified for biodiesel. All values were compared to those specified in the United States biodiesel standard (ASTM D 6751) and the European biodiesel standard (EN 14214)

III. RESULT AND DISCUSSION

Adsorption of FFA in Waste Cooking Oil

The data from this study demonstrate the significant improvement in FFA adsorption efficiency and adsorption capacity achieved by deep eutectic solvents, among the samples tested in this study.Table 4. FFA Adsorption in Waste Cooking Oil provides a comprehensive dataset of the experiments. It shows that increasing the concentration of DES in the mixture (as indicated by the decreasing molar ratio of waste cooking oil to DES) generally leads to a significant increase in adsorption efficiency. The titrant volume, a measure of the remaining FFAs, consistently decreases as the adsorption efficiency rises, confirming the successful removal of FFAs from the waste cooking oil. For example, in DES 2, the titrant volume drops from 15.5 ml (0% efficiency) to 2.5 ml (84% efficiency) when the DES concentration is increased from a 1:0 to a 1:6 molar ratio. This trend indicates that a higher concentration of the DES facilitates more effective FFA removal, likely due to a greater number of active sites for adsorption or a more favorable mass transfer process.

Table 4. FFA Adsorption in Waste Cooking Oil

Composition (Molar Ratio)		Titrant Vol.	FFA_0	FFA_1	Adsorption Efficiency (%)
DES	Product	(ml)			
(ChCl + EG)	(WCO: DES)				
DES 2	1 : 0	15.5	7.94	7.94	0%
	1: 1	8.7	7.94	4.45	44%
	1 : 3	5	7.94	2.56	68%
	1: 4	3	7.94	1.54	81%
	1: 6	2.5	7.94	1.28	84%
DES 3	1 : 0	15.5	7.94	7.94	0%
	1: 1	12.1	8.7	6.2	29%
	1 : 3	6	8.7	3.07	65%
	1: 4	3.3	8.7	1.69	81%
	1: 6	2.9	8.7	1.48	83%
DES 4	1 : 0	15.5	7.94	7.94	0%
	1: 1	12.9	7.94	6.6	17%
	1 : 3	10.5	7.94	5.38	32%

	1: 4	7.4	7.94	3.79	52%
	1: 6	5.9	7.94	3.02	62%
DES 5	1 : 0	15.5	7.94	7.94	0%
	1: 1	13.9	7.94	7.12	10%
	1 : 3	11.5	7.94	5.89	26%
	1: 4	8.3	7.94	4.25	46%
	1: 6	7.4	7.94	3.79	52%

The positive correlation between DES concentration and adsorption efficiency is consistent across all DES types. This is most pronounced in DES 2 and DES 3, which exhibit the highest overall efficiencies. For instance, DES 2's efficiency skyrockets from 44% at 1:1 to 84% at 1:6. DES 3's efficiency jumps from 29% to 83% over the same range. This trend is expected, as a higher proportion of the extracting agent (DES) provides more capacity to interact with and remove the target molecules (FFAs). The data suggest that, for practical application, using a higher concentration of DES is more beneficial, albeit with potential economic considerations (Martins et al. 2024). While all DES types show improved performance with increasing concentration, there is a significant difference in their maximum achievable efficiencies. DES 2 and 3 consistently outperform DES 4 and 5, especially at higher concentrations. DES 2 achieves the highest efficiency of 84% at the 1:6 ratio, closely followed by DES 3 at 83%. In contrast, DES 4 and DES 5 peak at 62% and 52%, respectively, at the same ratio. This variation in performance suggests that the specific chemical composition of DES 2 and DES 3 is more effective at interacting with and adsorbing FFAs from waste cooking oil. As indicated in the table, the difference in their ChCl + EG (Choline chloride + ethylene glycol) composition is likely the key factor behind this varied performance. These specific DES compositions' structural and chemical properties might create a more favorable environment for FFA extraction through mechanisms like hydrogen bonding or other intermolecular forces. The results highlight the importance of carefully selecting the DES composition to optimize adsorption.The visual representation in the bar graph makes these performance differences immediately apparent, with the longer bars for DES 2 and DES 3 visually confirming their superior performance. This visualization further supports the conclusion that DES 2 and 3 are the most effective solvents for this application.

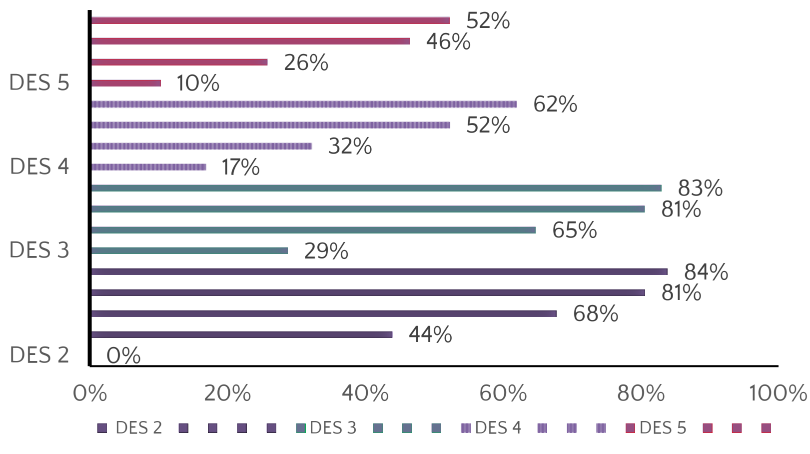

Fig 2. FFA Adsorption Visualization

A fundamental trend is strikingly evident from the graph: for every DES type, the adsorption efficiency increases as the WCO: DES molar ratio decreases. The consistent increase in the length of the bars within each colored group shows this. For instance, the yellow-green bars representing DES 2 show a progressive increase in length, starting from 0% efficiency at a 1:0 molar ratio and peaking at 84% at the 1:6 ratio. This visual trend confirms that a higher concentration of the DES in the mixture leads to a significantly better removal of Free Fatty Acids (FFAs). The longer bars signify a greater adsorption capacity, likely due to a higher availability of active sites or a more favorable kinetic environment for the extraction process. (Ishak et al. 2025). The graph's side-by-side comparison makes this positive correlation between DES concentration and efficiency an undeniable visual conclusion.

Biodiesel Quality Characterization

After converting WCO to Biodiesel, we obtained waste cooking oil biodiesel, which was tested for key fuel properties as per ASTM standards before the start of engine experiments. The result is shown in Figures 3, 4, and 5

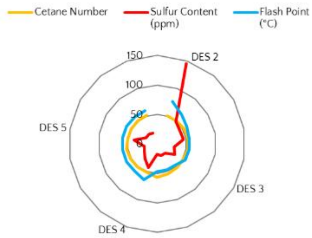

Figure 3. Characterization of Biodiesel Based on Cetane Number, Sulfur Content, and Flash Point

The cetane number is a crucial measure of a diesel fuel's ignition quality. A higher cetane number indicates a shorter ignition delay time, meaning the fuel combusts more quickly and smoothly. This leads to better engine performance, reduced engine knocking, and lower emissions. The data shows that increasing the WCO to DES molar ratio generally increases the cetane number for all DES types. This is likely due to the more complete removal of undesirable components or modifying the fuel's chemical structure at higher solvent concentrations. For instance, in the case of DES 2, the cetane number increases from 50.4 (without DES treatment) to 53.8 at a 1:6 WCO: DES molar ratio. DES 3 and 4 are the most effective at increasing the cetane number. DES 3 achieves the highest cetane number of 56.3 at a 1:6 molar ratio, while DES 4 reaches 52.5 at the same ratio. The cetane numbers for DES 2 and DES 5 are slightly lower, but still improve over the untreated WCO. This suggests that the specific composition of DES 3 is particularly well-suited for improving the ignition quality of the WCO-derived product (Kailas et al. 2025).Sulfur content is a critical environmental and regulatory parameter for fuels. High sulfur levels contribute to air pollution through sulfur oxides (SOx), which can cause acid rain and respiratory problems. Lowering sulfur content is a key goal in modern fuel production. The data consistently show that treating WCO with DES significantly reduces the sulfur content. The sulfur content decreases sharply as the WCO to DES molar ratio increases. The untreated product has a sulfur content of 145 ppm, which is relatively high. Comparison of DES: DES 3 and DES 5 are the most effective at reducing sulfur content. DES 3 lowers the sulfur to a remarkably low 18 ppm at a 1:6 molar ratio, while DES 5 achieves 20 ppm at the same ratio. These values are well within the acceptable limits for many modern diesel fuels. DES 2 and DES 4 also perform well, reducing sulfur to 30 ppm and 22 ppm, respectively. The effectiveness of DES in sulfur removal is a significant advantage, highlighting its potential in producing cleaner fuels (Lei et al. 2023).The flash point is the lowest temperature at which a liquid can vaporize to form an ignitable mixture in air near the liquid's surface. A higher flash point is generally desirable for safety, indicating a lower fire risk during storage, handling, and transportation. The data suggest that treating WCO with DES typically decreases the flash point. This is an interesting and potentially concerning trend.

The untreated product has a flash point of 77 °C, a good safety margin. The flash point drops significantly with the addition of DES. The flash point reduction is most pronounced with DES 3, which lowers the flash point to 47 °C. DES 2 and DES 4 also cause a notable decrease, with the flash point reaching 55 °C and 60 °C, respectively. DES 5 has a flash point of 60 °C at higher molar ratios. While a lower flash point can be a safety concern, it is important to note that many modern diesel fuels have flash points in this range. The flash point reduction might be due to the removal of heavier, less volatile components and the presence of the DES in the final product (Bow et al. 2022).

IV. CONCLUSION

Based on the data, deep eutectic solvents (DES) are highly effective at upgrading waste cooking oil (WCO) into a higher-quality biofuel. Increasing the DES concentration consistently and significantly improved the fuel's cetane number, and reduced its sulfur content and total acid number (TAN). Among the four solvents tested, DES 3 was the most effective overall, yielding the highest cetane number (56.3) and the lowest sulfur content (18 ppm) and TAN (0.12 mgKOH/g) at the 1:6 molar ratio. This confirms that selecting the right DES composition is crucial for optimizing performance. The study also noted a trade-off: DES treatment reduced the flash point of the fuel, which may be a safety concern, but the benefits of cleaner, higher-quality fuel likely outweigh this risk. To improve this research, the following steps should focus on a deeper understanding of the process and its scalability, evaluate sustainability, and a critical next step is to perform a techno-economic analysis and a life cycle assessment (LCA). These studies would evaluate the cost-effectiveness and environmental impact of using DES for WCO refining, including the potential for DES reusability. This would be essential for moving the technology from the lab to a viable industrial scale. Future research should use advanced analytical techniques like FTIR and NMR spectroscopy to understand the chemical mechanisms behind the superior performance of DES 3. This will allow for the rational design of even more effective DES formulations.

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