Review on Polyurethane Solubilization in Deep Eutectic Solvents (DES) for Plastic Recycling

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ABSTRAK

Pelarut eutektik dalam (Deep Eutectic Solvents/DES) adalah kategori baru pelarut ramah lingkungan yang dibuat melalui interaksi antara donor ikatan hidrogen (hydrogen bond donors/HBDs) dan akseptor ikatan hidrogen (hydrogen bond acceptors/HBAs) dalam rasio molar tertentu, yang menunjukkan karakteristik fisikokimia unik. DES memiliki volatilitas rendah, tidak mudah terbakar, dan stabilitas tinggi, menjadikannya pengganti yang ramah lingkungan untuk pelarut konvensional. Sifat fisik utama DES, termasuk densitas, viskositas, dan stabilitas termal, sangat penting untuk fungsinya. Karakteristik ini dipengaruhi oleh parameter seperti suhu, komposisi komponen, dan rasio molar, sehingga memungkinkan penyesuaian untuk tujuan tertentu. DES telah menunjukkan potensi besar dalam berbagai bidang, termasuk elektrokimia, sintesis material, dan proses kimia ramah lingkungan, berkat kemampuan adaptasi dan keamanannya. Dalam bidang pelarutan poliuretan (polyurethane/PU), DES memiliki potensi yang signifikan. Proses pelarutan ini dikaitkan dengan penghancuran ikatan hidrogen dalam polimer dan pelarutan ikatan uretan oleh komponen DES, yang diperkuat oleh jaringan ikatan hidrogen yang kuat. Dengan menyesuaikan sifat-sifat DES, para peneliti dapat meningkatkan proses degradasi PU, menawarkan solusi berkelanjutan untuk masalah limbah plastik. Artikel ini menyoroti karakteristik dasar, sifat fisik dan kimia, berbagai aplikasi, serta potensi DES dalam pengembangan teknologi pelarutan PU.

Kata Kunci: Pelarut eutektik dalam, fisikokimia unik, pelarutan, poliuretan

ABSTRACT

Deep eutectic solvents (DES) are a novel category of environmentally friendly solvents created by the interaction of hydrogen bond donors (HBDs) and acceptors (HBAs) in precise molar ratios, exhibiting unique physicochemical characteristics. DES have low volatility, non-flammability, and great stability, making them ecologically benign substitutes for traditional solvents. The essential physical qualities of DES, including density, viscosity, and thermal stability, are pivotal to its operation. These characteristics are affected by parameters like as temperature, component mix, and molar ratios, enabling customisation for particular purposes. DES have shown considerable promise in several domains, including electrochemistry, material synthesis, and chemical environmentally friendly processes, owing to their adaptability and safety. In the realm of polyurethane (PU) solubilisation, deep eutectic solvents (DES) have notable potential. The solubilization process is ascribed to the breakdown of the polymer's hydrogen bonds and the dissolving of urethane connections by the DES components, helped by their strong hydrogen bond network. By customising DES characteristics, researchers may enhance the breakdown process for PU, offering a sustainable solution to plastic waste issues. The basic features, physical and chemical properties, wide range of uses, and potential for developing PU solubilisation technologies of DES are highlighted in this paper.

Keywords: Deep eutectic solvents, physiochemical properties, solubilization, polyurethane

INTRODUCTION

The most versatile polymer, polyurethane, finds applications in several industries: construction, automobile, furniture, and textiles (1,2). The unique properties it offers are great flexibility, durability, and resistance. Therefore, it could be used to make a variety of products ranging from soft foams to rigid insulation materials (3,4). However, due to the extensive use of this product, PU has become a real problem in regard to its waste products. Being highly resistant to biodegradation, unlike many other polymers, PU results in massive accumulation in landfills and the environment. This persistence contributes to serious ecological risks, such as soil and water contamination, and adds to the mounting plastic waste problem (5). Traditional PU recycling methods usually involve organic solvents, which are toxic and pose a threat to the environment. These methods generally focus on recovering polyols through such processes as glycolysis or hydrolysis (6). For example, some researchers have investigated glycolysis to reclaim polyols from PU foams and elastomers, which can then be reused in the production of new polyurethane products (7). However, most of these methods have some drawbacks, such as excessive use of solvents. complicated purification steps, and low overall efficiency. All these factors constitute a real need for the development of more effective and greener recycling techniques to tackle the environmental impact of PU waste.

In recent years, deep eutectic solvents (DES) have been identified as a viable alternative for the dissolution and recycling of polymers, including polyurethane (PU). DES are synthesised through the combination of a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA), leading to the formation of a eutectic mixture characterised by a melting point that is lower than that of the constituent components (8,9). The distinctive characteristics of this property, combined with their minimal toxicity and elevated solvency, render DES a compelling choice for a range of applications within the

domain of green chemistry (10). DES demonstrates the capability to dissolve a diverse array of polar and nonpolar compounds, thereby rendering it appropriate for the solubilisation of intricate polymers such as PU.

The capacity of DES for the solubilisation of polymers is attributed to their capability to alter the physical architecture of the polymer via hydrogen bonding and van der Waals interactions. The degradation of polymer chains through DES enables the extraction of valuable monomers, thus contributing to the framework of a circular economy (11). This methodology not only decreases dependence on virgin materials but also mitigates the environmental impact linked to the production and disposal of polyurethane. Even though DES has great potential for recycling PU, there are still a number of obstacles to overcome. The efficiency of PU solubilisation in DES is affected by several factors, such as temperature, reaction time, and the solvent's specific composition (12). Moreover, the potential release of hazardous by-products during the degradation process warrants additional investigation. Therefore, the aim of this review is to systematically evaluate the potential of deep eutectic solvents (DES) for the solubilization of polyurethane (PU), by analyzing their physicochemical properties, mechanisms of polymer degradation, and current challenges in applying DES for sustainable plastic recycling

METHODOLOGY

Identification

A total of 189 records were identified through Scopus searches and 125 records through Web of Science (WoS) searches. This was achieved using an advanced search strategy that employed three specific keywords: "Deep Eutectic Solvents," "Plastic," and "Recycling." These terms were carefully selected to capture studies relevant to the application of DES in plastic recycling.

The use of advanced search techniques enabled a refined and focused scope, targeting publications specifically addressing the role of DES in breaking down and recycling plastic materials, a rapidly growing area of interest due to the environmental impact of plastic waste.

The results from both databases (Scopus and WoS) represent a significant portion of the existing research on this topic, providing a strong foundation for the review. Scopus typically offers a broader range of scientific and technical papers, whereas WoS often emphasises high-impact journals, thereby ensuring that the review includes both comprehensive and high-quality studies.

At this stage, it is vital to ensure that no important studies are overlooked. Utilising multiple databases helps minimise bias and provides a well-rounded view of the available literature. Following identification, the next steps involve removing duplicates and applying inclusion and exclusion criteria to further refine the list of publications for detailed analysis. This systematic approach supports the development of a robust and relevant body of literature to understand how DES can be effectively applied in plastic recycling technologies.

Screening Process

In the screening process, the number of relevant records was reduced following initial filtering from Scopus and Web of Science. After this preliminary screening, 88 records remained from Scopus and 78 from Web of Science, resulting in a total of 148 papers. At this point, 33 duplicate records were removed to avoid redundancy.

Subsequently, additional exclusion criteria were applied to refine the selection. Firstly, only papers written in English were included to ensure the review focused on widely accessible scientific literature. Secondly, the scope was narrowed to specific research areas, including Chemistry, Chemical Engineering, Environmental Science, Materials Science, Biochemistry, Genetics and Molecular Biology, Energy, and Engineering. Studies outside these disciplines were considered less relevant to the objectives of the review.

Following the application of these criteria, the number of records was further reduced, ensuring that the remaining literature was both relevant and of high quality. This focused approach aims to highlight the most recent and reliable research on the application of Deep Eutectic Solvents (DES) in plastic recycling within the appropriate scientific domains.

Eligibility

Total of 131 records were assessed after full-text access was obtained. At this stage, further exclusions were made based on several criteria. Papers were excluded if they fell outside the scope of the study, had titles that were not significantly related to the research topic, or contained abstracts that did not align with the study's objectives. Additionally, records without full-text availability were also removed.

Following the application of these exclusion criteria, 98 records were eliminated. Consequently, 33 studies were deemed suitable and included in the qualitative analysis. This step ensured that only the most relevant and accessible studies were retained for in-depth review, thereby enhancing the overall focus and quality of the analysis on the application of Deep Eutectic Solvents (DES) in plastic recycling.

Data Abstraction and Analysis

An integrative analysis was employed as one of the assessment methods in this study to review and synthesise various research designs, particularly quantitative studies. The primary aim was to identify key themes and subthemes related to the research. The initial step in developing these themes involved data collection. Significant research on Deep Eutectic Solvents (DES) for plastic recycling was reviewed, with particular attention paid to methodologies and study outcomes.

In the subsequent stage, the authors collaborated with co-authors to formulate themes based on the evidence gathered. Throughout the data analysis, a reflective log was maintained to record thoughts, interpretations, and any issues that arose. This facilitated a deeper understanding of the data and its implications. Upon establishing the initial themes, the authors compared their findings to identify any inconsistencies in the theme development process. Where discrepancies in interpretation occurred, discussions were held to reach a consensus.

To ensure consistency, themes were refined as necessary. Two subject matter experts, one in Deep Eutectic Solvents and the other in plastic recycling reviewed the analysis to validate the relevance and clarity of the identified subthemes. This expert review phase was critical to ensure each subtheme was appropriately aligned with the research focus. Based on the experts' feedback, further revisions were made to enhance the study's findings.

The guiding research questions were as follows:

- What are the fundamental interaction mechanisms that enable the solubilization of polyurethane in Deep Eutectic Solvents (DES)
- 2. How do the physicochemical properties of different DES formulations influence their effectiveness in dissolving polyurethane?
- 3. What are the current challenges and future prospects of using DES-based

systems for polyurethane recycling compared to conventional methods?

RESULTS AND DISCUSSION Deep Eutectic Solvents (DES)

A deep eutectic solvent can be defined as a combination of two or more pure substances that, upon mixing, demonstrate a notable reduction in melting point relative to the melting points of the constituent components (13,14). The name "eutectic" derives from the Greek word "ευτηκτος," signifying easy or lowest melting, and describes the occurrence in which a mixture solidifies at a temperature lower than that of any of its individual components (15). DES generally consist of a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA). The formation of DES involves the interaction between a quaternary ammonium salt and either metal salts or a HBD which is capable of establishing a complex with the halide anion present in the quaternary ammonium salt. Hydrogen bond acceptors (HBA) include quaternary ammonium chloride and various metal salts such as choline chloride, ChCl. ChCl is one of the most commonly used quaternary ammonium salts because of its relatively inexpensive and available nature, as well as its ability to form stable complexes with HBDs (16,17). Figure 1 illustrates the common hydrogen bond acceptors and donors used in the preparation of deep eutectic solvents, which play a critical role in determining the physicochemical properties of DES. Other HBAs include ZnCl₂, and AlCl₃. These compounds provide the DES backbone and its basic ionic character and solvation properties.

Hydrogen bond donors (HBD) include a variety of compounds such as alcohols, carboxylic acids, amides and amino acids exemplified by ethylene glycol, glycerol, levulinic acid, and urea (18,19). The nature of HBD critically affects the properties that ensue from DES, related, for example, to its viscosity, solubility, and thermal stability. The ability to form the eutectic mixture depends on the capability for interaction between HBAs and HBDs, which causes a significant depression in its melting point. Figure 2 shows the phase diagrams of deep eutectic solvents (green) and a typical ideal eutectic combination (red).

DES is formed by the mixture of HBAs and HBDs in certain molar ratios (20). The ratios can be changed to attain the desired properties of the solvent. The eutectic point, at which the mixture has the lowest melting temperature, is considered the point of strongest interactions between the components. This property enables DES to remain liquid at temperatures where its individual components would normally solidify. DES preparation is quite simple, involving only the mixing of components at slightly higher temperatures to ensure they are fully dissolved (21). The final mixture is homogeneous and ready for use as a solvent. By adjusting the HBA and HBD ratios, researchers can design DES with tailored properties for different purposes.

DES are non-volatile, non-flammable, and easy to store, making them practical for various applications. DESs offering ecofriendly, cost-effective, and easily producible solvents. Their synthesis is atom-efficient and does not require purification, making large-scale use feasible (22). Table 1 shows the advantages, disadvantages, and limitation of DES. Overall, DESs combine the advantages of other green solvents without their drawbacks, positioning them as a promising medium for organic reactions. DES dissolve polyurethane primarily by disrupting hydrogen bonds and other intermolecular interactions that hold the polymer chains together, causing the material to swell, weaken, and eventually break down into smaller molecules.

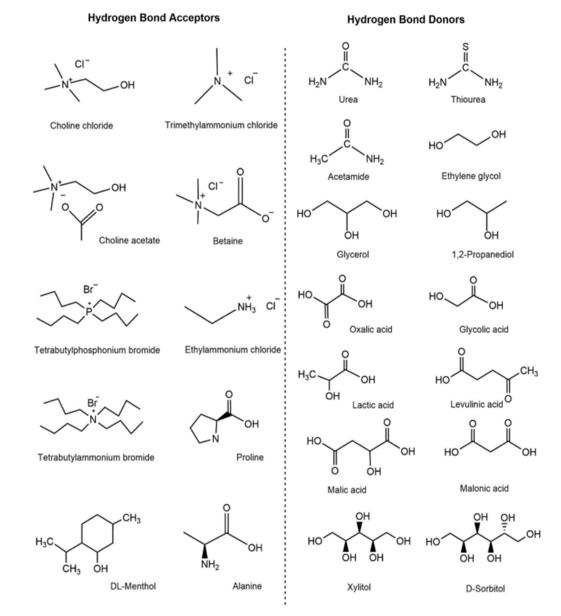


Figure 1: Common hydrogen bond acceptors and hydrogen bond donors used for preparation of deep eutectic solvents (15).

Physiochemical Properties Of DES

DES exhibit a range of beneficial characteristics that make them appropriate for diverse applications. One of the most significant characteristics of DES is their density. The density of deep eutectic solvents represents a significant physicochemical characteristic that performance across affects their diverse applications. DES exhibit densities greater than that of water, with values spanning from 1.0 to 1.6 g.cm⁻³ at a temperature of 25 °C. The density of a DES is contingent upon its compositional elements, wherein the incorporation of heavier HBAs or HBDs results in an elevation of density. The observed densities of DES exceed those of

pure HBDs indicating potential alterations in molecular structure throughout the formation of DES (25,26). The molar ratios of the components in DES significantly influence their density characteristics.

The elevation of the ratio of hydrogen bond acceptors to hydrogen bond donors results in a decrease in the density of the mixture. DES incorporating metal salts exhibit greater density compared to their counterparts devoid of such additives. Conversely, DESs characterised by hydrophobic properties may present densities that fall below that of water (24).

Phase Deep eutectic solvents (DESs)	Advantages Fast and easy synthesis	Disadvantages	Limitations	
		Low conductivity compared to aqueous electrolytes		
	Low cost	Low affinity toward hydrocarbons	Low conductivity	
	Biodegradable	Viscosity		
	Green solvents	-		
	Low vapour pressure			
	High chemical stability			

 Table 1:
 The advantages, disadvantages and limitation of DESs (23).

The variation in density of DESs is influenced by temperature, typically exhibiting a decrease with an increase in temperature (25).

Viscosity is an essential feature that influences the flow characteristics and mass transfer of solvents. It has been established that most of DESs exhibit characteristics of a viscous liquid at ambient temperature. The presence of a comprehensive hydrogen bond network, along with interactions such as van der Waals forces and electrostatic interactions between hydrogen bond donors and acceptors in the components of DESs, contributes to the elevated viscosity and reduced ionic mobility observed in the limited void volume within the liquid DESs. The viscosities of DESs are influenced by the chemical characteristics of HBAs and HBDs, as well as by temperature, molar mass, and the molar ratio (28). The hydrogen bond network between HBAs and HBDs is broken down by temperature, which results in decreased DES viscosities as the temperature rises. In the technology, **DESs** realm of green

characterized by low viscosities present significant interest. Their design can be approached by utilizing small-sized HBD and cations, in accordance with the principles outlined in Hole theory (29).

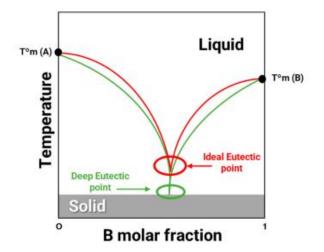


Figure 2: Phase diagrams of deep eutectic solvents (green) and a typical ideal eutectic combination (red).

Abbott et al. indicated that the viscosity of deep solvents formulated with choline eutectic chloride and glycerol diminishes upon the incorporation of organic salts (26). This phenomenon is attributed to a significant decrease in glycerol viscosity, which results from the partial disruption of its hydrogen bond network. Thermal stability is a crucial physiochemical characteristic of DES. These solvents can endure high temperatures without decomposition, which is crucial for several industrial applications necessitating increased thermal conditions (27). The thermal stability of DES is affected by their composition, specifically the selection HBAs and HBDs. For example, DES comprised of quaternary ammonium salts and polyols have significant heat stability, making them appropriate for applications in catalysis and electrochemistry. The capacity to sustain stability at elevated temperatures enables

DES to be used in procedures that conventionally need more volatile and less stable solvents.

Applications Of DES

Deep eutectic solvents (DES) exhibit distinctive characteristics that render them suitable for electrochemical applications, including electrolytes in batteries. supercapacitors, and electrochemical cells. Their increased ionic conductivity, stability, low volatility, and non-flammability improve efficiency and safety (28,29). DES may function as a medium for electrosynthesis, electroplating, and ionic liquid solvents, with adjustable their qualities facilitating optimisation for energy storage and conversion technologies. In accordance with green chemistry principles, DES enhance sustainability by minimising environmental

effect. They possess minimal toxicity, are biodegradable, recyclable, and economically viable, making them appropriate for extensive use as substitutes for hazardous organic solvents in chemical processes (11,20). Their clear synthesis further endorses environmentally sustainable techniques in industrial applications. DES are also advantageous in the synthesis of polymers and nanomaterials, providing superior solvating capabilities for polymer blends, composites, and nanoparticle dispersion (10,31). They improve the mechanical and thermal characteristics of materials and facilitate applications such as electrospin nanofiber production for filtration, tissue engineering, and drug delivery. The adjustable characteristics of DES enable customisation of their qualities for certain material applications.

Mechanisms of Polyurethane Solubilization in DES

Eutectic Solvents (DES) Deep have distinctive physicochemical characteristics, including strong hydrogen bonding and ionic interactions, rendering them efficient in polyurethane (32). The dissolving solubilisation of PU in DES is mainly facilitated by hydrogen bonding between the urethane linkages and hydroxyl groups in PU and HBDs in DES, such glycerol or urea (33)

DES	Molar ratio	Т (°С)	Density (g/cm^3)	Viscosity (cP)	Conductivity (<i>mS/cm</i>)	Biodegradability (%)	Preparation, T (°C)
ChCl-Urea	1:2	12	1.25	750 (25°C)	0.199 (40°C)	97.1	75
ChCl- Glycerol	1:2	-40	1.18	376 (20°C)	1.05 (20°C)	95.9	100
ChCl- Glucosa	1:2	14	1.21 (85°C)	8045.1 (25°C)	-	82.0	80
ChCl- Malonic acid	1:2	10	1.25	1124 (25°C)	0.36	76.3	100
ChCl- Tartaric acid	1:2	47	1.27 (30°C)	66441 (30°C)	0.014 (30°C)	84.6	100
ChCl-Citric acid	1:2	69	1.33 (30°C)	289 (75°C)	0.018 (30°C)	81.6	100
ChCl-p- TsOH	1:1	27	1.21 (30°C)	183 (30°C)	0.04 (30°C)	80.4	100
ChCl-ZnCl ₂	1:2	24	-	8500 (25°C)	0.06 (42°C)	-	100
ChCl-SnCl ₂	1:2	37	-	-	-	-	100
ChCl-FeCl ₃	1:2	65	-	-	-	-	100
Tartaric acid-DMU	3:7	70	-	-	-	-	90
L-Carnitine- Urea	2:3	74	-	-	-	-	80

Table 2:Physical properties of some DESs (25) (34).

This interaction interferes with the intermolecular forces sustaining the PU structure, resulting in

swelling and polymer degradation. Ionic interactions between quaternary ammonium

salts (HBAs) in deep eutectic solvents (DES) and the polar groups of polyurethane (PU) enhance solubilisation by destabilising the polymeric structure of PU and inhibiting re-aggregation (33). Temperature and reaction duration considerably influence the solubilisation process, since higher temperatures increase molecular kinetic energy and facilitate DES penetration into the PU matrix (12). Optimising is necessary since greater temperatures can increase energy usage and the possibility of hazardous by-products. The composition of DES, particularly the ratio of HBA to HBD, affects solubilisation efficiency, with modifications enhancing hydrogen bonding and ionic interactions. Table 2 summarizes the physical properties of selected deep eutectic solvents (DES), including their molar ratio, preparation temperature, density, viscosity, biodegradability. conductivity, and These physicochemical characteristics are essential because they directly influence the ability of DES to interact with and solubilize polymer structures like polyurethane. For instance, lower viscosity and higher conductivity are often associated with enhanced solvent penetration and more efficient polymer degradation. Understanding these properties allows researchers to tailor DES formulations for optimal performance in recycling and green chemistry applications.

The solubilisation of PU in DES has significant implications for recycling and sustainability, facilitating the recovery of important monomers and promoting circular economy concepts. DES, being a biodegradable and less toxic option compared to conventional organic solvents, provides a safer and more sustainable solution for PU recycling and disposal, in accordance with green chemistry principles.

CONCLUSION

Deep eutectic solvents constitute a promising and sustainable method for the solubilisation and recycling of polyurethane materials. Utilising their distinct characteristics, DES enables the decomposition of PU into recoverable constituents, thereby promoting sustainable methodologies within the plastic economy. Ongoing investigation in this domain is crucial to comprehensively harness the capabilities of DES in tackling the issues associated with PU waste.

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