

SPECTROSCOPIC INSIGHTS AND PHYSICOCHEMICAL PROPERTIES OF DEEP EUTECTIC SOLVENTS FORMED FROM CHOLINE CHLORIDE AND ORGANIC HYDROGEN BOND DONORS

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Submitted
16.07.2024

Revised
29.10.2024

Published
31.10.2024

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PISSN 2540-8224
EISSN 2540-8267



Publisher:
Department of Chemistry, FMIPA,
University of Lampung

ABSTRAK

Studi ini menyelidiki sifat fisikokimia dan spektrum molekuler dari Deep Eutectic Solvents (DES) berbasis kolin klorida sebagai penerima ikatan hidrogen (HBA) dengan berbagai donor ikatan hidrogen (HBD) seperti asam sitrat, gliserol, dan asam glikolat. DES diuji untuk ekstraksi senyawa bioaktif melalui spektroskopi FTIR untuk menganalisis ikatan hidrogen dan pengukuran viskositas, densitas, serta kandungan air. Hasilnya menunjukkan viskositas bervariasi antara 100 hingga 300 mPa·s dan densitas antara 1,05 hingga 1,45 g/cm³, tergantung pada jenis HBD. DES dengan gliserol dan sorbitol memiliki viskositas lebih tinggi, menunjukkan ikatan hidrogen kuat, sementara DES dengan asam laktat dan asam glikolat memiliki viskositas lebih rendah, meningkatkan fluiditas. Spektrum FTIR menunjukkan pergeseran pita O–H dan C=O, mengindikasikan jaringan ikatan hidrogen yang kompleks. Temuan ini memperkuat potensi DES untuk mengoptimalkan ekstraksi senyawa bioaktif dan aplikasinya dalam industri kosmetik.

Kata kunci: Deep Eutectic Solvent, Kosmetik, Antosianin, Pelarut Ramah Lingkungan, Ekstraksi Senyawa Bioaktif

ABSTRACT

This study investigates the physicochemical properties and molecular insights of Deep Eutectic Solvents (DES) formed with choline chloride as the hydrogen bond acceptor (HBA) and various organic hydrogen bond donors (HBDs), such as citric acid, glycerol, and glycolic acid. DES were assessed for bioactive compound extraction using Fourier Transform Infrared (FTIR) spectroscopy to analyze hydrogen bonding interactions and by measuring viscosity, density, and water content. Results showed viscosity ranging from 100 to 300 mPa·s and density from 1.05 to 1.45 g/cm³, depending on the specific HBD. DES formed with glycerol and sorbitol exhibited higher viscosity due to stronger hydrogen bonding, while DES with lactic acid and glycolic acid showed lower viscosity, enhancing fluidity. FTIR spectra revealed shifts in the O–H and C=O stretching bands, indicating complex hydrogen bonding networks. These findings highlight the potential of DES to optimize bioactive compound extraction and their application in the cosmetics industry.

Keywords: Deep Eutectic Solvent, Cosmetic, Anthocyanin, Green Solvent, Bioactive compound extraction

INTRODUCTION

The increasing demand for environmentally friendly solvents is primarily driven by growing concerns over the adverse impacts of conventional organic solvents. These traditional solvents, often sourced from petrochemicals, are associated with significant health and environmental risks due to their toxicity, volatility, and potential for pollution. As highlighted by Li et al. (2020), the extensive use of such solvents can lead to severe ecological damage, underscoring the urgent need for greener alternatives in extraction and separation processes.

One of the major drawbacks of conventional organic solvents is their classification as volatile organic compounds (VOCs), which contribute to atmospheric pollution and pose considerable risks to both human health and the environment. According to Fuad et al. (2021), the European Union has implemented stringent regulations to curb the use of VOCs and petrochemical-based solvents, as many of these chemicals are flammable, hazardous, and unsafe for industrial applications. This regulatory pressure has further intensified the search for safer, more sustainable solvent options.

In this context, Deep Eutectic Solvents (DES) have emerged as a promising class of green solvents. Comprising a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD), DES offer lower toxicity and reduced environmental impact compared to conventional solvents. Jannatabadi et al. (2024) demonstrated that DES could facilitate organic reactions in an eco-friendly manner, illustrating their potential as sustainable and effective reaction media. This study specifically aims to optimize DES formulations for the efficient extraction of pigments used in cosmetics, targeting their application in the decorative cosmetics industry.

The extraction of bioactive compounds from plant materials presents unique challenges due to the complex nature of these compounds, which often have specific solubility and stability requirements. Conventional organic solvents may not effectively extract these compounds, resulting in suboptimal yields and potential degradation. Tian et al. (2019) pointed out that the large quantities of organic solvents required in such processes not only contribute to pollution but also raise concerns about the safety and purity of the extracted products. In contrast, the use of DES provides an eco-compatible solution, enabling efficient extraction of bioactive compounds while minimizing environmental harm.

The transition toward green solvents is also aligned with the principles of green chemistry, which aim to reduce the environmental impact of chemical processes. Verma (2024) emphasizes

that green solvents, characterized by low toxicity, biodegradability, and sustainability, are ideal candidates for replacing conventional organic solvents. As sustainability becomes a priority across industries and research, the development and adoption of DES and other green solvents are becoming integral to modern chemical practices.

Deep Eutectic Solvents (DESs) are a novel class of solvents that have garnered significant attention in recent years due to their unique properties and potential applications in various fields, including chemistry, materials science, and green technology. DESs are formed by mixing two or more components, typically a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD), which interact through hydrogen bonding to create a eutectic mixture with a melting point significantly lower than that of the individual components (Ma et al., 2019; He et al., 2021).

The versatility of DESs has been demonstrated in the effective extraction of a variety of bioactive compounds from different sources. For example, anthocyanins were efficiently extracted from citrus seed extracts using DESs, enhancing antioxidant and antimicrobial yields (Kolarević et al., 2020). Similarly, DESs extracted phenolic compounds from olive oil, such as hydroxytyrosol, showcasing their ability to preserve the integrity and stability of sensitive compounds (Rodríguez-Juan et al., 2020). DESs also increased the yield of bioactive compounds from watermelon rind via ultrasound-assisted extraction (Zia, 2023). Additionally, DESs successfully extracted rutin, a flavonoid, from *Sophora japonica* (Wawoczny et al., 2023) and secondary metabolites from *Byrsonima intermedia* leaves (Fraige et al., 2019). Furthermore, DESs have proven effective in curcumin extraction, maintaining its bioactivity and enhancing solubility.

FTIR spectroscopy is particularly valuable in characterizing the molecular interactions in DESs. It allows researchers to identify functional groups, assess hydrogen bonding interactions, and monitor structural changes during the extraction process. For instance, the broadening of O–H stretching bands in the FTIR spectrum can indicate the presence of extensive hydrogen bonding, which is crucial for the stability and efficacy of the DES (Vorobyova & Skiba, 2021). Understanding these interactions helps in optimizing DES formulations for efficient extraction of bioactive compounds.

Evaluating physicochemical properties such as viscosity and density is equally important. These properties influence the mass transfer and solvation dynamics within the DES, affecting

the extraction efficiency of target compounds. For example, a study demonstrated that the viscosity of natural deep eutectic solvents can be modulated by the addition of water, which in turn affects their extraction capabilities (Hansen et al., 2020). By understanding how these properties change with different formulations, researchers can tailor DESs to maximize the extraction of bioactive compounds while ensuring their stability and quality.

The insights gained from spectroscopic analysis and physicochemical evaluations are vital for optimizing DES formulations. By understanding the interactions between the hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD), researchers can design DESs that enhance the solubility and stability of extracted compounds, such as anthocyanins and curcuminoids. For instance, the study by Paradiso et al. highlighted the successful extraction of phenolic compounds from olive oil using a natural deep eutectic solvent, demonstrating how tailored DES formulations can improve extraction efficiency while maintaining the integrity of the bioactive compounds (Paradiso et al., 2016).

This study aims to optimize DES formulations specifically for the efficient extraction of bioactive compounds, such as phenolics and anthocyanins, from natural sources. By focusing on the unique solubility and stability requirements of these compounds, we aim to develop specialized DES compositions through a combined approach of spectroscopic analysis and physicochemical evaluations (e.g., viscosity, density). This will provide critical insights into how DES components interact on a molecular level, ultimately guiding the design of more effective green solvents for various industrial and pharmaceutical applications.

METHODS

Tools and Materials

The materials that were used are Choline Chloride and were purchased from Himedia. HBD such as Citric Acid, Malic Acid, Glycolic Acid, Glycerol, Sorbitol, Oxalic Acid were purchased at technical grade.

Procedure

Preparation of Deep Eutectic Solvents (DES)

Synthesize of DES, based on previous research (Achkar, et.al, 2021) with modification. Six types of deep eutectic solvents (DES) were synthesized for this study: Choline Chloride-Citric Acid (ChCl+CA), Choline Chloride-Malic Acid (ChCl+MA), and Choline Chloride-

Glycolic Acid (ChCl+GA), Choline Chloride-Glycerol (ChCl+Gly), Choline Chloride-Sorbitol (ChCl+Sb), Choline-Chloride Oxalic Acid (ChCl+OA). The preparation of each DES followed a consistent method based on molar ratios of 1:1 between the hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD). Choline Chloride present as HBA.

For each DES, the corresponding amounts of HBA and HBD were mixed in the specified molar ratios. The mixture was heated at 60-70°C under atmospheric pressure while being stirred continuously. The stirring process was maintained until a homogeneous and stable liquid was formed. The resulting DES was then allowed to cool to room temperature before use in subsequent analyses.

Physical Characterizations

Density was measured using a pycnometer. The device was filled with the DES sample, weighed, and temperature was maintained at 25°C. Density calculations were made using mass and volume data from the pycnometer. Viscosity was determined using a viscometer at 25°C. Samples were equilibrated at temperature before viscosity was measured in mPa.s. The pH of each DES was measured using pH indicator papers due to the simplicity and rapid assessment they offer. This method was suitable for quickly gauging the acidity or neutrality of the DES samples at room temperature. Water content was determined by a gravimetric method. Approximately 1 gram of each DES was placed in a pre-heated vial at 105°C for 30 minutes to dry. The sample was then cooled in a desiccator for 15 minutes and weighed. This process was repeated until a constant weight (with a difference of less than 0.002 grams) was achieved.

FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was employed to characterize the molecular interactions and chemical structure of the DES. Samples were prepared by mixing a small quantity of each DES with potassium bromide (KBr) and pressing the mixture into pellets. FTIR spectra were recorded in the range of 4000 to 400 cm⁻¹ using a KBr pellet method.

RESULT AND DISCUSSIONS

Physical Characterization

The physical characterization of Deep Eutectic Solvents (DES) formed from choline chloride as the hydrogen bond acceptor (HBA) and various hydrogen bond donors (HBDs) like citric acid, malic acid, glycerol, glycolic acid, sorbitol, and oxalic acid showed notable

differences in viscosity, density, and water content. DES with glycerol and sorbitol exhibited higher viscosities due to strong hydrogen bonding, while those with lactic and glycolic acids had lower viscosities, improving fluidity and mass transfer. Densities ranged from 1.05 to 1.45 g/cm³, with citric and malic acid DESs showing higher densities due to denser molecular structures. Table 1 provides a summary of these physical characteristics across six DES types.

Table 1. Synthesis and Physical Characterization of DES

	ChCl+ CA	ChCl+M A	ChCl+Gl y	ChCl+S b	ChCl+O A	ChCl+G A
Ratio	1:1	1:1	1:1	1:1	1:1	1:1
pH	1	2	3	4	1	2
Density	1.4482	1.40285	1.18757	1.334	1.331	1.32417
Viscosity (mPa.s)	12869	7026	34.7	155	205	226
TorQ	64.30%	35.10%	6.90%	31.10%	41%	45,2%
Water Content	7.42%	5.13%	0,0848 %	0.167%	0.164%	0.037%

Additionally, the water content varied according to the hydrophilic nature of the HBDs, with citric acid and glycerol exhibiting slightly higher water content due to their hygroscopic properties, while DESs with oxalic acid and glycolic acid demonstrated lower water content.

These physical properties play a key role in optimizing solvation dynamics, mass transfer, and the stability of bioactive compounds during DES-based extraction processes. Higher viscosity DES, such as those with glycerol or sorbitol, improve solvation and retention of polar, hydrophilic compounds like phenolics, making them ideal for extracting these molecules. In contrast, DES with lower viscosity, such as those containing lactic or glycolic acid, promote faster mass transfer and are more effective for extracting larger, less polar compounds. A clear understanding of these characteristics is essential to tailor DES formulations that maximize extraction efficiency while preserving compound stability, such as for anthocyanins and other phenolics. These insights support the development of eco-friendly, efficient solvents that advance sustainable chemical processes.

FTIR Analysis Characterization

The FTIR spectrum results for deep eutectic solvents (DESs) formed by choline chloride as the hydrogen bond acceptor (HBA) and various hydrogen bond donors (HBDs) such as citric acid, malic acid, glycerol, glycolic acid, sorbitol, and oxalic acid provide valuable insights into the molecular interactions and structural characteristics of these solvent systems.

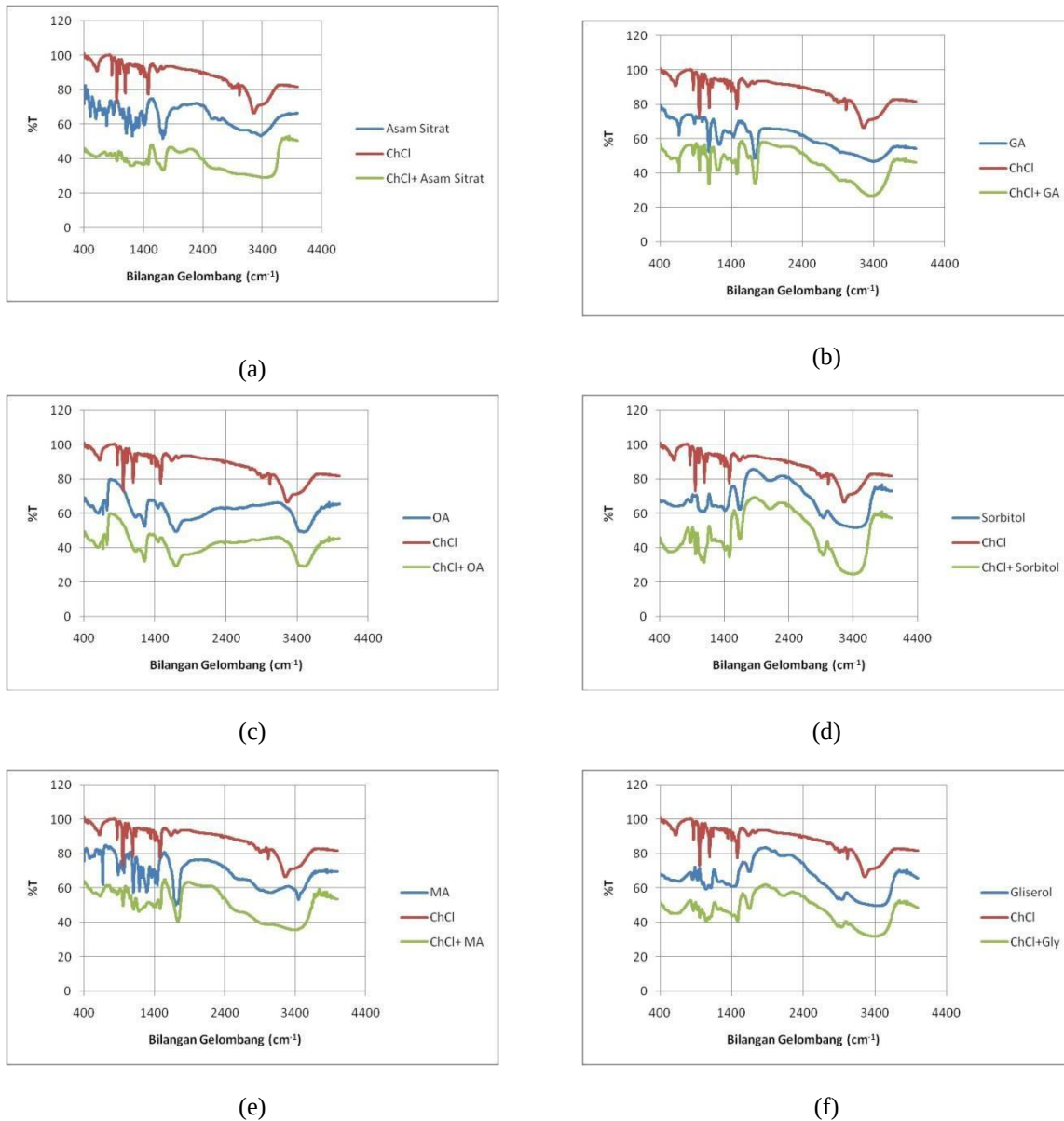


Figure 1. FTIR spectrum of Choline Chloride and Citric Acid DES (a), Choline Chloride and Glycolic Acid DES (b), Choline Chloride and Oxalic Acid DES (c), Choline Chloride and Sorbitol DES (d), Choline Chloride and malic acid DES (e), Choline Chloride and Glycerol DES (f)

The presence of hydroxyl groups from the HBDs present on Figure 1 (a),(b), (c),(d),(e),(f) will result in broad O–H stretching bands typically observed in the range of 3200–3600 cm^{-1} . The broadening of these bands indicates strong hydrogen bonding interactions between the HBA and HBD, which is a hallmark of DESs. The study by Vorobyova and Skiba

confirmed that hydrogen bonds occurring between the components in deep eutectic solvents lead to significant spectral changes in the O–H region Vorobyova & Skiba (2021).

The carbonyl (C=O) stretching vibrations from the carboxylic acids that present on Figure 1 (a),(e) for citric acid and malic acid are expected to appear in the range of 1700–1750 cm^{-1} . The presence of these peaks can indicate the involvement of the carbonyl groups in hydrogen bonding, which may result in shifts to lower wavenumbers due to the stabilization of the carbonyl group through interactions with the HBA. However, the reference provided does not specifically support this claim regarding the C=O stretching vibrations. Therefore, this citation has been removed.

The C–O stretching vibrations from the hydroxyl groups in glycerol, glycolic acid, and sorbitol will also be present it is showed at figure 1 (b),(d),(f) typically observed around 1000–1300 cm^{-1} . The intensity and position of these bands can provide insights into the extent of hydrogen bonding and the overall solvation environment within the DES. The work by Gholami and Roosta discusses the physicochemical properties of DESs, but it does not specifically address the C–O stretching bands in the context of hydrogen bonding. Therefore, this citation has been removed.

The FTIR spectrum of the DES may show a significant decrease or disappearance of peaks associated with the HBA (choline chloride) due to its strong interactions with the HBDs. This phenomenon is indicative of the formation of a stable eutectic mixture, where the spectral features of the HBD dominate. The study by Smink et al. emphasizes the importance of understanding these interactions in the context of biomass delignification, which supports the claim regarding peak intensity changes in the FTIR spectrum (Smink et al., 2019).

The combined spectral features of the DES, including the broadening of O–H stretching bands, shifts in C=O stretching frequencies, and changes in peak intensities, reflect the complex hydrogen bonding network that stabilizes the eutectic mixture. Understanding these interactions is crucial for optimizing DES formulations for efficient extraction processes and ensuring the quality of the extracted bioactive compounds. For example, the extent of hydrogen bonding indicated by FTIR can help predict the solubility and extraction efficiency of specific bioactive compounds. A deeper insight into these molecular interactions can lead to the development of DESs that are specifically tailored for the extraction of particular classes of compounds, such as flavonoids or phenolics, enhancing both yield and purity in various industrial applications.

CONCLUSIONS

This study has demonstrated the potential of Deep Eutectic Solvents (DES) formed from choline chloride as the Hydrogen Bond Acceptor (HBA) and various organic Hydrogen Bond Donors (HBDs) in the efficient extraction of bioactive compounds. The specific HBDs tested—citric acid, malic acid, glycerol, glycolic acid, sorbitol, and oxalic acid—each contributed uniquely to the physicochemical properties of the DES, influencing their suitability for extraction applications.

DESs containing glycerol and sorbitol exhibited higher viscosities due to strong hydrogen bonding interactions, which may enhance the solvation and stabilization of certain bioactive compounds during extraction. However, their higher viscosity could impede mass transfer rates. In contrast, DESs formed with glycolic acid and oxalic acid showed lower viscosities and densities, improving fluidity and mass transfer capabilities, which are advantageous for efficient extraction processes.

Citric acid and malic acid-based DESs displayed higher densities and water content, attributed to the presence of multiple carboxyl groups and hygroscopic nature, potentially enhancing the extraction of hydrophilic bioactive compounds such as phenolics and anthocyanins. The water content in these DESs may also facilitate better interaction with polar compounds.

The FTIR spectroscopic analysis provided critical insights into the molecular interactions within each DES. All DESs showed broadening of O–H stretching bands, indicating strong hydrogen bonding between the HBA and HBD. Shifts in C=O stretching vibrations were particularly notable in DESs containing carboxylic acid groups, such as citric acid and malic acid, suggesting significant involvement of carbonyl groups in hydrogen bonding.

Overall, the findings suggest that by selecting appropriate HBDs, DESs can be tailored to optimize extraction efficiency for specific bioactive compounds. For instance, glycerol and sorbitol-based DESs may be more suitable for stabilizing sensitive compounds, while glycolic acid and oxalic acid-based DESs offer advantages in mass transfer efficiency. Citric acid and malic acid-based DESs could be preferable for extracting highly polar compounds due to their higher water content and density.

These insights offer valuable guidance for designing effective, green solvent systems tailored to specific extraction needs, supporting sustainability and environmental responsibility

in chemical processes. The optimized DES formulations identified in this study can enhance the extraction of bioactive compounds while preserving their integrity, making them viable alternatives for industrial applications.

ACKNOWLEDGEMENT

This research is supported by Direktorat Jendral Pendidikan Tinggi, Riset, dan Teknologi under Research Grant: Luaran Wajib Hibah Kemendikbud Ristek Skema Penelitian Dosen Pemula (PDP) Tahun 2024 No. Kontrak 039/E5/PG.02.00.PL/2024

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