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RESEARCH ARTICLE

Lignin extraction from empty fruit bunch fibre using different types of alpha-hydroxy acid-based deep eutectic solvents via microwave-assisted heating

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Abstract - This study investigates the efficiency of deep eutectic solvent (DESs) synthesized using choline chloride (ChCl) as a hydrogen bond acceptor (HBA) and alpha-hydroxy acids (AHAs) as hydrogen bond donors (HBDs) for lignin extraction from oil palm empty fruit bunches (EFB) via microwave-assisted reaction. DESs with varying molar ratios of ChCl to AHAs, specifically oxalic acid (OA) dihydrate, known as dicarboxylic acid, and lactic acid (LA), a monocarboxylic acid, were prepared at 1:1 and 1:10 ratios, respectively. The presence of hydroxyl (OH) groups in AHAs facilitated DES formation at both ratios. ChCl: LA demonstrated a significantly higher lignin extraction yield, 76.61%, compared to ChCl: OA dihydrate, 29.36%. This is due to the lower viscosity and higher mobility of the DESs, which enhance solvent-solute interactions. The Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed strong intermolecular bonding in the extracted lignin, with characteristic peaks corresponding to phenolic, aliphatic hydroxyl, and benzene ring vibrations, indicating the preservation of the lignin's aromatic structure. Thermogravimetric Analysis (TGA) showed that lignin extracted with ChCl: OA dihydrate and ChCl: LA exhibited higher thermal stability and char yield than raw EFB, with distinct degradation stages. Differential Scanning Calorimetry (DSC) determined the lignin samples' glass transition temperatures (T_g), with ChCl: LA showing the highest T_g at 90 °C, attributed to its higher purity and reduced carbohydrate content. Overall, the study highlights the critical role of HBD functional groups and DES composition in optimizing lignin extraction efficiency and thermal properties, with potential implications for biomass pretreatment processes via microwave-assisted reaction.

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1. Introduction

Global warming, the depletion of fossil fuels, and other environmental concerns have made finding sustainable alternative renewable energies a top priority in recent years. The process of converting biomass waste into valuable energy or products is known as "biomass valorisation" and is a growing trend [1]. The world's biomass production is estimated at 1,880 billion tons annually. Malaysia, the second-largest palm oil producer after Indonesia, accounts for 38% of the global market and produces almost 50 million tons of dry biomass annually [1] -[2]. Palm oil extracted from fresh empty fruit bunch (FFB) accounts for only 10%, with 90% discarded. Common disposal methods for oil palm empty fruit bunches (EFB) include burning or dumping at plantation sites for natural decomposition. However, such practices result in greenhouse gas emissions and create breeding habitats for rats and snakes [2]. Thus, EFB, with a rich composition of cellulose, hemicellulose, and lignin biopolymers, has been explored as a renewable feedstock for producing various bio-products [3]. Lignin, after cellulose, is the most abundant biopolymer on Earth, comprising 30% of all organic carbon. Since lignin is an amorphous substance with a greater molecular weight and a highly branched and substituted polymer of phenylpropane units, its structure is complex. The three main phenylpropane units in lignin are syringyl (S), guaiacyl (G), and p-hydroxyphenyl (H), which vary in the aromatic rings of o-methyl substitution [2]. Consequently, lignin has a variety of functional groups available. Lignin is considered a versatile resource because of its unique aromatic structure and renewable nature [3]. Although a significant amount of lignin is utilised as fuel to power the pulping process, it is often regarded as a waste product of the pulp and paper industries. As the primary renewable source of many aromatic compounds, lignin has the potential to serve as an effective feedstock for the synthesis of aromatic chemicals and fuels. Approximately 50% of lignin's composition comprises aromatic hydrocarbons [4].

Since lignin is the most recalcitrant and underutilised component, it can be upgraded into valuable polymers, materials, and chemicals. Therefore, pretreatment was an important step in the biomass conversion process, as it could break down the naturally recalcitrant structures of lignocellulosic biomass. A practical, affordable, and environmentally friendly delignification method would meet requirements such as ease of use, cost-effectiveness, recyclability, and the absence of specialised equipment [5]. Several pretreatment techniques are available to convert biomass. Previous studies revealed the use of physical methods (such as grinding and refining), chemical methods (such as alkaline, acid, ionic liquid, and deep eutectic solvent), physicochemical methods (such as ammonia fibre expansion (AFEX) and carbon dioxide (CO_2) explosion), biological methods, and combined approaches [6]. Deep-eutectic solvents (DESs) have emerged as a green solvent among various pre-processing techniques, offering numerous benefits, including ease of preparation, high purity,

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low toxicity, high biodegradability, low melting points, high thermal stability, low volatility, non-flammability, and high air stability. DESs might adhere to the twelve guiding principles of Green Chemistry [7].

DESs are currently considered a highly effective pretreatment technique for reducing the recalcitrance of lignocellulosic biomass. The most effective DESs were found to dissociate the lignin-carbohydrate complex and lignin substructures, specifically aryl ether linkages and carbon-carbon bonds [8]. DESs typically consist of a hydrogen-bond acceptor (HBA, such as quaternary ammonium salts) and a hydrogen-bond donor (HBD, such as amides, carboxylic acids, and polyols), thereby initiating an alternative biomass processing method. It can be prepared using two or three ingredients, with a specific HBD-to-HBA ratio [9]. Figure 1 shows an example of a delignification mechanism using DESs (Choline chloride: oxalic acid dehydrated) [10]. By combining a dicarboxylic acid (oxalic acid dihydrate) and a monocarboxylic acid (lactic acid) with choline chloride via microwave-assisted heating, DESs are utilised in this work to improve lignin extraction from EFB.

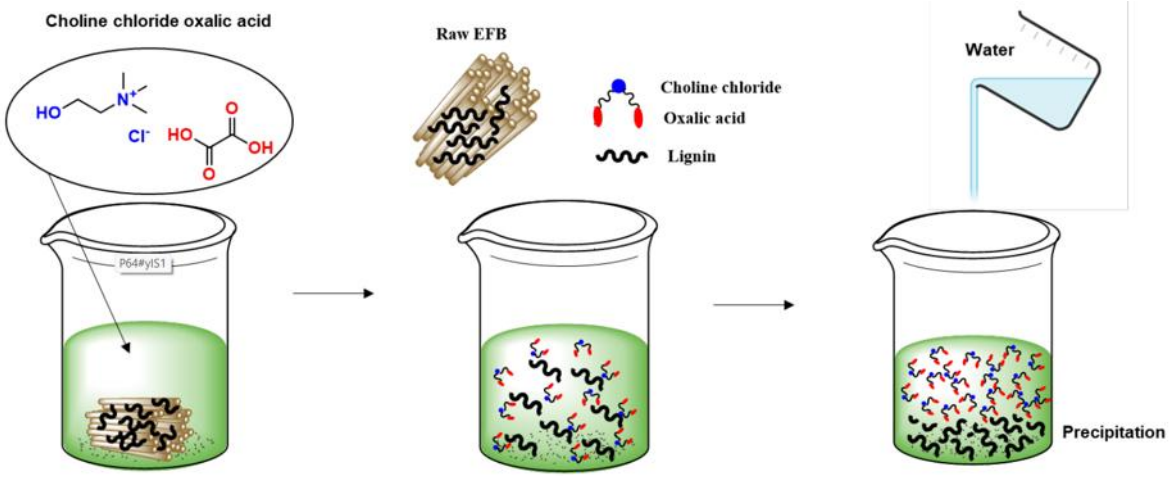

Figure 1. Delignification using DESs (Choline chloride: oxalic acid dehydrated) [10]

DESs combined with microwave-assisted heating (MAH) had a synergistic effect on efficiently cleaving the lignin-carbohydrate complex structure and increased the delignification rate [11]. Besides, microwave-assisted heating (MAH) processes have proven promising in pre-treating biomass due to the shorter processing time required compared with other technologies. This technology also lowers the severity of ionic liquid and alkaline pretreatments. The potential for easy control of reaction conditions, fast and selective heating, low reaction temperatures, and low energy requirements is an additional advantage of microwave-assisted heating [5], [12]. Previous researchers reported that 80% of the lignin was removed from poplar within 3 min during microwave-assisted heating (MAH) using a choline chloride: oxalic acid DES pretreatment. However, pretreatment with choline chloride: oxalic acid DESs alone required 9 h at 110 °C to achieve similar lignin-removal activity [13]. The use of DESs in microwave-assisted processes seems to be a promising approach for the clean fractionation of lignocellulosic biomass. This approach valorises the available biomass, namely, modified lignin into UV-curable coating, since lignin has UV-absorbing properties, which make it suitable for coating. Therefore, this work aims to develop an efficient method using two different types of DESs, namely choline chloride: lactic acid (1:10) and choline chloride: oxalic acid (1:1), for the removal of lignin from oil palm empty fruit bunch via microwave-assisted heating. The lignin yield was calculated according to National Renewable Energy Laboratory (NREL) standards. After the pretreatment, the lignin samples were characterised using different advanced techniques such as FTIR, TGA, and DSC.

2. Materials and Methods

2.1 Materials

Raw materials used in this study are empty fruit bunch (EFB) fibres taken from LCSB Palm Oil Mill Lepar, Gambang, Kuantan, Pahang. The EFB was washed using soap to remove dirt and rinsed at least three times with tap water to ensure the soap was fully removed. The EFB was then dried in an oven at 80 °C for 24 h before being further ground using a hammer mill and a sieve with a mesh size to obtain a particle size in the 0.3-1 mm range. All chemicals such as choline chloride (ChCl), Lactic acid (LA) (≥ 85%), oxalic acid (OA) dihydrate, and ethanol (99.9%) were purchased from Sigma-Aldrich.

2.2 Methodology

2.2.1 Deep eutectic solvent preparation

In this study, deep eutectic solvents (DESs) prepared from choline chloride (ChCl) as a hydrogen bond acceptor (HBA) with oxalic acid (OA) dihydrate and lactic acid (LA) as hydrogen bond donors (HBDs) in different ratios were studied for the fractionation of lignocellulosic biomass obtained from the EFB. Each DES was prepared by mixing choline chloride with its corresponding hydrogen-bond donor in the specified ratio. Mixing was carried out at 90 °C for 24 h for choline chloride (ChCl) and oxalic acid (OA) dihydrate, while at 80 °C for 30 min to 1 h for choline chloride (ChCl) and

lactic acid (LA) until a clear liquid was formed. The DESs used in this work and the HBA-to-HBD ratios are listed in Table 1.

Table 1. Compositions and reaction conditions used for the synthesis of different deep-eutectic solvents (DESs). [HBD-hydrogen bond donor and ChCl-choline chloride]

HBD (Alpha-Hydroxy Acid, AHA)	HBA (ChCl): HBD molar ratio	Temperature, ˚C	Time, min	Designations
Monocarboxylic acids-Lactic acid (LA)	1:10	120	30	ChCl: LA
Dicarboxylic acids-Oxalic acid (OA) dihydrate	1:1	120	30	ChCl: OA

2.2.2 Lignin extraction from EFB fibre

Approximately 5g of empty fruit bunch (EFB) was mixed with 100 mL of deep eutectic solvents (ChCl:LA and ChCl:OA) at a 1:20 ratio and poured into a 250 mL round-bottom flask for microwave-assisted heating (MAH) at 120 °C for 30 min while stirring. Two products are produced after the reaction process: black liquor and cellulose-rich fibres. Both were separated by vacuum filtration. After that, 50 mL of ethanol was used to wash the cellulose-rich fibres by vacuum filtration, thereby collecting all the black liquor. Then, the ethanol in the black liquor was removed using a rotary evaporator at 80 °C. The black liquor was poured into hot deionised water at a 1:2 ratio and immediately placed in a chiller overnight. This process is caused by lignin precipitation. After that, the precipitated lignin was washed several times with deionised water until neutralised, then collected by freeze-drying. The lignin is stored in a desiccator until further use. The schematic diagram of the lignin extraction process is shown in Figure 2.

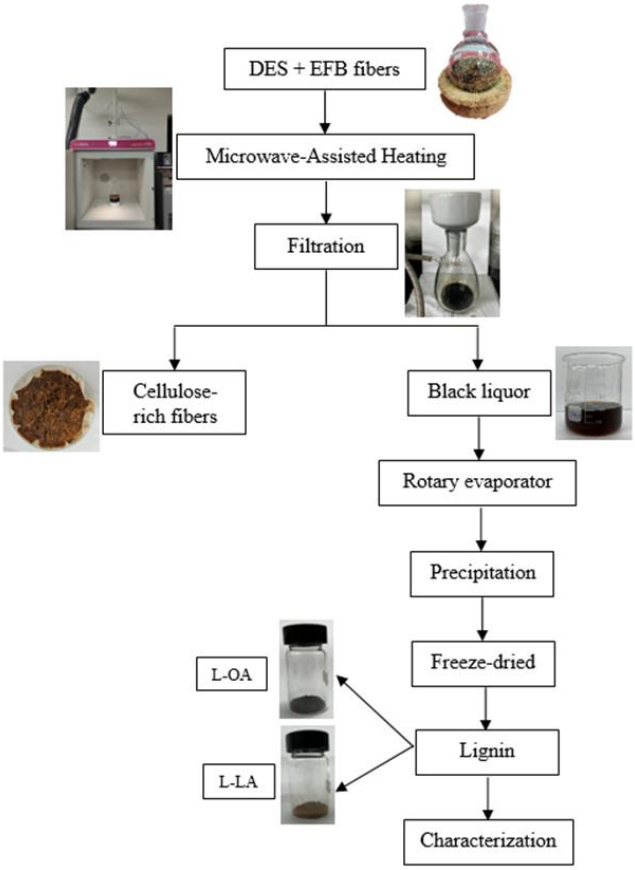

Figure 2. Schematic diagram showing various steps in the fractionation of lignin using DESs via microwave-assisted heating

2.3 Materials Characterisation

2.3.1 Percentage yield of lignin

The amount of lignin extracted using a deep eutectic solvent with different hydrogen-bond donors (HBAs) has been calculated from lignin yield to determine the percentage of crude lignin. The lignin yield was calculated based on data from the National Renewable Energy Laboratory (NREL). Thus, the lignin yield of the pretreatment has been calculated using Eq. 1:

$$L\ (\%) = \frac{m_{EXTRACTED}}{m_{NREL}} \times 100 \quad (1)$$

where L is crude lignin yield (%), mEXTRACTED (g) is the mass of lignin extracted in microwave-assisted heating (MAH) and mNREL (g) is the mass of lignin calculated using NREL method.

2.3.2 Fourier transform infrared spectroscopy

Infrared transmission measurements have been taken via an FTIR Spectrometer from Perkin Elmer in attenuated total reflectance (ATR) mode [14]. The spectra are measured over the range 700-4000 cm^{-1}. Each measurement consists of 32 scans with a resolution of 4 cm^{-1}, and the measurements will be taken at room temperature. Before starting the measurement, a background spectrum has been recorded. The received spectra have been analysed using PerkinElmer Spectrum software version 10.03.07.

2.3.3 Thermogravimetric analysis

In a typical measurement, all samples are placed into an alumina heating pan in the TGA Q500 Thermal Analyser instrument [15]. Approximately 15 mg of each sample will be weighed and ramped from 25 to 900 °C at a heating rate of 10 °C/min under a nitrogen flow rate of 40 mL/min.

2.3.4 Differential scanning calorimetry analysis

DSC analysis was performed on a NETZSCH DSC 214-Polyma under a nitrogen atmosphere with a 50 mL/min flow rate. Approximately 10 mg of each sample was weighed into an aluminium crucible and heated from 25 to 300 °C at a heating rate of 10 °C/min.

3. Results and Discussion

3.1 Percentage Yield of Lignin

It has been discovered that DESs can be synthesised at various molar ratios of HBA to HBD. DESs with choline chloride as HBAs and alpha-hydroxy acids (AHAs), namely oxalic acid dihydrate for dicarboxylic acid and lactic acid for monocarboxylic acid as HBDs, were formed at a molar ratio of 1:1 and 1:10, respectively. The presence of a hydroxyl group (OH) in AHA might be responsible for the formation of DESs at ratios 1:1 and 1:10. The OH group in AHA can form extra hydrogen bonds with HBA to stabilise the solvent [3], [16].

In comparison to dicarboxylic acid, monocarboxylic acid produced a greater lignin extraction yield in the alpha-hydroxy acid (AHA)-based DESs group. As shown in Figure 3, ChCl: LA and ChCl: OA dihydrate pretreatments yielded lignin at 76.61% and 29.36%, respectively. This result is in good agreement with an earlier study that found that, for rice straw and EFB pretreatment, the delignification effectiveness of a dicarboxylic acid was lower than that of a monocarboxylic acid [3], [17]. Although OH and COOH groups are the same functional group, the number of COOH groups in the AHA-based DES used in this investigation may account for the observed performance difference. The extra COOH groups in oxalic acid dihydrate facilitate the formation of more hydrogen bonds with HBA [3], [18]. Therefore, this observation collectively suggests that the functional groups of HBD play a major role in DES formation. The OH-containing acid can form DESs with HBA at ratios of 1:1 and 1:10. Previous researchers stated that in dicarboxylic acid DES, the two COOH groups can form extensive chains of dimers, which restricts the mobility of the solvent with higher fluidity, which can promote higher solute dissolution [16]. From a mass-transfer perspective, ChCl: OA dihydrate was visibly more viscous than ChCl: LA. The lower mobility of solvent molecules might have weakened the solvent-solute interaction with lignin, resulting in decreased lignin extraction efficiency [3], [16].

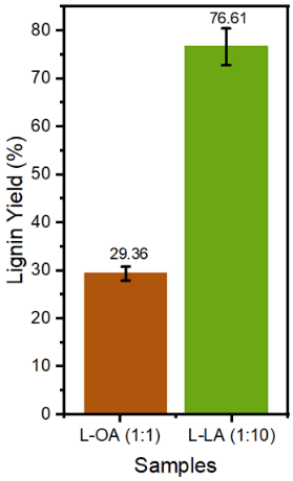

Figure 3. DESs extracted lignin yield

On the contrary, the impact of aliphatic chain length was examined using AHA-based DES with carbon chains ranging from C_3 to C_4. Figure 3 clearly shows that lignin extractability decreased as chain length increased from C_3 to C_4. The alkyl group in an acid increases the electron density at the oxygen in the OH group by donating electrons. As a result, the hydrogen link between oxygen and hydrogen becomes stronger, creating a weaker acid with a lower acid ionisation strength. Therefore, in solvent-solute interactions, DESs' capacity to donate protons will increase with a decrease in the

length of their aliphatic chain [3], [19]. Short alkyl chains, OH groups, and double bonds in carboxylic acid HBDs improved DES performance. However, the presence of multiple COOH groups weakened DES performance. This underscores the importance of selecting a suitable acid component paired with a compatible HBA to produce DESs with high lignin-production efficiency [16]. Acid strength is an essential component and a solvent-dependent factor. An acidic environment is necessary for hydrolysis, which breaks down biomass into cellulose, hemicellulose, and lignin. This affects the efficiency of DESs by contributing to delignification [5], [13]. Besides, using a microwave-assisted process markedly enhances the efficiency of DES pretreatment compared to utilising DESs alone. By directly applying an electromagnetic field to the molecular structures of heated objects, microwaves induce biomass breakdown primarily through molecular collisions driven by dielectric polarisation [5]. In addition, microwave irradiation can maximise ionic characteristics and increase the molecular polarity of DESs. Thus, microwave irradiation can significantly shorten the reaction time for DES pretreatment while achieving a similar or even higher degree of effectiveness compared to DES pretreatment alone [20].

3.2 Fourier Transform Infrared Spectroscopy

As shown in Figure 4, a broadband at 3450 and 3369 cm^{-1} was detected, representing strong intermolecular force bonding of a stretching vibration between phenolic and aliphatic hydroxyl groups, which are abundant in the extracted lignin, namely L-OA and L-LA, with the raw EFB. The strong absorption peaks at 1599, 1511, and 1418 cm^{-1} in both lignin fractions correspond to the characteristic vibrations of the benzene ring skeleton of lignin [21]. Meanwhile, a stretching vibration of the alkene group has been detected at 1649 cm^{-1} in raw EFB. Moving to the lignin fractions, the peak at 1460 cm^{-1} is attributed to C-H deformation combined with aromatic ring vibration, indicating that the separated part was indeed a lignin compound. Thus, the benzene ring skeleton was undamaged and well protected [22]. At approximately 1325 and 1214 cm^{-1} for both lignin and 1241 cm^{-1} are attributed to the aromatic C-O stretching of S units and condensed G units. The weak peak observed in the L-OA and L-LA fractions at 1272 cm^{-1} corresponds to the C-O stretching vibration of guaiacyl [23]. After the DES pretreatment, the extracted lignin contained a small amount of guaiacyl structural units (G), indicating that the lignin extracted by DES belonged to the G and S types [23]. The absorption peak of the ether bond C-O-C was observed at approximately 1164 cm^{-1}. At 1029 $cm^{-1,}$ which corresponds to the C-H bending vibration in carbohydrates, the peak of lignin extracted by DESs was weak, which indicates that the content of carbohydrates was lower and the lignin purity was significantly higher [23]. Whilst for raw EFB, the peak at 1029 cm^{-1} was quite broad, indicating the presence of primary alcohol [24].

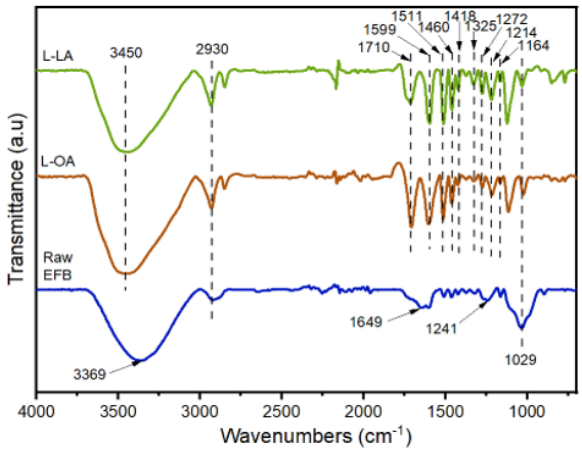

Figure 4. FTIR spectra for raw EFB, L-OA, and L-LA

3.3 Thermogravimetric Analysis and Differential Thermogravimetric

Thermogravimetric analysis (TGA) provides information on the thermal properties of lignin and helps predict its behaviour under thermochemical treatment. As a heterogeneous polymer, lignin decomposes over a wide temperature range because its functional groups exhibit varying degrees of thermal stability [16]. Figure 5(a)-(b) depicts the thermogravimetric (TG) and differential thermogravimetric (DTG) curves of the raw EFB, L-OA, and L-LA in the present study. Despite being extracted using different types of HBDs in DESs, the lignin generally exhibited a similar degradation trend, as shown in Figure 5 (a). There are three stages in the thermal degradation of raw EFB and both extracted lignins, namely L-OA and L-LA. The weight loss in the first stage for raw EFB and both extracted lignins occurs below 200 °C, which may be due to water evaporation [25]. The most significant weight loss in raw EFB occurs between 105 °C and 411 °C due to the decomposition of hemicellulose, cellulose, and other organic components [24]. For L-OA and L-LA, the second breakdown stage involves the residual lignin-carbohydrate complex (LCC) and hemicellulose at temperatures ranging from 112 °C to 600 °C. The second stage can be further divided into two processes, where the first one was the cracking of α- and β- aryl-alkyl ether bonds at about 270 °C and the second one (300-400 °C) mainly resulted from the cleavage of methyl-aryl ether and carbon-carbon bonds [25], [26], [27]. At temperatures above 800 °C, the raw EFB shows a lower residual mass compared to L-OA and L-LA. This indicates that L-OA and L-LA have higher char yields (41.73% and 34.50%, respectively), suggesting they form more thermally stable residues than raw EFB (18.68%).

The differential thermogravimetric (DTG) curves of raw EFB and both lignin samples are shown in Figure 5(b). The DTG curve for the raw EFB shows a prominent peak around 350 °C, indicating the temperature at which the rate of weight loss is highest. This peak also corresponds to the rapid decomposition of cellulose and hemicellulose components in the raw EFB. As for L-OA and L-LA, the weight loss in the 200 to 500 °C region is further divided into 2 to 3 different regions depending on the lignin samples [5]. The L-OA and L-LA samples show broader, less intense peaks than the raw EFB, with maximum weight-loss rates at 339 °C and 348 °C, respectively. This weight loss is related to the fragmentation of lignin interunit bonds [28]. However, the peaks for L-OA and L-LA are lower and less sharp due to the high thermal stability of this lignin. The results confirm that after DES pretreatment of biomass, the lignin samples exhibited different structural features and composition. On the other hand, the highest weight-loss rates for L-OA and L-LA occur at 201 °C and 187 °C, respectively. These weight losses are most likely representative of oxalic and lactic acid contamination [25], [29].

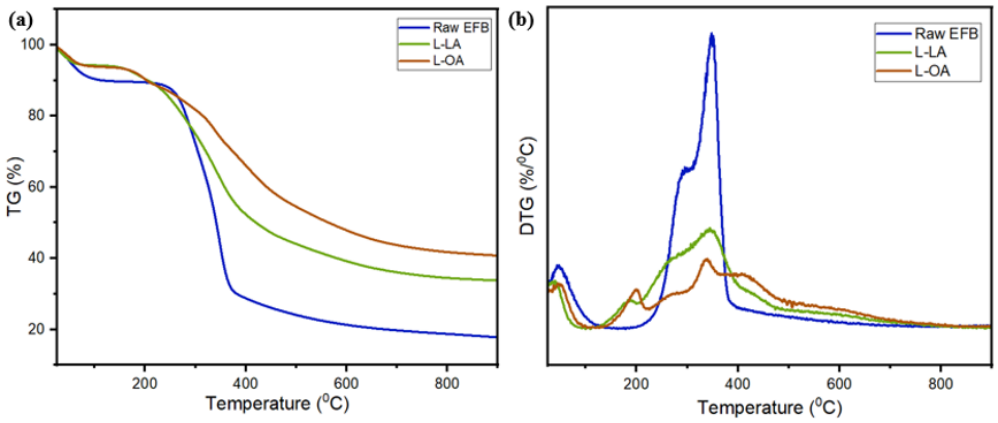

Figure 5. (a) TGA and (b) DTG analysis of raw EFB, L-OA, and L-LA

3.4 Differential Scanning Calorimetry Analysis

DSC measurements were performed to determine the glass transition temperature (T_g) of raw EFB and both extracted lignins, namely L-OA and L-LA. T_g is the temperature at which the polymer substrate transforms from a rigid glassy state into a soft substance [24]. The T_g of lignin is affected by several factors, such as molecular weight, degree of crosslinking, and thermal history, which are stored in the glassy state of the samples. Given the polyphenolic structure of lignin, various interactions can occur, including classical hydrogen bonding, hydrogen bonding with aromatics, and π-stacking between aromatic units [30]. Figure 6 shows the T_g values for the raw EFB, L-OA, and L-LA, and the obtained values are summarised in Table 2.

Table 2. T_g values obtained for raw EFB, L-OA, and L-LA by DSC analysis

Samples	T_g (˚C)
Raw EFB	85
L-OA	83
L-LA	90

Table 2 shows that the T_g values of raw EFB, L-OA, and L-LA are 85 °C, 83 °C, and 90 °C, respectively. The highest value was obtained for the L-LA samples. Since the L-OA samples had the lowest T_g, the significant amount of residual carbohydrates in their structure could contribute to the observed Tg due to increased hydrogen bonding between the polymeric chains, which could reduce their mobility [30].

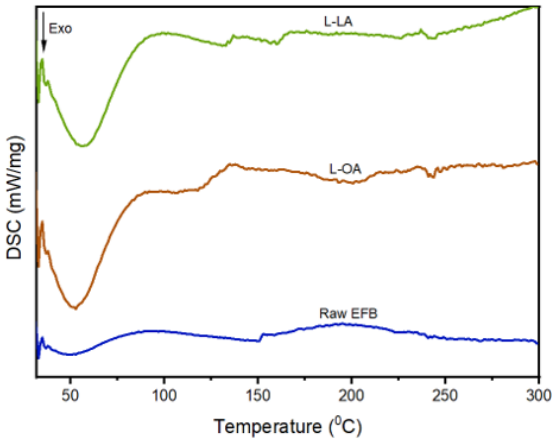

Figure 6. DSC thermogram of raw EFB, L-OA, and L-LA

4. Conclusions

The study shows that monocarboxylic acid-based DESs, such as lactic acid, are more effective in extracting lignin than dicarboxylic acid-based DESs, which are oxalic acid dihydrate. The higher lignin extraction yield from ChCl: LA (76.61%) compared to ChCl: OA dihydrate (29.36%) indicates that the type of acid in the DESs plays a crucial role in extraction efficiency. Besides, the hydroxyl group (OH) in alpha-hydroxy acids (AHAs) helps form DESs at different molar ratios by stabilising the solvent through additional hydrogen bonds with the hydrogen-bond acceptor (HBA). The presence of multiple COOH groups in oxalic acid promotes the formation of extensive hydrogen bonds, resulting in more viscous DESs that negatively impact the efficiency of lignin extraction. The study also finds that lignin extractability decreases as aliphatic chain length increases from C_3 to C_4 in AHA-based DESs. Shorter aliphatic chains enhance the DESs ability to donate protons, which is crucial for effective lignin extraction. Then, the FTIR spectra reveal specific peaks corresponding to functional groups, confirming the successful extraction of lignin. As for the TGA, it shows that lignin extracted using DESs, namely L-OA and L-LA, has higher thermal stability and char yield than raw EFB, indicating that the DES pretreatment process yields more thermally stable lignin. The DSC analysis indicates that the glass transition temperature (T_g) of lignin varies depending on the type of acid used in the DESs. L-LA exhibits a higher T_g than L-OA, which may be attributed to differences in molecular structure and residual carbohydrates that affect polymer chain mobility.

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Declaration of Competing Interests

The authors declare no conflicts of interest.

CRediT Author Contribution Statement

N.A.S. Yuharmon: Writing – original draft; Methodology; Investigation; Formal analysis; Conceptualization.
N. Salim: Investigation; Writing – review & editing.
S.N.H. Mustapha: Writing – review & editing.
I.I. Misnon: Investigation; Writing – review & editing
M.H. Ab Rahim: Validation; Formal analysis; Writing – review & editing; Supervision.
R. Roslan: Conceptualization; Investigation; Writing – review & editing; Formal analysis; Funding acquisition; Project administration; Supervision.

Availability of Data and Materials

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Ethics Statement

This research involved extraction and characterization of biomass-derived materials and did not involve human participants, animal subjects, or sensitive personal data. Therefore, ethical approval and informed consent were not required for this study.

Generative Artificial Intelligence Disclosure

Language editing only: AI-based tools were used solely for language editing and grammar refinement. No AI content generation or data interpretation was performed. All intellectual contributions are original and solely attributable to the authors.

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