

The Influence of Choline Chloride/Glycerol Molar Ratio in Deep Eutectic Solvent Pretreatment on the Physicochemical Properties of Recovered Corncob Biomass

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ABSTRACT

Deep eutectic solvents (DESs) are considered promising green solvents for lignocellulosic processing due to their ability to form effective hydrogen-bonding networks, along with low toxicity, biodegradability, and reasonable cost. In this study, choline chloride/glycerol (ChCl/Gly) DES system was combined with hydrogen peroxide for corncob biomass fractionation and recovered biomass production. The effect of ChCl/Gly molar ratios (1:1, 1:3, and 1:5) on extraction efficiency and the physicochemical properties of the recovered solid biomass were systematically evaluated. The calculated results showed that the recovered biomass yield increased with increasing glycerol content, reaching a maximum of $68.06 \pm 1.85\%$ at a ratio of 1:5. The color parameters (L^* , ΔE , and WI) indicated an improvement in whiteness, reflecting the effective removal of chromophoric compounds. FTIR analysis confirmed a significant reduction in hemicellulose and lignin, while XRD results showed that the cellulose I structure was preserved, with a crystallinity index of 39.90% at the 1:1 ratio. SEM and TGA further demonstrated that the recovered biomass exhibited higher purity and improved thermal stability compared to the raw material. These results demonstrate that the ChCl/Gly ratio directly affects the removal efficiency of amorphous components as well as the properties of the recovered biomass. The recovered biomass shows potential for applications in reinforcement materials and adsorption, while the DES-based process represents an effective and environmentally friendly approach for lignocellulosic biomass valorization.

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1. Introduction

Lignocellulosic biomass is considered one of the most sustainable raw materials to produce bio-based materials [1]. The main components of lignocellulosic biomass include cellulose, hemicellulose, and lignin, which are tightly interconnected through hydrogen bonds and complex intermolecular interactions [2]. Among these components, cellulose is the most abundant natural polymer and possesses several attractive properties such as biodegradability, renewability, high mechanical strength, and chemical stability [3]. Owing to these characteristics, cellulose has been widely applied in various fields including biodegradable materials, composites, biomedical applications, energy, and agriculture [4].

Vietnam is an agricultural country where large quantities of agricultural residues are generated after harvesting. The efficient management and utilization of these biomass resources have become an important issue to reduce environmental pressure and mitigate negative impacts on human health [5]. Among various agricultural residues, corncob is an abundant by-product generated during corn processing and is considered a promising lignocellulosic feedstock for cellulose-rich biomass production. In the lignocellulosic structure of corncob, cellulose fibers are embedded within a rigid

matrix composed of hemicellulose and lignin, which significantly hinders the direct extraction of cellulose [6]. Conventional extraction methods such as acid hydrolysis, alkaline treatment, or organosolv processes can remove non-cellulosic components [7]. However, these approaches often require harsh reaction conditions, consume large amounts of chemicals, and may cause environmental concerns [8]. Moreover, these processes may partially degrade the structure and properties of the extracted cellulose.

In recent years, deep eutectic solvents (DESs) have emerged as promising green solvents for biomass processing. DESs are formed by combining a hydrogen bond acceptor and a hydrogen bond donor to create a eutectic mixture with unique physicochemical properties [9]. Several studies have demonstrated the effectiveness of DES pretreatment for lignocellulosic biomass. Zhang et al. reported that a choline chloride-based DES system could remove up to 87.00% of lignin from corn stover, significantly improving cellulose saccharification efficiency [10]. Similarly, Ma et al. reported that DES pretreatment of wheat straw could extract about 81% of lignin while retaining most of the cellulose, resulting in an enzymatic digestibility of nearly 90% [11]. In another study demonstrated that using a choline chloride/glycerol DES to pretreat sugarcane bagasse achieved a lignin removal rate of up to 81%, highlighting the effectiveness of DES systems in lignocellulosic biomass fractionation [12].

Among various DES systems, mixtures based on choline chloride and glycerol have attracted particular attention due to their low cost, low toxicity, and high availability [13]. In the choline chloride and glycerol system, the three hydroxyl groups of glycerol form extensive hydrogen bonds with the chloride anion and with hydroxyl groups present in lignocellulosic polymers [14]. This hydrogen-bonding network can penetrate the lignocellulosic matrix and weaken the interactions among cellulose, hemicellulose, and lignin, thereby facilitating biomass fractionation and cellulose enrichment [15], [16]. In addition, oxidative bleaching using hydrogen peroxide is commonly applied to remove residual lignin and improve cellulose purity, since this oxidizing agent decomposes into water and oxygen after reaction without generating harmful chlorinated by-products [17].

Although studies on DES-assisted pretreatment of lignocellulosic biomass have increased in recent years, research focusing on the combined use of DES and hydrogen peroxide bleaching for biomass fractionation and cellulose-rich solid recovery from corncob remains limited. In particular, the influence of DES composition on the structural and morphological characteristics of the recovered solid biomass has not been fully clarified. In this study, cellulose-rich solid biomass was recovered from corncob using a deep eutectic solvent system composed of choline chloride and glycerol, followed by hydrogen peroxide bleaching. The structural and morphological properties of the recovered solid biomass were characterized by using modern analytical techniques, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA).

2. Materials and Methods

2.1. Materials

Corn cob was collected from Ho Chi Minh City, Vietnam. Hydrogen peroxide (H_2O_2 , 30%), Sodium hydroxide (NaOH , $\geq 96\%$), Choline chloride (ChCl , $\text{C}_5\text{H}_{14}\text{ClNO}$, 99%), Glycerol (Gly , $\text{C}_3\text{H}_8\text{O}$, $\geq 99\%$), were purchased from Sigma-Aldrich. All chemicals were used as received without further purification.

2.2. Methods

Corn cob raw materials were collected in Ho Chi Minh City, Vietnam. After collecting, the corncobs were naturally air-dried for 3 days and subsequently oven-dried at $70\text{ }^\circ\text{C}$ for 24 h in a convection oven to remove moisture. The dried samples were then ground into powder and sieved through a 100-mesh sieve to obtain fine corncob (CC).

The process for recovering biomass using a deep eutectic solvent (DES) combined with hydrogen peroxide treatment is illustrated in Figure 1. First, the deep eutectic solvent was prepared by dissolving choline chloride in glycerol at molar ratios of 1:1, 1:3, and 1:5, following the method reported by previous studies with slight modifications [18]. The mixture was heated at $80\text{ }^\circ\text{C}$ for 2 h under magnetic stirring until a homogeneous DES solution was obtained.

Next, the corncob powder was added to the DES solution at a solid-to-liquid ratio of 1:10 (w/v) and treated at 110 °C for 2 h to disrupt the lignocellulosic structure and partially dissolve lignin and hemicellulose. After treatment, the mixture was filtered to collect the solid fraction, which was subsequently washed several times with distilled water until the pH of the washing solution was close to neutral. The obtained solid was then dried in a convection oven at 50 °C for 24 h to obtain an intermediate product.

The intermediate solid was further treated with a 2 % hydrogen peroxide solution under alkaline conditions at a solid-to-liquid ratio of 1:10 (w/v). The pH of the system was adjusted to 11.5 using a 5 M NaOH solution. The mixture was magnetically stirred and heated at 50 °C for 4 h to enhance the oxidation process and remove the remaining lignin. Finally, the solid fraction was filtered and washed repeatedly with distilled water until a stable pH was reached and then dried at 50 °C for 24 h to recovered solid biomass. The recovered solid biomass samples were labeled as CFs-DESxy-HPK, where x and y denote the molar ratio of ChCl to Gly used in the DES preparation.

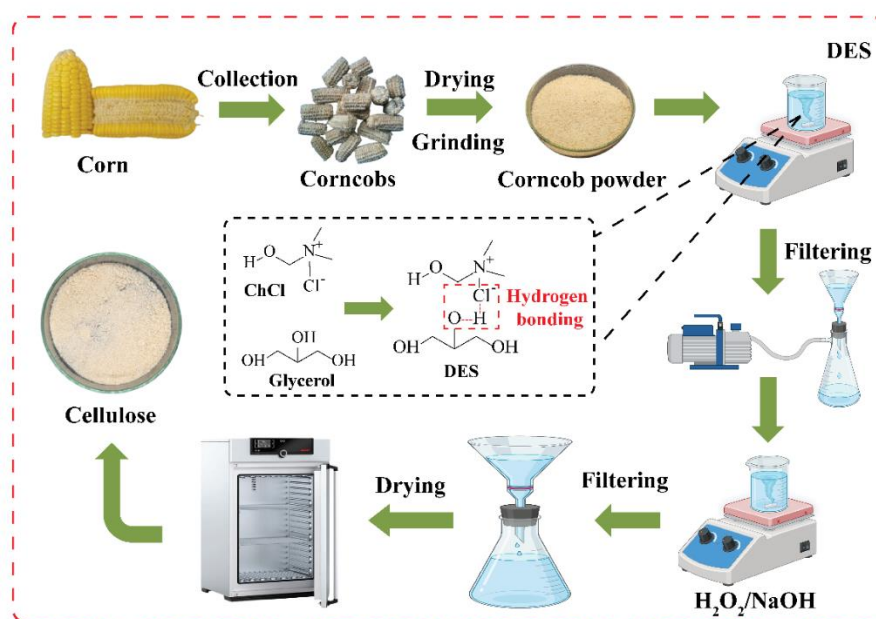


Figure 1. Schematic illustration of biomass treatment and solid recovery from corncob using ChCl/Gly-based DES combined with H₂O₂/NaOH treatment.

2.3. Characterization

2.3.1. Remaining solid biomass content (%)

The remaining solid biomass content was calculated according to the method described by Duy et al. using Equation (1) [19]:

$$Y(\%) = \frac{m_f}{m_i} \times 100 \quad (1)$$

Where m_f is the weight of recovered solid biomass (gram), and m_i is the weight of corncob (gram).

2.3.2. Color property analysis of recovered solid biomass

Color analysis of the recovered biomass was conducted to evaluate the extent of removal of non-cellulosic components, particularly lignin and naturally occurring colored compounds present in the original corncob biomass. Prior to measurement, the recovered solid biomass was evenly spread in a sample holder to obtain a relatively uniform surface.

Color measurements were performed using a CR-400 Chroma Meter colorimeter under a 10° standard observer. The color parameters were determined according to the CIELAB color system (L^* ,

a^* , and b^*), and the total color difference (ΔE) was calculated following the method described by Choque-Quispe et al, as shown in Equation (2) [20]:

$$\Delta E = \sqrt{(L^* - L_0)^2 + (a^* - a_0)^2 + (b^* - b_0)^2} \quad (2)$$

In this system, L^* represents the lightness of the sample, with values ranging from 0 (black) to 100 (white). The parameter a^* indicates the position on the red-green axis, where positive values correspond to redness and negative values correspond to greenness. The parameter b^* represents the position on the yellow-blue axis, where positive values indicate yellowness and negative values indicate blueness.

To further evaluate the whiteness of the recovered biomass powder, the whiteness index (WI) was determined based on the obtained L^* , a^* , and b^* values according to Equation (3). For each sample, measurements were taken at three different locations, and the average value was reported to improve the reliability of the results.

$$WI = 100 - \sqrt{(100 - L^*)^2 + (a^*)^2 + (b^*)^2} \quad (3)$$

2.3.3. Fourier transform infrared spectroscopy (FTIR)

The chemical structures of corncob powder and the recovered biomass were characterized using Fourier transform infrared (FTIR) spectroscopy with a NICOLET 6700 spectrometer. Spectra were recorded in the wavenumber range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} , and each spectrum was obtained by averaging 32 scans. Prior to analysis, the dried samples were ground and mixed with KBr to prepare pellets.

2.3.4. X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed to examine the crystalline structure of corncob powder and the recovered biomass using an EMPYREAN diffractometer. The diffraction patterns were collected in the 2θ range of 5° - 80° with CuK_α radiation ($\lambda = 1.54056 \text{ \AA}$) at 40 kV and 45 mA. The relative crystallinity index (CrI, %) was determined following the method proposed by Segal et al, using Equation (4) [21].

$$\text{CrI (\%)} = \frac{I_{200} - I_{\text{am}}}{I_{200}} \times 100 \quad (4)$$

Where I_{200} is the maximum intensity of the diffraction peak associated with the (200) plane of cellulose I at around $22^\circ(2\theta)$, and I_{am} is the intensity of the amorphous region at around $18^\circ(2\theta)$.

2.3.5. Field Emission Scanning Electron Microscope (FESEM)

The surface morphology of corncob powder and the recovered biomass was observed by field emission scanning electron microscopy FESEM, S-4800 at an accelerating voltage of 10 kV. Before analysis, the samples were fixed on metal stubs with carbon tape and coated with a thin platinum (Pt) layer.

2.3.6. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was performed on corncob powder and the recovered biomass using a TGA STA PT 1600 analyzer. The samples were heated from room temperature to 600 $^\circ\text{C}$ at a rate of $10^\circ\text{C}/\text{min}$ under Argon atmosphere to avoid oxidation. Temperature and mass calibrations were conducted before the measurements.

2.3.7. Statistical analysis

All experiments were conducted in triplicate, and the results are presented as mean \pm standard deviation (S_D). Data processing was performed using IBM SPSS Statistics 20. To evaluate differences among sample groups, one-way analysis of variance (ANOVA) was applied, followed by Tukey's post hoc test, with the level of statistical significance set at $p < 0.05$.

3. Results and Discussion

3.1. Remaining solid biomass content (%)

The remaining solid biomass from corncob was calculated according to Equation (1), and the results are presented in Table 1. The results indicate that the recovered biomass depends on the molar ratio of ChCl/Gly in the DES system. ChCl/Gly plays an important role in disrupting the lignocellulosic structure through hydrogen bonding interactions and promoting lignin dissolution by breaking ester bonds between lignin and hemicellulose, thereby facilitating cellulose separation and purification [22]. Among the samples, CFs-DES15-HPK exhibited the highest recovery yield ($68.06 \pm 1.85\%$), which may be attributed to the presence of more residual lignin and hemicellulose in the solid fraction, resulting in a higher remaining mass after treatment. In contrast, CFs-DES11-HPK and CFs-DES13-HPK showed lower recovery yields of $51.82 \pm 3.36\%$ and $56.60 \pm 2.17\%$, respectively, suggesting more effective removal of non-cellulosic components. One-way analysis of variance (ANOVA) followed by Tukey's test confirms that the differences among the samples are statistically significant ($p < 0.05$). Overall, the relatively high recovered biomass demonstrates the potential of the DES system as an effective pretreatment approach for converting lignocellulosic biomass from corncob into value-added cellulose-rich materials.

Table 1. Effect of the ChCl/Gly molar ratio on recovered biomass yield.

| Samples | Y (%) |
|---------------|--------------------|
| CFs-DES11-HPK | 51.82 ± 3.36^b |
| CFs-DES13-HPK | 56.60 ± 2.17^b |
| CFs-DES15-HPK | 68.06 ± 1.85^a |

3.2. Color property analysis

Table 2 presents the color parameters (L , a , b^*), color difference (ΔE), and whiteness index (WI) of cellulose samples derived from corncob at different ChCl/Gly molar ratios, while Figure 2 visually illustrates the noticeable changes in sample color. The results show that the L^* value increased from 79.78 ± 0.37 (CC) to 84.74 ± 0.26 (CFs-DES13-HPK), followed by a slight decrease to 82.28 ± 0.08 (CFs-DES15-HPK), indicating an overall improvement in the brightness of the cellulose. Meanwhile, the a^* and b^* values exhibited a decreasing trend, reflecting a reduction in red and yellow hues within the samples. Compared to the CC sample, the color difference (ΔE) and whiteness index (WI) showed significant changes after chemical treatment, with statistically significant differences ($p < 0.05$). These results suggest the effective removal of lignin and other chromophoric impurities from the corncob. Overall, the DES system enhances the color quality of cellulose, highlighting its potential for applications requiring high whiteness, such as biopolymer films and packaging materials.

Table 2. The total color difference (ΔE) and the whiteness index (WI) of recovered solid biomass samples.

| Samples | L^* | a^* | b^* | ΔE | WI |
|---------------|--------------------|-------------------|--------------------|-----------------------|--------------------|
| CC | 79.78 ± 0.37^c | 3.82 ± 0.21^a | 21.91 ± 0.28^a | 35.73 ± 0.42^a | 69.94 ± 0.45^c |
| CFs-DES11-HPK | 82.65 ± 0.28^b | 0.76 ± 0.10^c | 18.46 ± 0.57^c | 31.41 ± 0.52^c | 74.65 ± 0.44^a |
| CFs-DES13-HPK | 84.74 ± 0.26^a | 1.15 ± 0.06^c | 20.32 ± 0.23^b | 32.19 ± 0.32^{bc} | 74.57 ± 0.34^a |
| CFs-DES15-HPK | 82.28 ± 0.08^b | 2.93 ± 0.25^b | 20.15 ± 0.05^b | 33.00 ± 0.07^b | 73.01 ± 0.10^b |

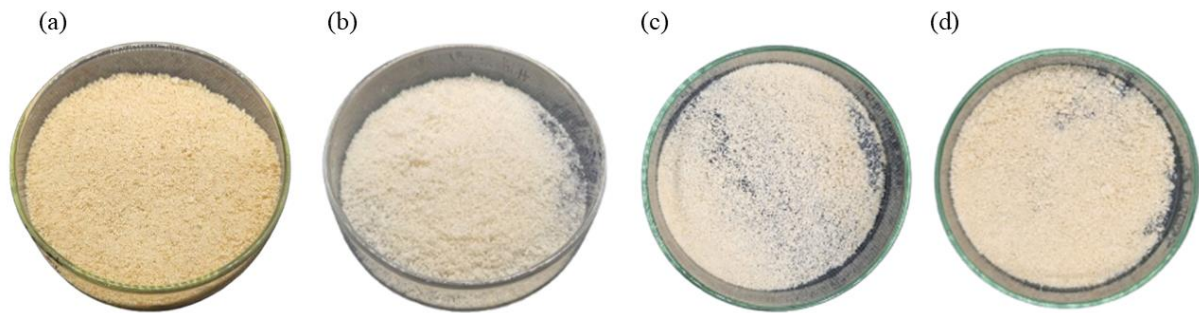


Figure 2. Photographs of (a) CC, (b) CFs-DES11-HPK, (c) CFs-DES13-HPK, and (d) CFs-DES15-HPK.

3.3. Fourier transform infrared spectroscopy (FTIR)

Figure 3a presents the FTIR spectra of the raw material (CC) and the recovered biomass after treatment with DES system combined with hydrogen peroxide under alkaline conditions at different molar ratios of ChCl/Gly. For the CC sample, the FTIR spectrum exhibits characteristic absorption bands of lignocellulosic materials. A broad absorption band around 3435 cm^{-1} is attributed to the stretching vibration of -OH groups, associated with hydrogen bonding in cellulose as well as amorphous components [23]. The peak observed at 2927 cm^{-1} corresponds to the C–H stretching vibration of aliphatic groups in polysaccharides [24]. In addition, the presence of a peak at approximately 1730 cm^{-1} is assigned to the stretching vibration of the C=O group in hemicellulose or acetyl groups [25].

After treatment with the DES-HPK system, the FTIR spectra of CFs-DES11-HPK, CFs-DES13-HPK, and CFs-DES15-HPK show a decrease in the intensity of the absorption band at 1730 cm^{-1} compared with that of the CC sample [26]. This change indicates that the treatment process partially removed hemicellulose and lignin from the biomass structure [27]. Meanwhile, the characteristic absorption bands of cellulose remain clearly identifiable after treatment. The band at around 1637 cm^{-1} is associated with the bending vibration of adsorbed water [28], whereas the peak at 1042 cm^{-1} corresponds to the C–O–C stretching of the pyranose ring. The presence of the band at 898 cm^{-1} , attributed to the β -1,4-glycosidic linkage, further confirms the preservation of the cellulose backbone [29]. These results suggest that the cellulose structure was largely preserved after pretreatment with the DES system.

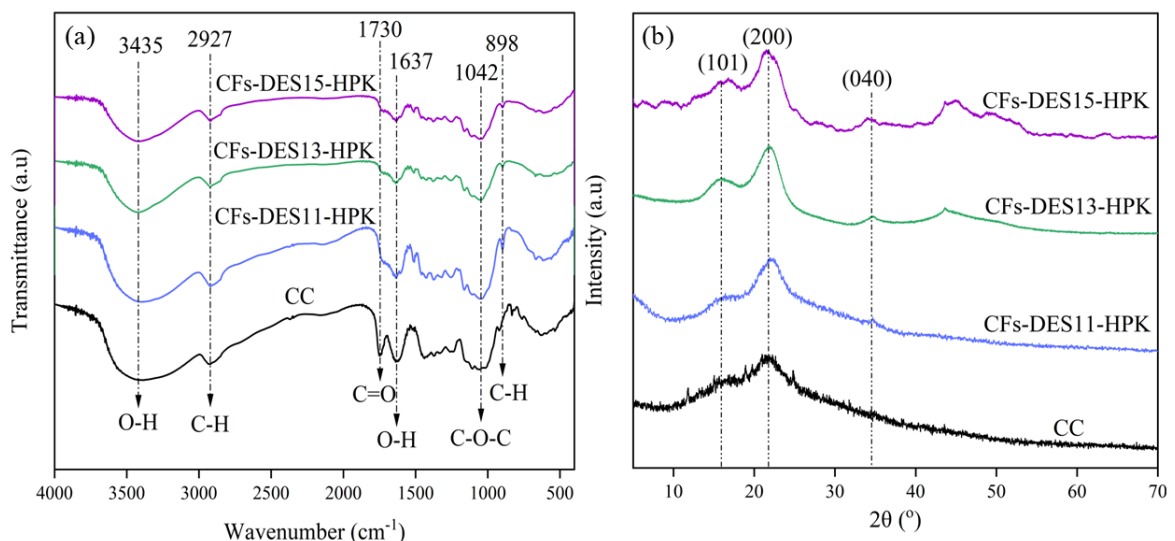


Figure 3. Structural characterization of the recovered solid biomass by (a) FTIR spectra and (b) XRD patterns after pretreatment with ChCl/Gly-based deep eutectic solvents at different molar ratios.

3.4. X-ray diffraction (XRD)

The X-ray diffraction (XRD) patterns of the recovered biomass using DES systems with different ChCl/Gly molar ratios are presented in Figure 3b. The results show that all samples (CFs-DES11-HPK,

CFs-DES13-HPK, and CFs-DES15-HPK) exhibit the characteristic diffraction peaks of cellulose I at 2 θ values of approximately 16°, 22°, and 34°, corresponding to the crystallographic planes (101), (200), and (040), respectively [30]. The crystallinity index (CrI), calculated using Equation (4), shows a slight decrease with values of 39.90%, 36.72%, and 37.00% (Table 3). This trend may be associated with the hydrogen-bonding characteristics within the DES system. Glycerol, containing multiple hydroxyl groups, can establish extensive hydrogen-bonding interactions with choline chloride as well as with the hydroxyl (–OH) groups along the cellulose chain [31]. As the glycerol content increases, the hydrogen-bonding network within the DES becomes denser, leading to higher viscosity and reduced diffusion of the solvent into the corncob structure [32]. However, compared with the raw material (CC), all treated samples still exhibit higher CrI values, indicating that the DES treatment effectively removed a significant portion of the amorphous components while largely preserving the crystalline structure of cellulose I.

Table 3. Crystallinity index (CrI) of CC and recovered biomass from corncob at different ChCl/Gly molar ratios

| Samples | CrI (%) |
|---------------|---------|
| CC | 28.50 |
| CFs-DES11-HPK | 39.90 |
| CFs-DES13-HPK | 36.72 |
| CFs-DES15-HPK | 37.00 |

3.5. Field Emission Scanning Electron Microscope (FESEM)

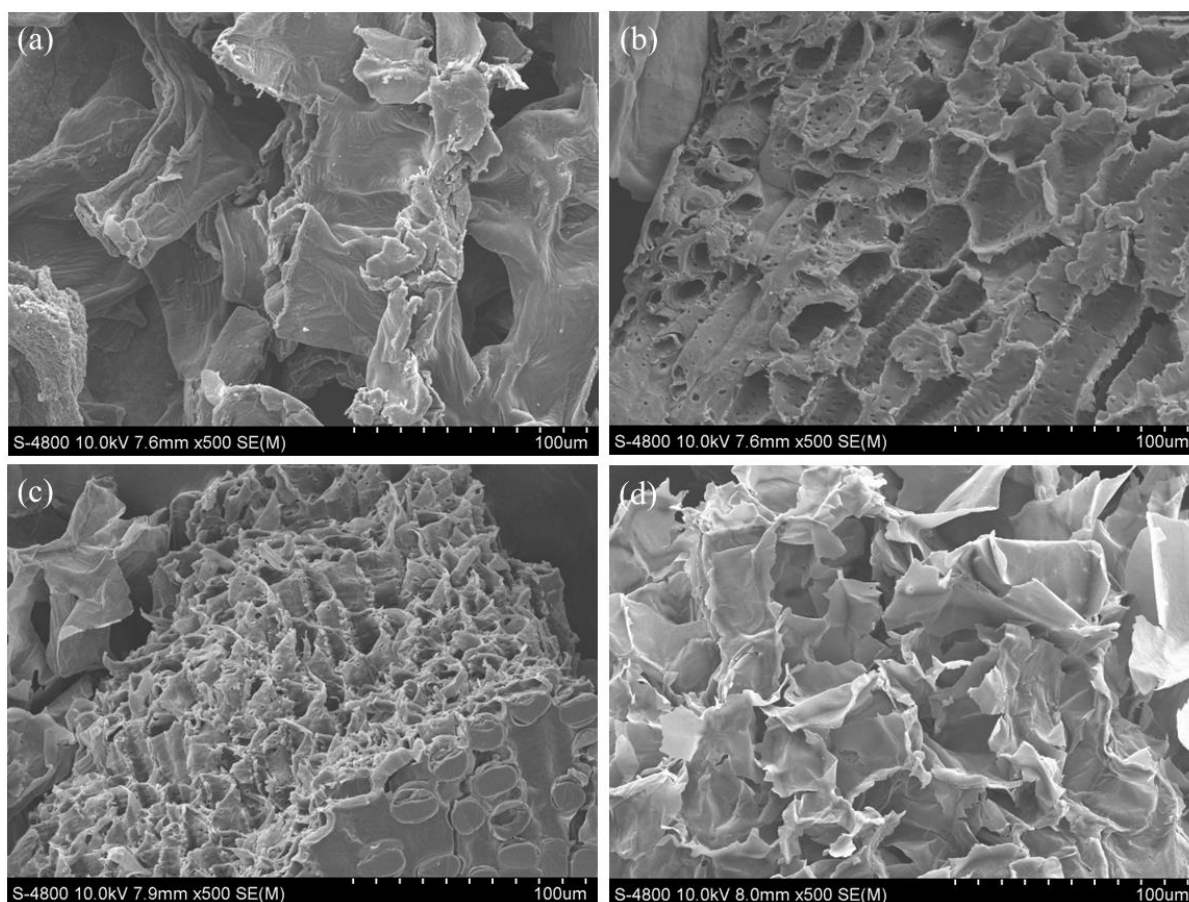


Figure 4. SEM images of recovered solid biomass from corncob at different ChCl/Gly molar ratios: (a) CC; (b) CFs-DES11-HPK; (c) CFs-DES13-HPK; (d) CFs-DES15-HPK.

Figure 4 presents the SEM images of the raw corncob (CC) and the recovered biomass obtained after treatment with deep eutectic solvent (DES) systems at different ChCl/Gly molar ratios, combined with H₂O₂ oxidation under alkaline conditions. The results reveal significant changes in surface morphology as the DES composition varies.

The corncob (CC) exhibits a relatively dense and rigid structure, in which cellulose fibers are tightly bound with lignin and hemicellulose, forming a typical lignocellulosic structure (Figure 4a) [33], [34]. After treatment with DES/H₂O₂ at a ChCl/Gly ratio of 1:1 (Figure 4b), noticeable pore formation and disruption of the lignocellulosic structure were observed. In addition, partial disintegration of the vascular bundle cell walls can be seen, suggesting the removal of amorphous components during the pretreatment process [35]. For the CFs-DES13-HPK sample (Figure 4c), the porous structures appeared less distinct and more unevenly distributed compared to those of CFs-DES11-HPK, suggesting lower pretreatment efficiency. This phenomenon may be attributed to the increased glycerol content, which enhances the hydrogen-bonding network within the DES system, thereby increasing viscosity and limiting solvent penetration into the biomass cell wall [32].

This effect becomes more pronounced in Figure 4d, where fewer pores are observed. Previous studies have reported a similar trend, where the high viscosity of DES hinders mass transfer during lignocellulosic biomass pretreatment [36]. These morphological observations, in conjunction with the FTIR and XRD results presented earlier, indicate that the ChCl/Gly molar ratio plays a significant role in lignin removal efficiency and influences the physicochemical properties of the cellulose-rich biomass.

3.6. Thermogravimetric analysis (TGA)

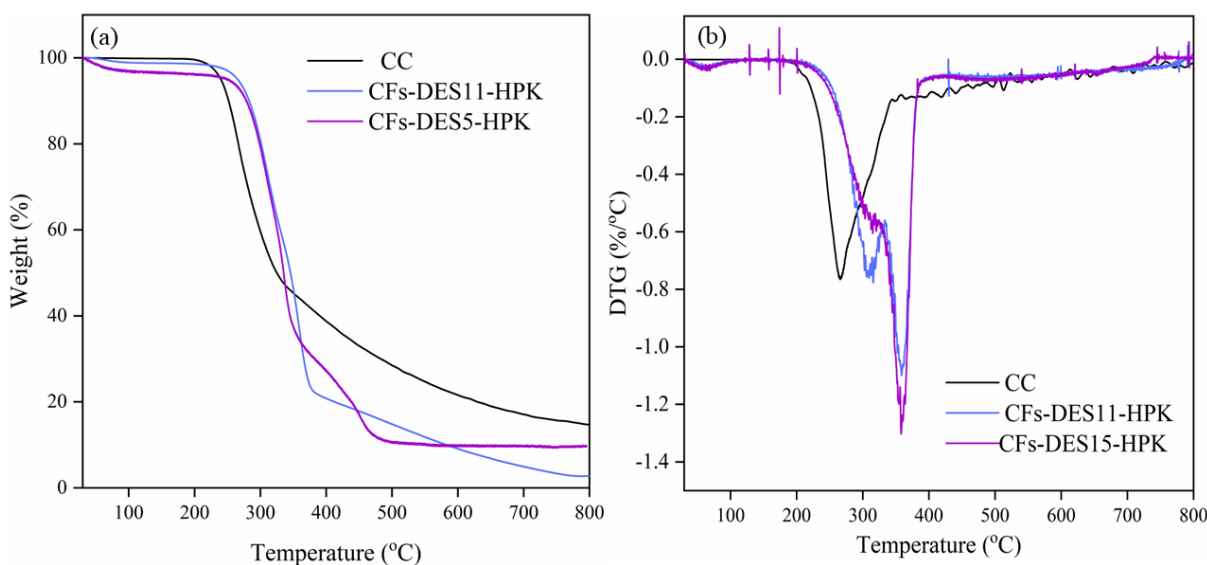


Figure 5. TGA (a) and DTG (b) curves of CC, CFs-DES11-HPK and CFs-DES15-HPK.

Figure 5 presents the TGA and DTG curves of the raw corncob sample (CC) and the recovered biomass after treatment with a deep eutectic solvent system combined with H₂O₂ oxidation (CFs-DES11-HPK and CFs-DES15-HPK). Overall, all samples exhibited the characteristic thermal degradation stages of lignocellulosic materials. In the temperature region below 120 °C, a slight weight loss is observed for both samples, which is mainly attributed to the evaporation of absorbed moisture and volatile compounds present in the biomass structure [37]. The main degradation stage occurs in the range of 215-380 °C, which is associated with the thermal decomposition of lignocellulosic components. Hemicellulose decomposes mainly between 200 and 300 °C, cellulose between 275 and 350 °C, while lignin degrades over a broader temperature range of 250-500 °C [38], [39]. This behavior is consistent with the structural characteristics of lignin, which possesses a complex aromatic polymer network that decomposes slowly over a wide temperature range.

Furthermore, the TGA curves showed that both CFs-DES11-HPK and CFs-DES15-HPK exhibited higher thermal stability than the raw CC sample, as indicated by the shift of the main degradation region toward higher temperatures. Cellulose with higher crystallinity generally exhibits greater thermal stability because the polymer chains are more tightly packed and stabilized by strong intramolecular hydrogen bonding, thus requiring higher energy to break these bonds during thermal decomposition [40], [41].

Finally, the carbonization stage occurred approximately within the temperature range of 380-600 °C, mainly due to the slow decomposition of residual lignin and char formation [42]. CFs-DES11-HPK showed a significantly lower residual mass (2.80%) compared to both CC (14.97%) and CFs-DES15-HPK (9.90%), indicating more effective removal of lignin, hemicellulose, and other non-cellulosic components during the chemical treatment process. The lower remaining char content of CFs-DES11-HPK further suggests better elimination of thermally unstable non-cellulosic constituents, resulting in a higher degree of cellulose enrichment, which is consistent with previous reports by Chae-Eun Yeo *et al.* [43].

4. Conclusions

This study shows that the use of a deep eutectic solvent (DES) based on ChCl/Gly, in combination with hydrogen peroxide, successfully enabled the recovery of biomass from corncob. The recovered biomass yield increased with increasing glycerol content and reached a maximum at a ChCl/Gly molar ratio of 1:5. Concurrently, the whiteness index improved, indicating the effective removal of non-cellulosic components. FTIR and XRD analyses confirmed that the cellulose I crystalline structure was preserved, with a crystallinity index of 39.90%. Meanwhile, SEM and TGA results revealed the disruption of fiber bundles, the formation of a porous surface morphology, and enhanced thermal stability of the recovered biomass compared to the raw biomass. These findings suggest that the DES composition plays a critical role in biomass pretreatment, influencing both the extraction efficiency and the physicochemical properties of the recovered biomass. Increasing the glycerol content in the DES system may enhance hydrogen-bonding interactions, thereby facilitating lignin removal. However, it also increases viscosity, which can hinder mass transfer and limit penetration into the biomass structure. Overall, this study proposes a green and feasible approach to producing cellulose-rich biomass materials, with promising potential for applications in reinforcement materials and adsorption systems.

Acknowledgments

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement


The data that support the findings of this study are available from the corresponding author upon reasonable request.

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