

Sustainable Valorization of Corncob Residues through Green Lignin Extraction for Functional Sunscreen Applications

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Abstract

Corncob residues represent an abundant agricultural biomass rich in cellulose, hemicellulose, and lignin. Lignin, an aromatic biopolymer containing benzene and ketone functional groups, exhibits inherent ultraviolet (UV) absorption, positioning it as a promising bio-based ingredient for sustainable sunscreen formulations. In this study, lignin was extracted from corncob residues—comprising 18.5% hemicellulose, 23.5% cellulose, and 16.5% lignin—using a green deep eutectic solvent (DES) system composed of choline chloride and citric acid, and benchmarked against conventional alkaline extraction using NaOH. FTIR analysis revealed attenuated peak intensities in DES-extracted lignin, indicating lower recovery and partial structural alteration relative to the NaOH-derived counterpart. Extraction yields were 10.06% for the DES method and 16.19% for the NaOH method. Sunscreen formulations containing 2%, 3%, and 4% lignin exhibited appreciable UV absorption, particularly within the UVA region, with the highest performance observed at 4% lignin loading. These results highlight the feasibility of valorizing corncob biomass through environmentally benign lignin extraction to produce functional bio-based materials for sustainable sunscreen applications.

Keywords: lignocellulosic biomass; corncob; deep eutectic solvent; lignin; bio-based sunscreen.

INTRODUCTION

Biomass waste, including agricultural and forestry residues, accounts for approximately 50% of global biomass waste, with an estimated production of 140 billion tons per year (Wang & Lee, 2021). Indonesia produces 146.7 million tons annually, primarily from agricultural waste (Yana et al., 2022). In the absence of effective management strategies, biomass waste can exert detrimental effects on ecosystems, pose risks to biotic communities, and contribute an estimated 1.6 gigatons of CO₂ emissions annually (VijayKumar et al., 2024). Biomass waste has been extensively utilized in various fields, including energy production, advanced materials development, and water treatment. For instance, bioethanol production from biomass waste can achieve yields of up to 65.6% (Sarao et al., 2022), and while biogas generation can reach 2.0 kWh/m³ (Náthia-Neves et al., 2018). In materials science, biomass-derived resources have been transformed into composite materials exhibiting tensile strengths of approximately 47.2 MPa (Behera et al., 2024) and as well as effective heavy-metal adsorbents for wastewater remediation

(Adegoke et al., 2022). Nevertheless, the application of biomass waste in the formulation of personal care products has received limited attention to date.

In fact, lignin, a key biomass waste organic substance alongside cellulose and hemicellulose, holds considerable potential as a natural active ingredient in sunscreen formulations. Lignin is notable for its strong resistance to ultraviolet (UV) radiation in the 280–400 nm range, attributed to its aromatic network composed of phenylpropane units and functional groups such as phenolics, ketones, and chromophores. These structural features enable lignin to effectively absorb both UVA and UVB rays, making it an excellent candidate for eco-friendly and sustainable sunscreen applications. Additionally, lignin demonstrates exceptional free radical scavenging capabilities, achieving an efficiency of up to 41.0% (Kozmelj et al., 2024), and exhibits notable antimicrobial properties against pathogens such as *Entamoeba coli*, *Staphylococcus aureus*, and *Candida albicans* (Reyes et al., 2024). As an active sunscreen ingredient, lignin offers Sun Protection Factor (SPF) values between 15 and 20, positioning it as a safer

alternative to synthetic UV filters like avobenzone and oxybenzone, which have been associated with skin irritation, allergic dermatitis, and endocrine disruption (Sander et al., 2020). Structurally, lignin is composed of phenolic monomer units such as guaiacol, syringol, and p-hydroxyphenol (Lee et al., 2019)(Widsten et al., 2020). This biopolymer features a complex aromatic framework enriched with functional groups, including methoxyl, hydroxyl, carbonyl, ketone, and additional aromatic structures (H. Zhang et al., 2019). These distinctive chemical characteristics highlight lignin's multifunctional nature, particularly its inherent ultraviolet (UV) protection capability, which makes it highly suitable for applications in cosmetic formulations.

Lignin is typically extracted using conventional solvents such as dilute acids, alkalis, or organic solvents, which pose significant environmental concerns (Faiz Norrahim et al., 2022). Deep eutectic solvents (DES) have recently gained attention as a promising green alternative to conventional solvents for lignin extraction, owing to their tunable physicochemical properties, low toxicity, and biodegradability. DES, such as choline chloride with malic or lactic acid, have achieved high lignin yields from biomass like wheat straw and sugarcane bagasse, with efficiencies reaching 75.2% (Y. Liu et al., 2017)(Chen et al., 2023). DES is formulated by combining hydrogen-bond donors (HBD) and hydrogen-bond acceptors (HBA) (Wen et al., 2013). A DES typically contains at least one HBD and one HBA that interact with each other, which helps reduce the electrostatic forces between cations and anions. As a result, DES mixtures lower the freezing point of the blend, facilitating the extraction of lignin compounds (J. Liu et al., 2020).

This research focuses on the extraction of lignin from corncob residues utilizing a deep eutectic solvent (DES) composed of choline chloride and citric acid in a 1:1 molar ratio. The extracted lignin is intended to serve as a natural active ingredient in sunscreen formulations, protection against UVA and UVB radiation. The study aims to valorize biomass waste by converting it into high-value materials for applications in the cosmetics industry.

MATERIALS AND METHODS

Materials and Instruments

Corncob waste was obtained from a local plantation in Karawang, Indonesia. Choline chloride (99%) was purchased from Himedia, while sodium hydroxide (99%) and citric acid (99.5%) were obtained from SmartLab. Ethanol (99.5%) and sulfuric acid (95–97%) were purchased from Merck. All other reagents were of analytical grade. Deionized water and a commercially available base cream without UV filters were also used in this study. The equipment used included a grinder, 100-mesh sieve, oven, mantle heater, spatula, standard

glassware, a reflux apparatus, desiccator, filter paper, analytical balance, pH meter, FTIR spectrophotometer (Jasco IR 4600 series), UV-Vis spectrophotometer (Jasco V730), Brookfield LV1 viscometer, centrifuge, and an overhead stirrer.

Preparation and Pretreatment of Corncob Residues

Corncob were dried in an oven at 105 °C for seven hours to remove residual moisture. The dried material was then ground into a fine powder and sieved through a 100-mesh screen to obtain uniform particle size. To remove impurities, the biomass powder was successively soaked in deionized water for 24 hours and 96% ethanol for an additional 24 hours. The resulting powder was subsequently characterized to determine its moisture content, lignocellulosic composition, and infrared absorption properties.

The moisture content and lignocellulosic composition of the corncob residues were determined using the oven-drying and Chesson methods, respectively (S et al., 2025). The moisture content was determined from the difference between the sample weight before and after drying. The Chesson method was carried out by first preparing 1 g of oven-dried biomass (weight a). The sample was mixed with 150 mL of distilled water and refluxed at 100 °C for 2 h, then cooled. After separating the solid residue from the extract, the residue was washed with 300 mL of hot water and dried at 105 °C for 24 h to obtain weight b. The dried residue was then treated with 150 mL of 0.5 M H₂SO₄, refluxed at 100 °C for 2 h, cooled, and subjected again to separation, washing, and drying, yielding weight c. Subsequently, 10 mL of 72% H₂SO₄ was added, and the mixture was allowed to stand at room temperature for 2–4 h. After dilution with 150 mL of 0.5 M H₂SO₄, the suspension was refluxed for another 2 h at 100 °C and cooled. The solid was separated, washed, and dried to obtain weight d. Finally, the residue was combusted in a furnace at 650 °C for 5 h to obtain the ash weight (weight e). The lignocellulosic fractions were calculated using the following equations.

$$\text{Hemicellulose(\%)} = \frac{\text{weight}_{(b)} - \text{weight}_{(c)}}{\text{weight}_{(a)}} \times 100\%$$

$$\text{Cellulose(\%)} = \frac{\text{weight}_{(c)} - \text{weight}_{(d)}}{\text{weight}_{(a)}} \times 100\%$$

$$\text{Lignin (\%)} = \frac{\text{weight}_{(d)} - \text{weight}_{(e)}}{\text{weight}_{(a)}} \times 100\%$$

Where:

- a = Oven-dry weight of the initial biomass sample.
- b = Oven-dry weight of the residue obtained after refluxing the sample with hot water.
- c = Oven-dry weight of the residue obtained after refluxing with 0.5 M sulfuric acid.

- d = Oven-dry weight of the residue obtained after maceration with 72% sulfuric acid followed by dilution to 0.5 M.
- e = Oven-dry weight of the final residue (ash) remaining after combustion.

Preparation of DES

The deep eutectic solvent (DES) was prepared by mixing choline chloride (ChCl) and citric acid (CA) in a 1:1 molar ratio. The mixture was stirred continuously on a hotplate at 90 °C until a clear and homogeneous solution was obtained. To improve solubility, deionized water (20% w/w) was subsequently added to the mixture, followed by thorough agitation to ensure complete blending. After preparation, the physicochemical properties of the DES, including pH and viscosity were measured.

Lignin Extraction from Corncob

Lignin from corncob was extracted using DES via a reflux-based extraction method. The extraction process involved a biomass concentration of 5% w/w at a temperature of 100 °C for a duration of six hours. After the extraction, the mixture was filtered to separate the liquid phase from the solid residue. The solid residue was then sequentially rinsed with 150 mL of 96% ethanol and 300 mL of deionized water to remove any residual solvents and contaminants. The resulting filtrate was allowed to sit at room temperature overnight to facilitate the precipitation of lignin. Once the lignin precipitate formed, it was centrifuged at 2000 rpm for 15 minutes to separate it from the liquid phase effectively. The lignin precipitate was then collected and washed with 100 mL of hot deionized water to eliminate any remaining solvents. After washing, it was dried in an oven. Finally, the purified lignin was analyzed using infrared spectroscopy to identify its chemical functional groups.

A control sample of lignin was extracted from corncob using sodium hydroxide (NaOH) as a solvent.

The extraction was performed using a 5% NaOH solution at a weight-to-weight ratio of 1:13, employing the reflux method for a duration of 3 hours. After the extraction, the mixture was filtered to separate the liquid phase from the solid residue. To eliminate any residual solvents, the solid residue was washed sequentially with 150 mL of 96% ethanol, followed by 150 mL of deionized water. The filtrate was then acidified with 20% nitric acid (HNO₃) until the pH reached one and was allowed to sit overnight to precipitate the lignin. The precipitate was centrifuged at 2000 rpm for 15 minutes for further separation, then filtered and rinsed with hot deionized water.

Sunscreen Cream Formulation

Lignin was incorporated into a non-UV commercial cream base at concentrations of 2%, 3%, and 4% (w/w). The lignin utilized for this purpose was obtained from both deep eutectic solvent (DES) and sodium hydroxide (NaOH) extraction methods. To ensure uniform dispersion of lignin within the cream matrix, the formulations were homogenized for two hours using an overhead stirrer operating at 1000 rpm. Following homogenization, the cream samples were evaluated for their ultraviolet (UV) absorption capacity and pH values.

RESULTS AND DISCUSSION

Characteristics of Corncob

The corncob biomass contained, on average (n = 2), 18.5% hemicellulose, 23.5% cellulose, and 16.5% lignin, indicating that its substantial lignin fraction could be utilized in the development of lignin-based sunscreen formulations. The moisture content of the corncob samples was measured at 6.42%. The relatively high lignin content highlights the potential of corncob biomass as a sustainable natural ingredient for personal care applications.

Table 1. Water and lignocellulose content of corncob.

Data	Water (%)	Hemicellulose (%)	Cellulose (%)	Lignin (%)
1	6.20	19.00	25.00	17.00
2	6.64	18.00	22.00	16.00
Average	6.42	18.50	23.50	16.50

The chemical structure of corncob biomass was investigated using IR spectroscopy to determine changes before and after the washing process. The IR spectra before and after washing displayed several notable changes (Figure 1). A broad peak around 3311 cm⁻¹, representing –OH groups (Tengku Yasim-Anuar et al., 2022), exhibited a reduction in intensity, indicating the partial removal of hydroxyl groups. The peak at 2922 cm⁻¹, corresponding to aliphatic C–H stretching, remained unchanged after washing. Meanwhile, the peak

at 1727 cm⁻¹, associated with C=O groups, showed a decrease in intensity. Peaks at 1630 cm⁻¹ and 1513 cm⁻¹, attributed to C=C stretching in lignin, were still observed. These changes suggest removing ethanol-soluble components, such as extractives and hemicellulose (Elliott et al., 2015) while maintaining the integrity of the lignocellulosic framework.

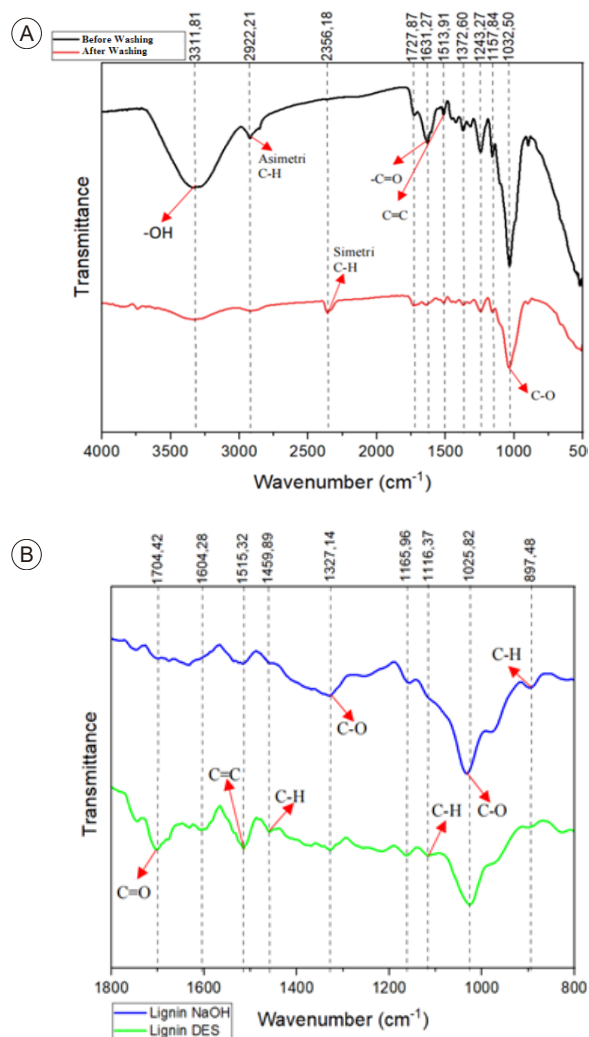


Figure 1. IR spectra of corncob biomass before and after washing (a), and lignin extracted using deep eutectic solvent (DES) and NaOH (b).

Lignin from Corncob Biomass

Multiple lignin extraction was conducted in this study, each utilizing regenerated DES. The prepared DES exhibited a viscosity of 118 cP at 50 rpm and maintained a pH within the range of 0–1. During the reflux process, the solution's color gradually changed from light brown to deep dark brown, indicating the progressive release of lignin. The initial light brown coloration represents the first interaction between the corncob biomass and the DES. After six hours of heating at 100 °C, the solution turned a deep dark brown, confirming the substantial dissolution and separation of lignin from the corncob structure.

Table 2. Viscosity and pH of the DES solution used.

DES	Viscosity (cP)	pH
1	111.6	0.96
2	117.6	0.46
3	124.8	0.70
Average	118.0	0.71

Lignin extracted with DES solvent yielded 3.36 g from six extractions of 33.40 g of corncob. Meanwhile, using NaOH as a control, lignin extraction yielded 9.85 grams of lignin from six extractions of 60.85 g of corncob. As a result, DES extraction yields 10.06% lignin, whereas NaOH extraction yields 16.19% lignin. The lignin extracted using DES exhibited a darker coloration than that obtained with NaOH, suggesting possible differences in the chemical structure or degree of condensation of the extracted lignin (**Figure 2**).



Figure 2. Visual appearance of corncob biomass and DES mixture before and after extraction (a), and color comparison of lignin extracted using DES and NaOH (b).

The IR spectra in Figure 1 shows differences between lignin extracted with DES and NaOH. The peak at 1704 cm^{-1} indicates C=O stretching vibrations, with higher intensity for DES lignin. Peaks at 1604 cm^{-1} and 1515 cm^{-1} represent aromatic ring vibrations, while the 1515 cm^{-1} peak specifically indicates guaiacyl units. The peak at 1459 cm^{-1} corresponds to asymmetric C–H deformation in $-\text{CH}_3$ and $-\text{CH}_2$ groups, and the 1327 cm^{-1} peak reflects contributions from syringyl and guaiacyl units. Peaks at 1165 cm^{-1} and 1116 cm^{-1} are associated with C–H aromatic deformations in guaiacyl units (Kline et al., 2010).

DES lignin exhibits lower peak intensities than NaOH lignin, indicating differences in extraction efficiency and potential structural modifications. The peak at 1025 cm^{-1} , related to C–H and C–O deformations, is sharper for NaOH lignin, while the 897 cm^{-1} peak, indicating aromatic C–H deformation, also shows variations. These results suggest that the extraction method significantly influences the chemical structure of the extracted lignin, with DES extraction leading to possible structural modifications.

Characteristics of Corn cob Lignin-based Sunscreens

Lignin extracted using DES and NaOH solvents was added to the non-UV commercial cream base at concentrations of 2%, 3%, and 4% w/w, resulting in a brown cream with increasing brownish intensity as the concentration increased. The pH of the lignin-based sunscreen has been found to be 6, which was consistent with both lignin extracted using DES and NaOH solvents. Achieving an appropriate pH demonstrates that lignin, whether extracted using DES or NaOH, has no substantial effect on the cream's pH above the permitted range of 4.5 to 8, as stated by the Indonesian National Standard (SNI) 16-4399-1996 for sunscreen formulation.

The UV-Vis spectra of sunscreen formulations containing DES- and NaOH-extracted lignin reveal clear differences in their UV absorption behaviours, reflecting the distinct chemistries of the two lignin fractions. Formulations containing NaOH-derived lignin exhibit broader absorption intensities across the UV-C, UV-B, and especially the UVA region, indicating the presence of a richer population of aromatic and conjugated chromophores that strongly absorb UV radiation. In contrast, the DES-extracted lignin exhibits a comparable absorbance profile but with a narrower and more sharply defined peak in the upper UVA region. This behavior is consistent with the previously observed attenuated FTIR signals and the lower extraction yield obtained for this fraction. These trends suggest that alkaline extraction preserves or exposes a greater number of UV-active functional groups—such as free phenolic hydroxyls and conjugated carbonyl structures—while the DES system, although more sustainable, may induce partial structural modification or selectively extract less chromophoric components. Increasing lignin concentrations from 2% to 4% enhances UVA absorption for both extraction methods, with the strongest performance observed at 4%, demonstrating that lignin can function as an effective natural UV-absorbing ingredient. The characteristic plateau-like absorption profile in NaOH-lignin formulations further indicates good dispersion and broad-band absorption, whereas the sharper response seen in DES-lignin creams may be influenced by narrower chromophore distribution or differences in particle aggregation within the matrix. Overall, the results highlight that lignin, particularly from NaOH extraction, has strong potential for application in bio-based sunscreen formulations. However, further work involving structural characterization, photostability assessment, and full SPF/UVA-PF evaluation is necessary to validate its performance and ensure suitability for practical cosmetic applications.

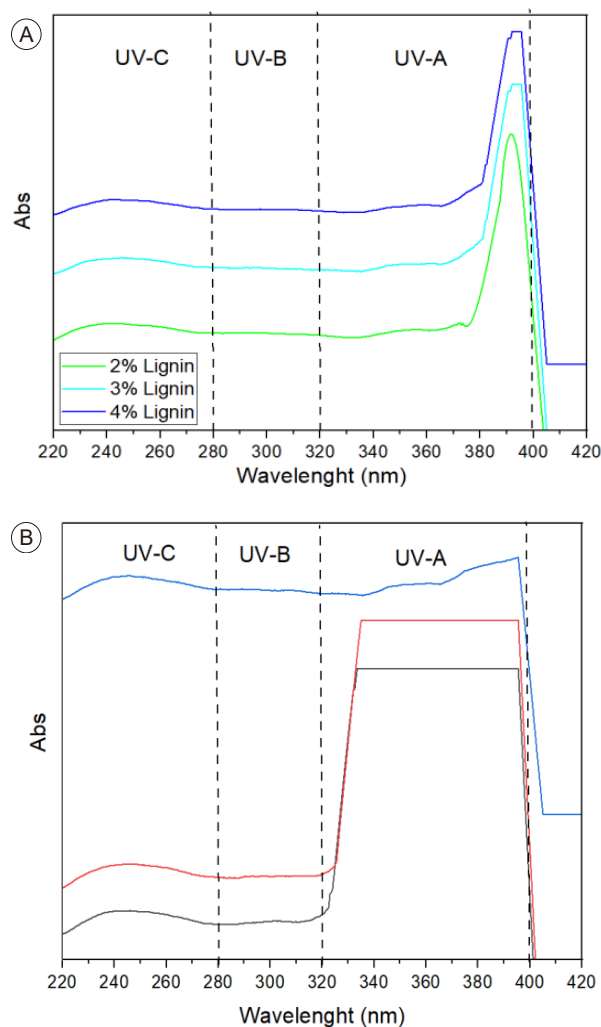


Figure 3. UV-Visible absorption spectra of sunscreen formulations containing varying concentrations of lignin extracted using (a) deep eutectic solvent (DES) and (b) sodium hydroxide (NaOH).

CONCLUSIONS

Lignin was successfully extracted from corn cob biomass using a green DES system (choline chloride–citric acid), yielding 10.06% compared to 16.19% from NaOH extraction. Structural analyses indicated that DES-derived lignin underwent partial modification, whereas the NaOH fraction retained a higher density of aromatic and conjugated chromophores. UV-Vis evaluation of sunscreen formulations further showed that NaOH-extracted lignin provided stronger and broader UV absorption across the UVC–UVA regions, while DES lignin exhibited narrower and lower-intensity absorption, consistent with its attenuated FTIR features and lower yield. Both lignin types met the SNI 16-4399-1996 pH standard and demonstrated enhanced UVA absorption at higher loadings, with optimal performance at 4%.

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