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SUSTAINABLE LIGNIN EXTRACTED FROM BANANA STEM BIOMASS AS A FUNCTIONAL UV-BLOCKING INGREDIENT IN SUNSCREEN

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Abstract

Banana stem biomass, an abundant agricultural by-product, contains significant lignin content and offers strong potential for valorization through green extraction technologies. In this study, lignin was extracted using an environmentally friendly Deep Eutectic Solvent (DES) composed of choline chloride and citric acid and subsequently incorporated into sunscreen cream formulations at concentrations of 2%, 3%, and 4%. The photoprotective performance of the formulations was evaluated through UV–Vis spectrophotometry, while pH measurements ensured compliance with SNI 16-4399-1996. FTIR analysis was conducted to compare the structural characteristics of DES-extracted lignin with those obtained using conventional NaOH extraction. The results revealed that all lignin-based formulations effectively absorbed UVA (320–400 nm) and UVB (280–320 nm) radiation, with UV absorbance increasing alongside lignin concentration; the 4% formulation showed the strongest UV-blocking capability. All samples maintained a skin-compatible pH of approximately 6. FTIR spectra further indicated that DES-extracted lignin preserved aromatic structures more effectively than NaOH-extracted lignin. Overall, the findings highlight the potential of banana stem-derived lignin as a natural UV-blocking and antioxidant ingredient for eco-friendly sunscreen products, supporting the advancement of sustainable cosmetic innovation.

Keywords: Lignin-based sunscreen, Banana stem biomass, Deep Eutectic Solvent (DES), UV protection, Sustainable cosmetics.

Abstrak

Biomassa batang pisang, sebagai produk samping pertanian yang melimpah, mengandung lignin dalam jumlah signifikan dan memiliki potensi besar untuk dimanfaatkan melalui teknologi ekstraksi hijau. Dalam penelitian ini, lignin diekstraksi menggunakan Deep Eutectic Solvent (DES) ramah lingkungan yang tersusun atas kolin klorida dan asam sitrat, kemudian diinkorporasikan ke dalam formulasi krim tabir surya pada konsentrasi 2%, 3%, dan 4%. Kinerja fotoprotektif formulasi dievaluasi menggunakan spektrofotometri UV–Vis, sementara pengukuran pH dilakukan untuk memastikan kesesuaian dengan SNI 16-4399-1996. Analisis FTIR digunakan untuk membandingkan karakteristik struktural lignin hasil ekstraksi DES dengan lignin yang diekstraksi menggunakan NaOH konvensional. Hasil penelitian menunjukkan bahwa seluruh formulasi berbasis lignin mampu menyerap radiasi UVA (320–400 nm) dan UVB (280–320 nm), dengan nilai absorbansi UV meningkat seiring meningkatnya konsentrasi lignin; formulasi 4% menunjukkan kemampuan

pemblokiran UV yang paling kuat. Nilai pH seluruh sampel berada pada kisaran pH 6, yang sesuai dengan rentang pH aman untuk kulit. Spektrum FTIR juga mengindikasikan bahwa lignin hasil ekstraksi DES lebih mampu mempertahankan struktur aromatik dibandingkan lignin hasil ekstraksi NaOH. Secara keseluruhan, temuan ini menegaskan potensi lignin dari batang pisang sebagai bahan alami penyerap UV dan antioksidan untuk produk tabir surya ramah lingkungan, sehingga mendukung pengembangan inovasi kosmetik berkelanjutan.

Keywords: Tabir surya berbasis lignin, Biomassa batang pisang, Deep Eutectic Solvent (DES), Perlindungan UV, Kosmetik berkelanjutan.

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1. Introduction

Indonesia possesses extensive agricultural land and remarkable biodiversity, with banana being one of the most widely cultivated crops. Indonesia contributes nearly 50% of Asia's banana production, reaching 9,245,247 tons in 2022 and 9,747,850 tons in 2023. Both production volume and cultivation area continue to increase annually (Rokhim et al., 2022; BPS, 2024). During harvest, only the fruit and leaves are utilized, while the stem is cut down and discarded as biomass waste. Globally, agricultural and forestry processing sectors generate approximately 50% of total available biomass waste (Wang & Lee, 2021). Indonesia alone produces an estimated 146.7 million tons of biomass waste per year, primarily sourced from agricultural residues (Yana et al., 2022). If unmanaged, such waste can lead to various environmental issues including land and aquatic ecosystem degradation, threats to organisms, greenhouse gas emissions, and air pollution from open burning (Muliari et al., 2019). Despite its abundance, the utilization of biomass waste remains limited, often restricted to handicrafts or artisanal purposes, although its potential is far greater. Biomass waste—commonly referred to as lignocellulosic waste—contains organic compounds such as cellulose $(C_6H_{10}O_5)_n$ generally ranges from 40-50%, while hemicellulose $(C_5H_{10}O_5)_m$ ranges from 20-30%, and lignin $(C_{10}H_{11}O_{3.5})$ ranges from 10-25%, together with small amounts of pectin, proteins, and other minor constituents(Bhatia et al., 2020; Usmani et al., 2020). However, its valorization has yet to receive significant attention from researchers.

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Lignin is an aromatic biopolymer rich in benzene and ketone functional groups, which naturally confer resistance against ultraviolet (UV) radiation. These intrinsic properties make lignin a promising natural active ingredient for sunscreen formulations. Lignin-based sunscreens offer a safer alternative for both the environment and consumers compared to many conventional UV-filter chemicals such as avobenzone, oxybenzone, homosalate, and octinoxate. Previous studies have indicated that lignin and lignin-derived products generally exhibit low cytotoxicity (Lee et al., 2019). In contrast, avobenzone has been associated with allergic dermatitis and eye irritation in individuals with sensitive skin. Moreover, chemicals such as oxybenzone, homosalate, and octinoxate can undergo metabolic transformation into stable intermediates that persist in biological systems, raising concerns about toxicity and endocrine disruption through enterohepatic recirculation (Sander et al., 2020).

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Conventionally, lignin extraction from biomass relies on synthetic solvents, including dilute acids, alkaline solutions, and various organic solvents. These solvents are often corrosive, volatile, toxic, and pose environmental risks when discharged without proper treatment (Faiz Norrrahim et al., 2022). The use of hazardous solvents can be mitigated by adopting environmentally friendly alternatives such as Deep Eutectic Solvents (DES). DES are widely recognized as green solvents due to their simple preparation, low toxicity, affordability, biodegradability, and good biocompatibility (Li et al., 2021). A DES system is typically composed of a Hydrogen-Bond Acceptor (HBA) and a Hydrogen-Bond Donor (HBD). Among various combinations, choline chloride–citric acid (ChCl:CA) DES has been reported to selectively solubilize lignin over hemicellulose(Li et al., 2021). The delignification process may involve the cleavage of lignin-carbohydrate ester bonds, as represented by the following reaction:

$$Lignin - OC - CO - Hemicellulose + H^{+} \rightarrow Lignin - OH + Hemicellulose - COOH \quad (1)$$

Previous studies demonstrated successful lignin extraction from rice straw, corncob, and sugarcane bagasse using DES, achieving yields of 75.9%, 98.5%, and 50.6%, respectively (Chen et al., 2020). These results highlight the potential of DES as an efficient and sustainable solvent system for lignin extraction.

In this study, lignin was extracted from banana stem biomass waste using a green-extraction approach with DES under mild processing conditions. The extracted lignin was then incorporated as a natural UV-blocking agent in sunscreen cream formulations, followed by characterization of its structural properties and evaluation of its UV-blocking performance. This research aims to develop lignin-based sunscreen formulations derived from biomass waste with effective UVA and UVB absorption and pH values compliant with the Indonesian National Standard (SNI 16-4399-1996), ensuring their suitability for typical cosmetic applications. Ultimately, this work seeks to contribute to biomass waste management by transforming agricultural residues into high-value cosmetic ingredients with economic and environmental benefits.

2.Methodology

2.1. Materials and Instruments

Banana stem biomass waste, choline chloride (Himedia), citric acid and sodium hydroxide (Smartlab), sulfuric acid and ethanol (Merck) were used as the primary materials in this study. All chemical reagents were of analytical grade. Deionized water, demineralized water, and Non-UV Moisturizer Soft Cream were also employed during the experimental work. The equipment utilized included a grinder, 100-mesh sieve, oven, mantle heater, spatula, standard laboratory glassware, a reflux apparatus, desiccator, filter paper, analytical balance, pH meter, FTIR spectrometer, UV–Vis spectrophotometer, viscometer, centrifuge, and an overhead stirrer.

2.2. Pretreatment of Banana Stem Waste

The pretreatment process began by drying the banana stem biomass in an oven at 105 °C for 7 h to remove residual moisture. The dried material was then ground and sieved through a 100-mesh screen to obtain uniform particle size. A 1 g portion of the powder was collected for FTIR analysis, and 2 g were used to determine moisture content and initial lignocellulosic composition. The remaining powder was soaked in deionized water for 24 h, followed by immersion in 96% ethanol for an additional 24 h to remove soluble organic impurities. Afterward, the material was dried again to ensure the complete removal of residual solvents.

The lignocellulosic composition, including lignin, cellulose, and hemicellulose, was determined following the Chesson methods (G S et al., 2025). First, 1 g of oven-dried biomass (weight a) was placed in a 250 mL Erlenmeyer flask and mixed with 150 mL of distilled water, followed by refluxing at 100 °C for 2 h. The mixture was then cooled, filtered and washed with 300 mL of hot water, and dried in an oven at 105 °C for 24 h to obtain weight b. The residue was returned to the same flask, mixed with 150 mL of 0.5 M H_2SO_4, and refluxed again at 100 °C for 2 h. After cooling, the filtration–washing–drying procedure was repeated to obtain weight c. Next, the residue was transferred to a new flask and treated with 10 mL of 72% H_2SO_4 at room temperature for 2–4 h. Subsequently, 150 mL of 0.5 M H_2SO_4 was added, and the mixture was refluxed for another 2 h at 100 °C. After cooling, the residue was filtered, washed, and dried to obtain weight d. Finally, the dried residue was combusted in a furnace at 650 °C for 5 h to obtain the ash weight (weight e). A schematic flowchart of the lignocellulosic composition analysis using the Chesson method is shown in Figure 1.

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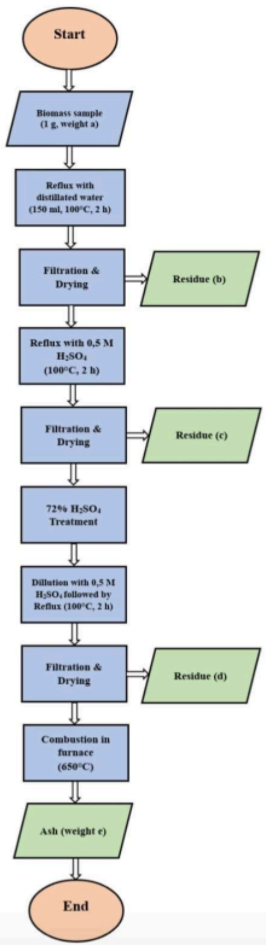

Figure 1. Flow diagram of lignocellulosic composition analysis using the Chesson method.

The lignocellulosic fractions were calculated using the following equations:

$$Hemicellulose\ content(\%) = \frac{weight\ b - weight\ b}{weight\ a} \times 100\% \quad (2)$$

$$Cellulose\ content(\%) = \frac{weight\ c - weight\ d}{weight\ a} \times 100\% \quad (3)$$

$$Lignin\ content(\%) = \frac{weight\ d - weight\ e}{weight\ a} \times 100\% \quad (4)$$

Where:
a = initial dry biomass weight
b = hot-water–insoluble residue
c = dilute-acid–insoluble residue (after hemicellulose removal)
d = lignin + ash residue
e = ash residue after furnace combustion

2.3. Pretreatment of the DES Solvent

The DES was prepared by mixing choline chloride (ChCl) as the hydrogen bond acceptor (HBA) and citric acid (CA) as the hydrogen bond donor (HBD) at a 1:1 molar ratio. In this preparation, 41.886 g of ChCl and 57.637 g of CA were used. The mixture was heated to 90 °C on a hotplate while continuously stirred to ensure complete homogenization. During heating, deionized water corresponding to 20% of the total mass was added to reduce the high viscosity of the DES, facilitating mixing and handling. Once the mixture became fully homogeneous and the DES had formed, its viscosity was measured using a Brookfield viscometer (Ametek DV1MLVTJ0). The pH of the resulting DES was also analyzed to determine its acidity or basicity.

2.4. Lignin Extraction

Lignin extraction from the banana stem biomass using DES was carried out under reflux at 120 °C for 6 h. After the extraction process, the mixture was filtered using filter paper to separate the filtrate from the solid residue. The residue was sequentially washed with 150 mL of 96% ethanol and 300 mL of deionized water to ensure that all soluble compounds, including lignin, were transferred into the filtrate. The filtrate was then allowed to stand overnight to promote lignin precipitation. Once the lignin had settled, the suspension was centrifuged at 2000 rpm for 15 min to isolate the precipitate. The collected lignin precipitate was filtered and washed with 100 mL of hot deionized water to remove any remaining solvent. Finally, the lignin was dried in an oven to obtain purified lignin for further use.

A conventional extraction process using NaOH was also performed as a comparison. In this method, banana stem biomass was treated with a NaOH solution prepared by dissolving 5 g of NaOH in 100 mL of water (solid-to-liquid ratio of 1:10, w/v) and refluxed for 3 h. After extraction, the mixture was filtered to separate the filtrate from the solid residue. The residue was then washed sequentially with 150 mL of 96% ethanol and 150 mL of deionized water to ensure complete removal of soluble constituents. The resulting filtrate was acidified with 20% HNO_3 until reaching pH 1 and allowed to stand overnight to facilitate lignin precipitation. The precipitated lignin was recovered by centrifugation at 2000 rpm for 15 min, followed by filtration and washing with hot deionized water. The purified lignin was then dried in an oven and stored for subsequent analyses.

2.5. Characterization of Extracted Lignin

The extracted lignin was characterized using IR spectroscopy to identify the functional groups present in lignin obtained from both the DES-based and NaOH-based extraction methods. Approximately 1 g of lignin from each extraction route was analyzed using an FTIR instrument. The analysis was performed to observe the resulting infrared spectra and to determine the presence of characteristic functional groups, including hydroxyl, methoxyl, and carbonyl groups.

FTIR measurements were conducted within the standard wavenumber range of 4000–400 cm^{-1} to obtain a comprehensive profile of the chemical structure of lignin derived from the two extraction methods. The characterization results provide insight into structural differences between the lignin samples produced through each extraction process.

2.6. Formulation and Characterization of Lignin-Based Sunscreen Cream

The sunscreen formulations were prepared by incorporating lignin extracted using two different methods—Deep Eutectic Solvent (DES; choline chloride–citric acid) and NaOH—into Moisturizer Soft Cream as the carrier matrix. Lignin was added at concentrations of 2%, 3%, and 4% (w/w). Each mixture was homogenized using an overhead stirrer at 1000 rpm for 2 h to ensure uniform dispersion of lignin within the cream matrix. Following homogenization, the resulting formulations underwent a series of characterization tests. A 0.5-g portion of each cream sample was diluted in 10 mL of ethanol and filtered to obtain a clear filtrate for analysis. UV–Vis spectrophotometry (Jasco model) was employed to record the UV absorption spectra of the formulations, providing insight into the contribution of lignin to the photoprotective performance of the cream. The pH of each formulation was measured using a calibrated pH meter to confirm that the products remained within a dermally safe pH range.

3. Results and Discussions

3.1. Pretreatment of Banana Stem Waste

The banana stem biomass was dried, ground, and sieved to 100 mesh prior to lignin isolation. The measured moisture content was 9.06%, indicating adequate dryness for further processing. Lignocellulosic composition was assessed using the Chesson method, revealing an average content of 12.00% lignin, 15.50% cellulose, and 18.50% hemicellulose. The lignin content is within the typical range for agricultural residues, indicating that banana stem waste can serve as a viable lignin source. From a functional perspective, the presence of native lignin chromophores offers potential advantages for applications requiring UV absorption, such as natural sunscreen formulations. The biomass was then subjected to a sequential washing process consisting of demineralized water (24 h) followed by ethanol (24 h). These steps facilitate the removal of water-soluble impurities, surface lipids, phenolics, and low-molecular-weight extractives.

Figure 2. (a) Chopped banana stem waste; (b) Ground banana stem powder; (c) Banana stem waste after washing.

FTIR analysis of the biomass before and after washing demonstrated notable spectral changes. The broad –OH stretching band (3400–3300 cm^{-1}) and the C–O stretching bands (~1030 cm^{-1}) exhibited reduced intensities after washing, indicating partial removal of hemicellulose and extractives, while maintaining the characteristic aromatic skeletal vibrations below 1000 cm^{-1}. Collectively, these modifications improved the accessibility of structural lignin during the extraction stage.

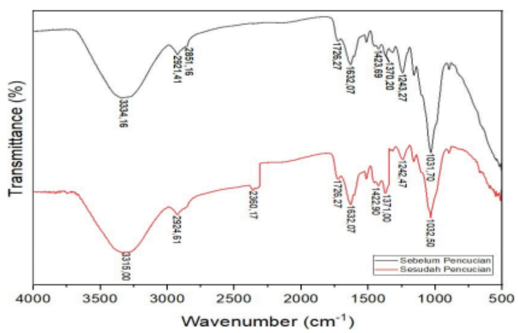

Figure 3. FTIR spectra of banana stem waste before and after washing.

3.2. Preparation of Deep Eutectic Solvent

The final DES exhibited an average viscosity of 110.67 cP at 50 °C, consistent with reported viscosities for organic-acid-based DES systems. The pH [illegible]e was markedly acidic (0.177), reflecting strong citric acid dissociation that release H^+ ions according to the reaction:

$$C_6H_8O_7 \leftrightarrow C_6H_7O_7^- + H^+ \quad (5)$$

The acidic environment facilitates acid-catalyzed cleavage of lignin-carbohydrate ester bonds in the biomass matrix:

$$Lignin - OC - CO - Hemicellulose + H^+ + H_2O \rightarrow Lignin - OH + Hemicellulose - COOH \quad (6)$$

The physicochemical consistency of the DES across replicates suggests thermal stability during extraction. This condition is essential for maintaining reproducibility and preventing solvent restructuring or decomposition during prolonged heating.

3.3. Extraction of Lignin from Banana Stem Waste

Lignin extraction using the prepared DES was conducted at 5% (w/w) biomass loading and refluxed at 100°C for 6 h. For comparison, alkaline extraction using NaoH (1:10 biomass-to-solvent ratio) at 100°C for 3 h was also conducted. Notably, NaOH extraction yielded 6.76 g lignin after six extraction cycles, compared to 1.48 g obtained using DES. The lower DES yield is attributable to stronger lignin–solvent interactions and the high viscosity of the DES matrix, which may impede complete lignin release. Color differences between the two lignin samples were evident, with DES-derived lignin appearing darker, suggesting preservation of aromatic chromophores, whereas NaOH-derived lignin exhibited a lighter shade indicative of partial structural degradation.

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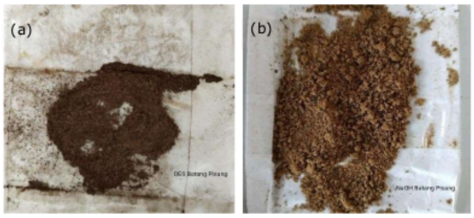

Figure 4. Colour comparison of (a) DES- and (b) NaOH-extracted lignin.

FTIR spectra of both lignin samples exhibited fundamental absorption bands characteristic of lignin. DES-extracted lignin displayed intense aliphatic C–H stretching at 2923 cm⁻¹, strong aromatic skeletal signals at 1513 and 1462 cm⁻¹, and well-defined C–O stretching bands at 1265, 1155, and 1019 cm⁻¹ associated with guaiacyl and syringyl units. These features indicate that DES extraction facilitated lignin isolation while preserving key structural motifs. Conversely, NaOH-extracted lignin exhibited a sharper carbonyl absorption band (~1687 cm⁻¹), consistent with oxidative cleavage and the formation of conjugated carbonyl groups. This peak is indicative of structural modifications commonly associated with alkaline pulping processes. Overall, the comparison suggests that DES extraction is less destructive, preserving the aromatic backbone essential for photoprotective applications.

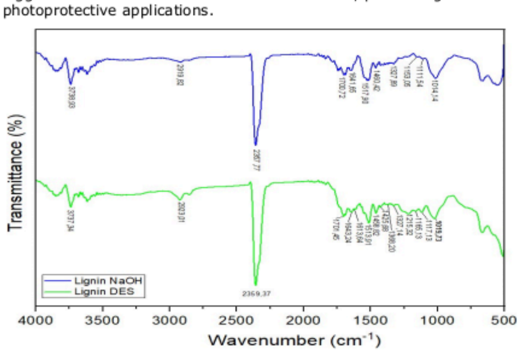

Figure 5. FTIR spectra of lignin NaOH and DES

3.4. Formulation of Lignin-Based Sunscreen Cream

The mixture of lignin obtained from both extractioon methods at various concentrations with the commmercial moiturizer base were homogenized at 1000 rpm for 30 min using an overhead stirrer to ensure even dispersion. Visual inspection revealed that creams containing DES-derived lignin were darker, reflecting the retention of aromatic chromophores, while formulations containing NaOH-derived lignin were relatively lighter. This visual distinction is consistent with the structural integrity of the lignin obtained through each method and may correlate with differences in UV-absorbing capacity. The pH of all formulated creams remained stable at pH 6. This value lies within the acceptable range defined by SNI 16-4399-1996 (4.5–8.0) and is compatible with human skin pH (4.5–6.75). These findings confirm that lignin incorporation does not adversely affect the acid–base balance of the cream matrix and supports the suitability of the formulations for topical application.

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Figure 6. Sunscreen cream with lignin concentrations 2%, 3%, and 4%

Figure 6 compares the UV–Vis absorbance profiles of sunscreen creams formulated with lignin extracted using (a) a choline chloride–citric acid deep eutectic solvent (DES lignin) and (b) conventional alkaline extraction (NaOH lignin). Both datasets were evaluated at lignin loadings of 2%, 3%, and 4% (w/w) to examine concentration-dependent optical performance across the UV-C (200–280 nm), UV-B (280–320 nm), and UV-A (320–400 nm) regions.

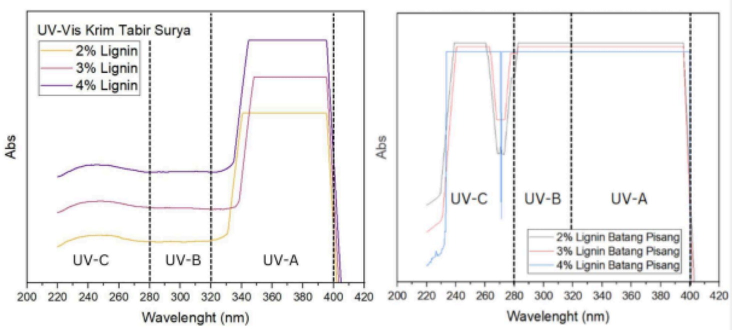

Figure 7. Sunscreen cream with lignin concentrations 2%, 3%, and 4% (a) DES-lignin and (b) NaOH-lignin

The DES lignin samples exhibit a distinct gradual absorbance increase from the UV-C to UV-B region, followed by a sharp transition around 320–330 nm, leading to a broad and stable absorption plateau throughout the UV-A region (Figure 6-left). This spectral behavior is consistent with the presence of conjugated phenolic structures, hydroxycinnamic derivatives, and aromatic chromophores typically retained in DES-based lignin. Notably, absorbance intensity increased proportionally to lignin concentration, with the 4% formulation demonstrating the strongest attenuation across the UV-B and UV-A regions. The smooth spectral profile and absence of significant band distortions suggest that DES extraction effectively preserves the native lignin chromophoric system, which enhances its ability to function as a broad-spectrum photoprotective agent. In contrast, the NaOH lignin formulations show a markedly different absorbance pattern (Figure 6-right). While all concentrations display strong absorbance in the UV-C and UV-B regions, the curves feature characteristic inflection points and depressions—particularly around 250–270 nm—indicating possible structural modifications arising from alkaline delignification. Alkaline treatment is known to promote fragmentation, demethoxylation, and partial condensation reactions, which may disrupt aromatic conjugation and modify the UV–Vis response. Nevertheless, NaOH lignin still provides high absorbance across UV-B and UV-A wavelengths, and similar to the DES samples, absorbance intensity increases with lignin concentration. The 4% NaOH lignin formulation records the highest plateau in the UV-A region, though its curve displays sharper transitions compared to the smoother DES lignin profile. These spectral distinctions suggest that alkaline lignin may possess higher light-scattering contributions or more heterogeneous chromophore distributions.

The collective results of this study provide a comprehensive evaluation of the structural, chemical, and functional characteristics of lignin extracted from banana stem waste. The compositional analysis confirmed that the lignin content of the biomass is comparable to other agricultural by-products, supporting its potential utility as a renewable precursor for high-value applications. Pretreatment and washing effectively removed hydrophilic and low-molecular-weight extractives, leading to a biomass matrix more receptive to solvent penetration and lignin release. The comparison between DES and NaOH extraction methods revealed pronounced differences in both yield and structural preservation. While the alkaline process resulted in a higher mass yield, the accompanying structural degradation—manifested by intensified carbonyl signals—may limit its suitability for applications requiring intact aromatic frameworks. In contrast, the DES system preserved key aromatic and aliphatic features, offering a structurally robust lignin suitable for photoprotective, antioxidant, or polymer-enhancing applications. The functional evaluation of lignin within a cosmetic formulation demonstrated that DES-extracted lignin consistently provided superior UV absorption compared to NaOH-derived lignin. This performance enhancement can be directly attributed to the preservation of conjugated aromatic structures, which function as natural UV chromophores. Moreover, the stability of the formulation pH and the absence of visible-light absorption position lignin as an attractive biobased ingredient for environmentally friendly sunscreen formulations. Collectively, these results highlight the importance of extraction methodology in determining lignin's structural and functional properties. The findings also provide compelling evidence that DES-based extraction offers a more sustainable, selective, and application-oriented approach compared to conventional alkaline extraction.

4. Conclusion

This study demostrates that banana stem waste is a promising renewable source for lignin isolation and valorization. Pretreatment and wasing effectively removed non-structural components, improving lignin extractability. Compared with alkaline extraction, the choine chloride-citric acid DES method better preserved the aromatic and aliphatic structures of lignin, although NaOH extraction produced higher yields. When incorporated into a topical cream formulation, DES derived lignin showed stronger UV absorption in the UVA and UVB regions while maintaining a pH compatible with cosmetic safety standards. Increasing lignin concentration enhanced UV absorption without affecting visible light transparency, indicating its potential as a natural photoprotective additive. Further studies on formulation stability, SPF determination, and dermatological safet are required to support its application in commercial sunscreen products.

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