

Green Synthesized Copper Oxide Incorporated on Transparent Nata de Coco Bacterial Cellulose Composite for Optical Detection of Cadmium(II) in Landfill Waste Leachate

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ABSTRACT

Reliable analytical techniques with low detection limits for cadmium are essential for environmental monitoring. This study developed transparent nata de coco bacterial cellulose films incorporated with copper(II) oxide (CuO) nanostructures for optical detection of cadmium(II) in landfill leachate. The cellulose–CuO composite film was produced through bacterial cellulose fermentation using *Acetobacter xylinum*, CuO nanostructure synthesis via alkaline precipitation and thermal treatment, and incorporation of CuO into transparent cellulose films. Dragon fruit peel extract served as a natural capping and stabilizing agent, while epoxy resin reinforcement improved film durability. The sensor's optical response to cadmium(II) was evaluated by UV–Vis spectrophotometry. The composite film showed excellent analytical performance with an RSD of 0.46%, recovery of 104.7%, LOD and LOQ of 0.04993 ppm and 0.05133 ppm, respectively, and robustness RSD of 0.7%, demonstrating its potential as an environmentally friendly sensor for rapid cadmium detection.

Keywords: Bacterial cellulose composite; Copper(II) oxide (CuO); Cadmium; Environmental monitoring; Optical sensor

1. Introduction

The utility of cellulose across diverse industries is attributed to its abundant natural presence and versatile fibrous structure. Beyond its conventional applications in paper and textiles, cellulose-based materials have garnered attention in fields such as medical devices, drug delivery systems, and wound healing due to their biocompatibility and biodegradability. Moreover, cellulose plays a pivotal role in nanotechnology, wherein cellulose nanocrystals and nanofibers exhibit exceptional mechanical strength and a high surface area, making them suitable for reinforced materials, coatings, and electronic components [1]. Cellulose nanocrystals (CNCs), which are defect-free rod-shaped crystalline products, can be obtained through the acid hydrolysis of cellulose [2]. Despite their impressive physical, chemical, and biological properties, the use of acid reagents poses environmental risks.

Bacterial cellulose, derived from bacteria such as *Acetobacter xylinum*, *Gluconacetobacter swingsii* sp., and *Acetobacter lovaniensis* HBB5, has gained significant attention due to its superior purity compared to plant cellulose and its distinct crystal structure [3, 4]. The synthesis of cellulose using *A. xylinum* has been extensively studied, with investigations into various carbon and nitrogen sources to optimize bacterial growth and cellulose production [5, 6]. This avenue of research could potentially lead to applications in optical sensor film matrices. In addition, several previous studies have demonstrated the successful utilization of nata de coco bacterial cellulose as a supporting matrix for nanomaterial synthesis, including bacterial cellulose/nano-graphite nanocomposite membranes [7], Fe-doped ZnO/bacterial cellulose-based composite for photocatalyt-

ics [8], micro-cellulose membranes for hydrogen fuel cells [9], and Pt-based bacterial cellulose membranes derived from carbon precursors [10], confirming its effectiveness as a structural scaffold for functional nanocomposites.

Noteworthy, previous studies have focused on innovative methods involving dispersing responsive nanostructures in an electrochromic (EC) membrane with casting formation, where cellulose membrane/film serves as a foundation for creating "smart" materials [11, 12]. Cellulose-based aerogels, known for their low thermal conductivity and lightweight properties, have been developed as insulation materials, making them suitable for high-performance composites, coatings, and films [2, 3].

Furthermore, enhancing the plasmonic activity of cellulose can be accomplished by incorporating gold nanorods for the easy and reproducible detection of different analytes using the Surface Enhanced Raman Scattering (SERS) technique. Skwierczyńska et al. [13] have developed regenerated cellulose fibers modified with gold nanorods in an efficient and simple way to fabricate SERS platforms. Their successful creation of nanocomposites has enhanced the detection of p-Mercaptobenzoic acid and Bovine Serum Albumin using the SERS technique. On the other hand, Gomes et al. [14] in a previous article mentioned the potential applications of these bacterial cellulose-based sensors as more sensitive and selective tools in chemical and biological detection. These principles of detection became an inspiration for developing bacterial cellulose composites incorporated with CuO nanostructures for optical sensor applications.

Based on these phenomena, an endeavor was made to synthesize CuO nanos-

structures using natural capping and stabilizing agents derived from plant extracts without the utilization of harsh chemicals or intense heat, which has emerged as a compelling research avenue. The synthesis offers straightforward and cost-effective procedures that are also environmentally friendly. CuO nanostructures coated with biomolecules have shown high catalytic efficiency in C-heteroatom (N, O, S) cross-coupling reactions. This supports the growing Nanostructure-Catalyzed Enhanced Organic Synthesis (NOSE) trend, offering a green and effective method to produce heterocycles with important biological and pharmaceutical applications [15].

Conversely, in Indonesia, the widespread use of cadmium across various industries, including battery manufacturing and pigment formulation, has raised alarms due to its environmental and human health repercussions. Cadmium is predominantly found in leachate from final processing sites, largely stemming from electronic waste, particularly discarded batteries, and if not managed appropriately, can lead to groundwater and surface water contamination. Cadmium, a toxic heavy metal, poses severe risks to the environment and human health. Prolonged exposure to cadmium can result in diverse health issues, including kidney and bone damage, as well as potential carcinogenic effects. Thus, timely detection and effective management of electronic waste are imperative to prevent environmental pollution and safeguard public health. Various methodologies, such as Atomic Absorption Spectrometry (AAS) and X-ray Fluorescence Spectrometry (XRF), have been deployed for cadmium detection, each carrying its own merits and limitations [16].

The development of specialized optical sensors utilizing nata de coco bacterial

cellulose composites with CuO nanostructures represents a potential breakthrough in the detection of cadmium in landfill leachate water. Harnessing the environmentally friendly attributes of bacterial cellulose and the semiconductor properties of CuO nanostructures, this innovative sensor system exhibits promising capabilities in selectively identifying hazardous cadmium ions in landfill leachate water, addressing critical environmental and health concerns. Within the realm of optical sensor systems, cellulose can function as a substrate supporting the growth of nanostructures or other plasmonic configurations. Cellulose-based substrates can amplify sensor sensitivity by enhancing light-matter interaction, while also providing essential structural stability and biocompatibility for biomedical and environmental applications. Consequently, cellulose plays a pivotal role in augmenting the performance and utility of optical sensors [17, 18].

Moreover, CuO nanostructures bound to the cellulose matrix can interact with cadmium(II) ions through surface functional groups, leading to measurable changes in optical absorption. The extent of optical response variation is contingent upon the concentration of cadmium(II) ions. Therefore, it is posited that cellulose-based optical films embedded with CuO nanostructures can serve as optical sensor membranes for the detection of cadmium(II) ions in samples. The rapid interaction between cadmium(II) ions and the cellulose matrix embedded with CuO nanostructures will heighten measurement sensitivity using this optical sensor via UV-Vis spectrophotometry, as the chelation process facilitates the measurement of absorption spectra.

Recent research in nanoscale optical sensing, focusing on the interaction of light

with resonant structures at the nanoscale, has emerged as a highly explored domain. Optical spectroscopy sensors, sensitive to environmental changes, rely on detecting shifts in the surface plasmon resonance spectrum triggered by alterations in the metal surface [19, 20]. Previous studies have utilized plasmonic sensor nata de coco film inserted with copper nanoparticles for the detection of mercury by UV-Vis spectrophotometry, showing high potential in applications for selective and sensitive detection of heavy metals [21]. This phenomenon is believed to apply to the analysis of cadmium ions utilizing the UV-Vis spectrophotometric technique. Given the urgent necessity to detect cadmium in landfill leachate, this study introduces the development of a specialized optical spectroscopic sensor employing nata de coco cellulose composite with CuO nanostructures as a potential avenue to enhance its sensitivity.

By leveraging the environmentally friendly attributes of nata de coco and the semiconductor characteristics of CuO nanostructures, this novel detection system showcases promising capabilities in specifically identifying detrimental cadmium ions in landfill leachate, thereby addressing significant environmental and health challenges. The functional groups of cellulose facilitate complexation with cadmium(II) ions, influencing the optical absorption characteristics in spectroscopic metal ion detection. This represents a novel step in heightening the sensitivity and selectivity of the sensor towards cadmium in landfill leachate. Furthermore, the use of optical composite films can enhance the speed and precision of detection, facilitating more efficient and environmentally friendly practices in waste management and environmental preservation. Consequently, this study unveils new potential in the advance-

ment of innovative and sustainable sensor technology.

2. Materials and Methods

2.1 Chemicals and materials

The materials utilized in this investigation comprised dragon fruit peel, coconut water, deionized water, ethanol (Emsure Merck), n-Hexane (Emsure Merck), Ethyl acetate (Emsure Merck), granulated sugar ($C_6H_{12}O_6$), ammonium sulfate (ZA), glacial acetic acid (Emsure Merck), silica gel-60 (Milipore Merck), *Acetobacter xylinum* stater, Whatman No filter paper 40, $CuSO_4 \cdot 5H_2O$ (Emsure Merck), NaOH (Emsure Merck), and Epoxy Resin.

The apparatus employed in this research encompassed a UV-Vis Spectrophotometer (Vernier), Rotary Evaporator (Buchi), Centrifuge 4000 RPM 220V (laborglas), Oven (YNC-OV50L), Hydraulic Hot Press Machine (HTM 634), Vacuum Liquid Chromatography (VLC), FTIR Spectrophotometer (Shimadzu), XRD (Phillips), and Scanning Electron Microscope (JEOL JSM-6360).

2.2 Methods

2.2.1 Extraction and bacterial cellulose preparation

The extraction process of dragon fruit peel waste entailed maceration. A quantity of 500 grams of *Simplicia* powder was placed into a 1000 mL beaker, followed by the addition of 96% ethanol. Subsequently, maceration was conducted for 3×24 hours with a filtration procedure every 24 hours, and the *simplicia* was then soaked again with a fresh solvent. The maserate underwent filtration using a funnel and filter paper, followed by separation. The collected maserate was evaporated for 3 hours using a rotary evaporator at $45^\circ C$ to yield red dragon fruit peel extract.

Purification of dragon fruit peel extract was achieved through vacuum liquid chromatography (VLC) based on the method outlined by Oetari et al. [17], utilizing gradient polarity solvents including n-hexane, ethyl acetate, and ethanol. Approximately 10 g of concentrated extract was loaded onto a silica gel stationary phase and sequentially eluted using increasing polarity solvent systems starting from n-hexane, followed by mixtures of n-hexane–ethyl acetate, ethyl acetate, and finally ethanol. This process involved separating the extract using a silica adsorbent column and collecting multiple semi-purified fractions based on polarity differences. The obtained fractions were concentrated using a rotary evaporator prior to further use.

Bacterial cellulose was produced through the fermentation of coconut water with added sugar (10% w/v) and ammonium sulfate (0.5% w/v) as a nitrogen source. The medium was inoculated with a starter culture of *Acetobacter xylinum* (10% v/v) and incubated statically at room temperature (28–30°C) for 10 days until nata sheets were formed. The resulting nata de coco was washed thoroughly and purified before further processing.

2.2.2 Synthesis of CuO nanostructure-dragon fruit peel extract

Sample codes CuO-DF5, CuO-DF10, CuO-DF15, CuO-DF20, CuO-DF25, and CuO-DF30 represent the volume (mL) of dragon fruit peel extract added into a fixed precursor solution during synthesis. CuO nanostructures were synthesized through a green-assisted alkaline precipitation method followed by thermal treatment, adapted from previous research [22]. Initially, 0.05 M copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), equivalent to 4.734 g, was dissolved in

500 mL of distilled water. This solution was combined with pure dragon fruit peel extract across the range of ratios mentioned above (CuO-DF5-30, (v/v)) where the extract functioned as a natural capping and stabilizing agent during nanostructure formation. Subsequently, the mixture underwent stirring via a magnetic stirrer for 120 minutes until a visible color change and precipitate formation were observed. The pH of the solution was adjusted to 12 by the addition of NaOH. Color changes within the mixture were monitored and recorded at intervals of 0, 10, 20, and 30 minutes. After that, the mixture was centrifuged at 3500 rpm for 10 minutes, and the precipitate was collected. Then it was carefully washed with distilled water and dried at 105°C overnight. The CuO nanostructure product was obtained by calcination at 400°C for 4 hours. Finally, the resulting product was quantified through yield calculation as a comparative synthesis efficiency parameter. All synthesis parameters including precursor concentration, pH adjustment, stirring duration, centrifugation conditions, and calcination temperature were maintained consistently to ensure experimental reproducibility.

Stability analysis was conducted using a UV-Vis spectrophotometer, while the crystallite size was determined using an XRD analyzer by applying the Debye-Scherrer equation, as shown in Eq. (2.1).

The average crystallite sizes (D) for CuO powders were calculated utilizing the Debye–Scherrer equation, where K is the Scherrer constant ($K = 0.9$) related to the crystallite shape, λ (nm) and θ (rad) are the X-ray radiation wavelength and the Bragg's angle, respectively, and β (rad) is the full width at half maximum (FWHM) of the diffraction peaks determined by a pseudo-

Voigt profile fit [23].

$$D = \frac{K\lambda}{\beta \cos \theta}. \quad (2.1)$$

2.2.3 Synthesis of transparent nata de coco cellulose incorporated CuO nanostructure

Fermented bacterial cellulose was immersed in 0.1 M NaOH solution for 1 minute to remove residual impurities, then rinsed with running water until reaching a neutral pH (pH $\approx$ 7). Subsequently, the bacterial cellulose was pressed using a hydraulic power press at a pressure of 300 psi and a temperature of 120°C for 10 min, producing thin film sheets with an approximate thickness of 0.05 cm. These films were cut to the size of a cuvette (1 $\times$ 5 cm) and coated using an epoxy resin. The coated films were air-dried for 12 h prior to nanostructure incorporation. The dried films were then immersed in an ethanolic dispersion of CuO nanostructure–dragon peel extract (0.1%) in various compositions for 30 min under static conditions until a visible color change was observed. It was ensured that the cellulose film was completely submerged, positioned horizontally, and free from air bubbles to promote uniform deposition. Transparent optical composite films were formed and left to dry at room temperature for 24 hours to yield transparent, robust films ready for characterization and optical sensor validation.

2.2.4 Characterization

The forthcoming characterization process will involve several stages. Initially, functional group analysis using FTIR will be conducted to identify chemical bonding and surface functional groups. Subsequently, an analysis of the crystal structure of the compound using X-ray diffraction (XRD) will be carried out to determine crystallinity and phase

composition. Finally, an analysis of the morphology and structure of the compound on a microscopic scale will be performed using a Scanning Electron Microscope (SEM).

2.2.5 Optical spectroscopic sensor test

The optical spectroscopic sensor test was executed using cadmium metal analyte samples interacting with optical composite films embedded with CuO nanostructure–dragon peel extract. Following this interaction, the films were analyzed using a UV-Vis spectrophotometer. During this stage, the film was placed into the cuvette and considered an interaction medium between analytes and UV-Vis light. Furthermore, the films were regarded as a substitute medium for complex solutions. The absorbance data obtained were utilized to construct a calibration curve for cadmium standard solutions with varying concentrations of 0.01, 0.02, 0.03, 0.05, 0.07, 0.09, and 0.10 ppm. From this calibration curve, the coefficient of determination (R^2) value was derived and subsequently tested for validity. The optical composite film demonstrating the highest validity was selected to determine the concentration of the unknown cadmium analyte.

3. Results and Discussion

3.1 Red dragon fruit peel extract and bacterial cellulose nata de coco

Extraction of red dragon fruit skin was carried out using the ethanolic maceration extraction method. The extraction results of red dragon fruit skin showed a deep red color as a result of evaporation, with a yield value of approximately 2% (w/w) crude extract. Ten grams of ethyl acetate fraction obtained from preliminary solvent partitioning produced a purified extract yield of approximately 20%. The vi-

sual representation of the extraction stages is shown in Fig. 1, which illustrates: a) the dragon fruit peel powder, and b) the ethyl acetate fraction of dragon fruit peel extract obtained after gradient elution purification.

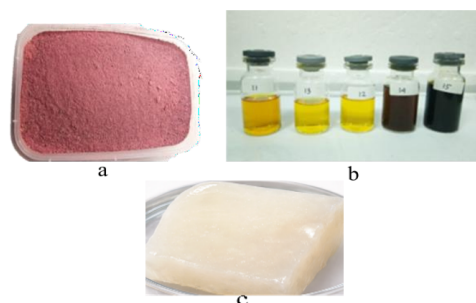


Fig. 1. (a) Dragon fruit peel powder, (b) Ethyl acetate fraction of dragon fruit peel extract obtained after gradient elution purification, (c) synthetic nata de coco.

The bacterial cellulose (nata de coco) produced through fermentation exhibited a translucent hydrogel-like appearance with a flexible sheet morphology, as shown in Fig. 1(c). The uniform thickness and smooth surface indicate successful formation of bacterial cellulose suitable for thin-film processing. The fibrous and highly hydrated structure of nata de coco provides a porous matrix that facilitates nanoparticle immobilization and enhances light–material interaction during optical sensor fabrication.

3.2 Synthesis of CuO-dragon fruit peel extract nanostructure

The synthesis results and visual color variations of precursor solutions mixed with different volumes of red dragon fruit peel extract are presented in Fig. 2. The observed color changes are associated with the formation and stabilization of CuO nanostructures under alkaline conditions.

Synthesis of CuO nanostructure-

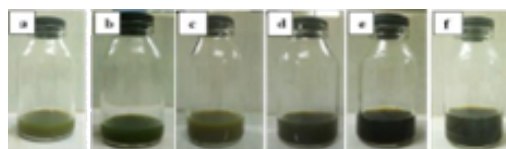


Fig. 2. Visual appearance and color variation of CuO precursor solutions during green-assisted alkaline precipitation synthesis using different volumes of dragon fruit peel extract: (a) CuO-DF5, (b) CuO-DF10, (c) CuO-DF15, (d) CuO-DF20, (e) CuO-DF25, and (f) CuO-DF30.

extract from fruit peel using copper sulfate pentahydrate precursor ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), was successfully synthesized by green chemistry using an alkaline precipitation process followed by thermal conversion (calcination). The synthesis of CuO nanostructures produces solutions of various colors. The observed color variation is associated with particle formation, complexation with biomolecules, and structural evolution during synthesis rather than any change in the oxidation state of copper.

The resulting CuO product can be seen in Table 1, which presents the results of dividing the yield weight by the theoretical weight. The theoretical yield was calculated stoichiometrically based on the conversion of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ precursor to CuO after calcination. This table describes the synthetic composition of copper oxide.

Table 1. Yield CuO.

Sample Code	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (g)	Extract (mL)	Experimental mass of CuO (g)	Theoretical mass of CuO (g)	Yield (%)
CuO-DF5	4.734	5	0.02322	1.51	1.54
CuO-DF10	4.734	10	0.03451	1.51	2.29
CuO-DF15	4.734	15	0.04461	1.51	2.95
CuO-DF20	4.734	20	0.05217	1.51	3.46
CuO-DF25	4.734	25	0.05654	1.51	3.75
CuO-DF30	4.734	30	0.05934	1.51	3.93

*CuO : Copper Oxide, DF : Dragon fruit

The yield percentage of CuO was calculated by comparing the experimentally obtained dry mass with the theoretical yield derived from the stoichiometric conversion of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ precursor to CuO under alkaline precipitation conditions. The theoretical yield was determined based on molar mass calculations assuming complete conversion of copper ions into CuO. Yield (%) was calculated using the equation: $\text{Yield (\%)} = (\text{experimental mass of CuO} / \text{theoretical mass of CuO}) \times 100\%$. Differences in yield among samples may be influenced by extract volume, nucleation efficiency, particle aggregation, and material loss during washing and drying processes.

CuO combined with dragon fruit peel extract undergoes a transformation in color from a verdant hue to a deep emerald shade. The emergence of particles is denoted by a shift in the color of the resultant nanostructure. This alteration in color signifies a reaction to the amalgamation induced by red dragon fruit peel extract, which harbors anthocyanin pigment compounds. The presence of conjugated double bonds within the chromophore segment of the anthocyanin framework enables the absorption of light energy, predominantly within the visible light spectrum. The intensity of the coloration strengthens as the anthocyanin molecules extend their conjugated double bonds [24].

Dye molecules are energized during the sensitization process by assimilating visible light, subsequently leading to the transfer of electrons from the highest-occupied molecular orbital (HOMO) to the lowest-unoccupied molecular orbital (LUMO). The successful synthesis of the CuO nanostructure is subject to various factors such as precursors, extract concentration, pH, temperature, and duration. Varying the volume ratio of the extract to the pre-

cursor and adjusting the pH level can yield diverse outcomes. The optimal pH for the biogenesis of CuO nanostructure resides in an alkaline environment. NaOH is gradually introduced during the synthesis process to regulate the pH of the solution. NaOH acts as a base to elevate the pH, fostering the formation of the CuO nanostructure. Alkaline conditions promote the formation of Cu(OH)_2 intermediates which are subsequently converted into CuO during calcination. Elevating the pH level during the biosynthesis reaction results in the production of smaller nanostructures. At acidic pH, the effectiveness of carboxyl-containing compounds in dragon fruit peel extract as capping and stabilizing agents decreases, leading to the formation of larger particles due to reduced stabilization. In contrast, alkaline pH promotes better stabilization of the CuO nanostructure core, preventing further particle growth [25, 26].

3.3 Stability analysis of copper sulfate-red dragon fruit peel extract

Utilizing a UV-Vis spectrophotometer entails scrutinizing optical absorption behavior to observe the interplay between copper oxide and the red dragon fruit peel capping/stabilizing compounds. This evaluation aids in assessing the stability of the copper oxide nanostructure by examining its absorption patterns. The synthesized nanostructure exhibits a peak absorption range spanning from 400 to 900 nm, signifying the formation of CuO-based nanostructures influenced by biomolecular stabilization from dragon fruit peel extract. The absorption peaks depicted at 0, 10, 20, and 30 minutes in Fig. 3 showcase consistent wavelengths, indicative of sound stability within the nanostructure amalgam.

A shift in the absorption peak towards longer wavelengths denotes a rela-

tively diminished stability owing to nanostructure agglomeration. Conversely, a shift towards shorter wavelengths signifies an escalation in the interaction energy between the copper oxide nanostructure and the stabilizing phytochemical compounds. This alteration arises from variations in the optical characteristics of the copper oxide nanostructure when the particles aggregate, causing delocalization and dispersion of conduction electrons near the particle surface. Consequently, surface plasmon resonance undergoes a transition to lower energy levels, prompting the absorption peak to shift towards longer wavelengths [27].

3.4 Synthesis of bacterial cellulose plasmonic film-CuO nanostructure

Optical sensor films are fabricated using nata de coco as a substrate due to its acknowledged potential for generating composites alongside CuO nanostructures. The fibrous composition and porous nature of nata de coco facilitate the integration of metal nanostructures, enabling them to interact and adhere onto the cellulose fibers' surface within the nata de coco framework. The synthesized optical sensor film exhibits a verdant to amber hue, corresponding to the color of the CuO nanostructure solution derived from red dragon fruit peel extract, validating the presence of the synthesized CuO nanostructures embedded in the nata de coco film. The affinity between metal ions and cellulose is believed to be mediated through the exchange of electron pairs from oxygen atoms, originating from hydroxyl and ether groups on cellulose, to transition metal cations, leading to the formation of complexes. An illustration depicting the interplay between cellulose and the nanostructured CuO with red dragon fruit peel extract is showcased in Fig. 4. The optical response of the CuO-cellulose film to-

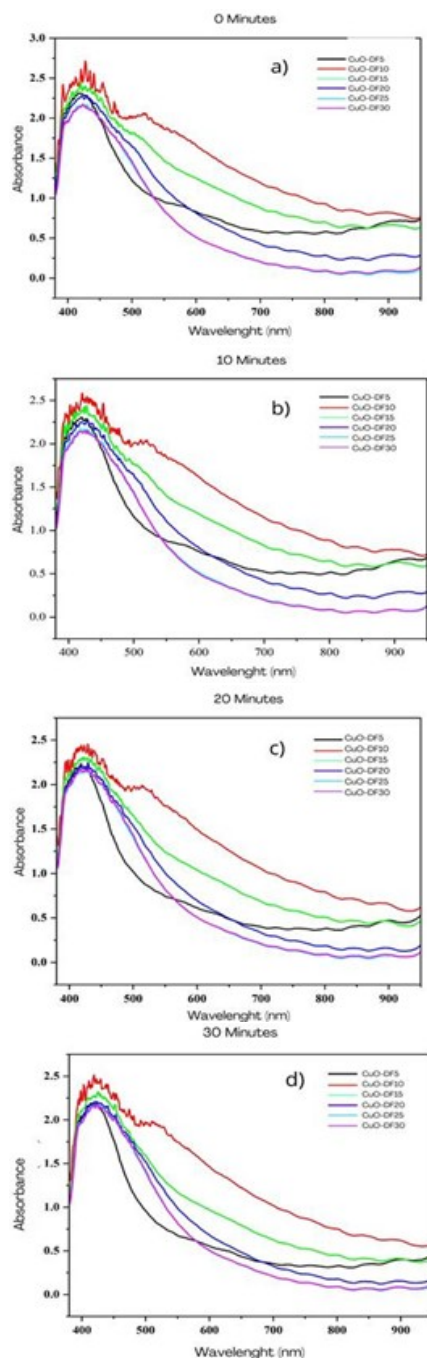


Fig. 3. Absorption spectrum of the UV-Vis spectrophotometer generated through the synthesis of nanostructured copper oxide (CuO) and red dragon fruit peel extract at various time intervals: (a) 0 minutes, (b) 10 minutes, (c) 20 minutes, and (d) 30 minutes.

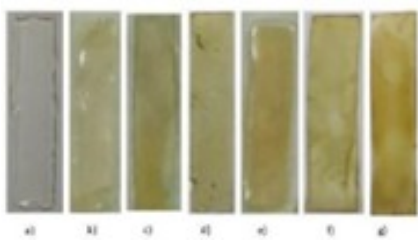


Fig. 4. 7 Comparisons of a) Cellulose film and the synthesis of optical sensor CuO films, b) CuO-DF5, c) CuO-DF10, d) CuO-DF15, e) CuO-DF20, f) CuO-DF25, and g) CuO-DF30.

ward Cd^{2+} was evaluated based on changes in UV-Vis absorbance spectra rather than direct visual observation, as the sensing mechanism relies on spectroscopic optical interaction.

CuO nanostructures are intricately dispersed within the cellulose matrix. The hydroxyl group is adept at securely binding copper ions to cellulose through electrostatic and ion-dipole interactions or complexations, facilitating the stabilization of CuO nanostructures through post-reduction surface interactions. This process offers the distinct advantage of leveraging the host macromolecular chain not only in the synthesis process but also in enhancing the dispersion of copper nanostructures within the cellulose matrix while averting the formation of aggregates. Simultaneously, polymer chains play a pivotal role in ensuring a narrow size distribution and a well-defined shape of the metal nanostructure [28].

3.5 Characterization

3.5.1 Functional group analysis utilizing fourier transform infrared (FTIR) technique

The FTIR characterization analysis was conducted to identify the functional groups present in the red dragon fruit peel extract, bacterial cellulose-CuO optical sensor composite film, and NP-CuO-DF pow-

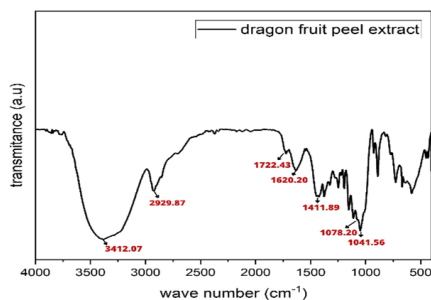


Fig. 5. Spectrum analysis of red dragon fruit peel extract.

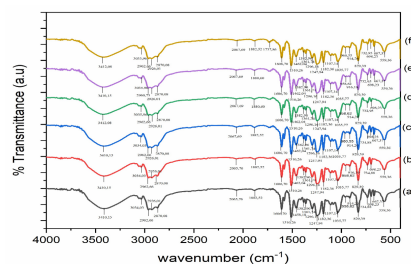


Fig. 6. FTIR spectrum of cellulose-NP-CuO-DF bacterial composite film, (a) CuO-DF5, (b) CuO-DF10, (c) CuO-DF15, (d) CuO-DF20, (e) CuO-DF25, and (f) CuO-DF30.

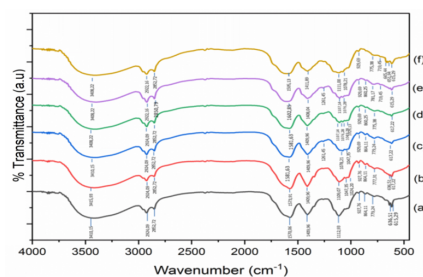


Fig. 7. FTIR spectrum of NP-CuO-DF powder, (a) CuO-DF5, (b) CuO-DF10, (c) CuO-DF15, (d) CuO-DF20, (e) CuO-DF25 and (f) CuO-DF30.

der. The outcomes of the analysis can be observed in Figs. 5-7.

The FTIR spectrum data of dragon fruit peel reveals a broad absorption peak at the wave number 3412.07 cm^{-1} , indicative of the presence of an -OH functional group. This absorption is complemented by

another peak at 2929.87 cm^{-1} , corresponding to the C-H alkane bond. Additionally, the absorption peak at 1722.43 cm^{-1} signifies the existence of C=O groups, while the peak at 1620.20 cm^{-1} indicates aromatic C=C or conjugated functional groups associated with plant-derived phytochemicals. The peaks at 1411.89 cm^{-1} and 1078.20 cm^{-1} are attributed to C-H bending and C-O-C stretching vibrations, and the peak at 1041.56 cm^{-1} is associated with a C-O bond.

The functional vibrational properties of the films were further characterized. The FTIR spectrum was acquired within the range of $400\text{--}4000\text{ cm}^{-1}$ (wavenumber). Fig. 6 illustrates the FTIR spectrum of the NP-CuO-DF optical sensor composite film. Nearly all samples exhibit a consistent vibration band. The absorption band observed at 559.36 cm^{-1} signifies the bending of the Cu-O bond [29], with a pronounced absorption band within the $400\text{--}600\text{ cm}^{-1}$ range. Characteristic bacterial cellulose bands were also observed around $\sim 3340\text{ cm}^{-1}$ (O-H stretching), $\sim 2890\text{ cm}^{-1}$ (C-H stretching), and $1030\text{--}1160\text{ cm}^{-1}$ (C-O-C and β -glycosidic linkage vibrations), confirming the presence of the cellulose matrix within the composite structure.

3.5.2 Crystal size analysis of copper oxide nanostructure

The 2θ values recorded in the diffraction patterns correspond to the diffraction angles detected during XRD analysis and reflect the distance between the planes in the crystal structure of the synthesized material. The XRD pattern of NP-CuO-DF powder is presented in Fig. 8(a), which shows the crystal characteristics of copper oxide nanostructures synthesized using red dragon fruit peel extract as a capping agent.

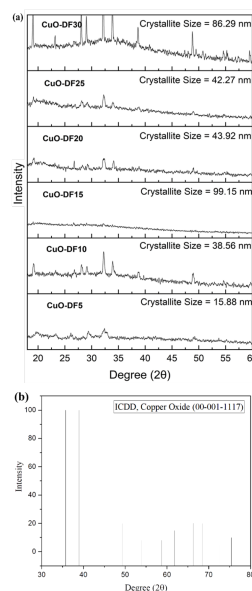


Fig. 8. XRD patterns of (a) NP-CuO-DF, and (b) standard CuO reference data obtained from the ICDD No. 00-001-1117.

The diffraction pattern exhibits several peaks located at approximately $2\theta = 19.05^\circ, 23.20^\circ, 28.02^\circ, 28.98^\circ, 32.15^\circ, 33.88^\circ, 38.95^\circ, 49.21^\circ, 50.76^\circ,$ and 54.63° . These diffraction peaks are associated with atomic plane reflections within the CuO crystal lattice. Phase identification was performed by comparison with standard copper oxide reference data obtained from the International Centre for Diffraction Data (ICDD No. 00-001-1117), as illustrated in Fig. 8(b). The characteristic peaks observed at 2θ values of $35.74^\circ, 38.95^\circ, 49.21^\circ, 53.88^\circ, 58.76^\circ, 61.79^\circ, 66.22^\circ, 72.67^\circ,$ and 75.37° correspond to the crystallographic planes $(-111), (111), (-202), (020), (202), (-113), (022), (220), (311),$ and (004) , respectively.

The presence of sharp and well-defined diffraction peaks indicates a good degree of crystallinity within the synthesized material. In particular, the peaks at $2\theta = 38.95^\circ$ and 49.21° can be attributed to

the (111) and (-202) planes of monoclinic tenorite CuO, confirming that CuO represents the dominant crystalline phase formed during synthesis. Minor diffraction features detected at lower angles may originate from residual organic components of the plant extract or contributions from the bacterial cellulose matrix in the composite structure.

3.5.3 Morphological analysis using digital SEM

The morphological analysis aimed to examine the surface structure and microstructural features of the nata de coco cellulose-CuO-DF optical sensor film, including fiber distribution, pore structure, and the dispersion of copper oxide particles under different extract volume variations. The SEM observations were conducted at a magnification of $2500\times$ to evaluate the interaction between the cellulose matrix and the incorporated CuO particles. The outcomes of the morphological analysis are presented in Fig. 9.

The SEM images reveal a fibrous cellulose network structure with irregular pores, indicating the inherent morphology of bacterial cellulose. The presence of granular or aggregated particles attached to the fiber surface suggests the successful incorporation of CuO particles within the cellulose matrix. Variations in extract volume ratio influence particle distribution and agglomeration behavior, where higher extract volumes tend to produce more uniform particle dispersion due to improved stabilization by bioactive compounds acting as capping agents.

3.6 Optical sensor evaluation

Initial assessments were conducted to determine R^2 for each comparison of optical sensor films. The optical sensor films underwent testing using standard cadmium metal solutions with concentrations rang-

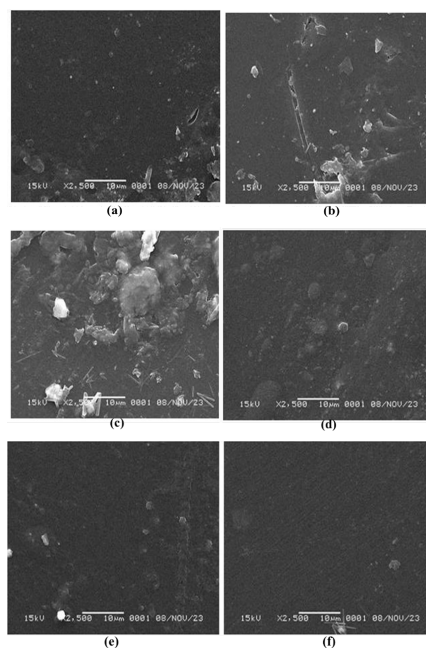


Fig. 9. Morphological Examination of CuO-DF optical sensor composite film utilizing Scanning Electron Microscopy over the range of precursor volume ratios: (a) CuO-DF5, (b) CuO-DF10, (c) CuO-DF15, (d) CuO-DF20, (e) CuO-DF25, and (f) CuO-DF30.

ing from 0.01 to 0.10 ppm. This allowed for the collection of absorbance data, which was then utilized to construct a calibration curve based on UV-Vis optical absorbance response. The outcomes of the optical sensor film calibration curve analysis on cadmium metal standard solutions are illustrated in Fig. 10.

The CuO-DF5 sample exhibits the highest R^2 value of 0.9913, indicating a superior level of alignment between the calibration curve and experimental data. This high R^2 value signifies strong linear correlation between absorbance response and cadmium concentration within the evaluated range, supporting the suitability of the optical sensor film for quantitative analysis.

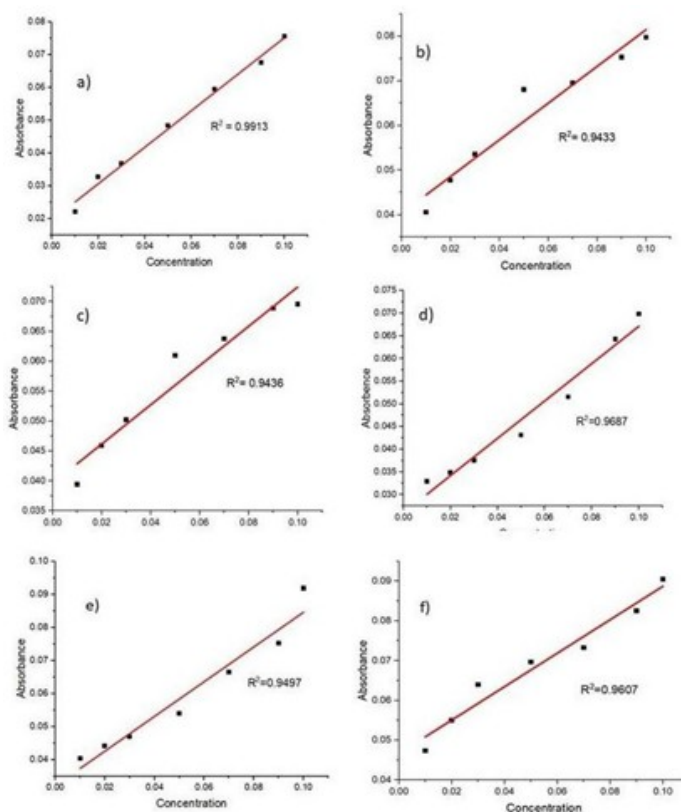


Fig. 10. Calibration curve of optical sensor CuO films in standard solutions of cadmium metal, a) CuO-DF5, b) CuO-DF10, c) CuO-DF15, d) CuO-DF20, e) CuO-DF25, f) CuO-DF30.

3.6.1 Precision

The percent relative standard deviation (%RSD) value obtained in this investigation stands at 0.46%, reflecting an exceptional level of precision. A test method is deemed precise if it yields a %RSD or precision of $\leq 2\%$, demonstrating compliance with validation criteria. The results of this analysis are presented in Table 2, which summarizes the determination of %RSD.

3.6.2 Accuracy

Determination of the accuracy value in this investigation is articulated through the absorbance value derived from optical sensor film assessments. The accuracy value is represented as a percentage of recovery obtained from an examination in-

Table 2. Determination of % RSD.

Replication	Absorbance of Analyte Samples	Absorbance of Analyte Sample + Standard Solution
1	0.0469	0.0482
2	0.0473	0.0478
3	0.0469	0.0485
4	0.0472	0.0482
5	0.0468	0.0478
6	0.0469	0.0477
7	0.0473	0.0475
Average	0.0470	0.0480
% RSD	0.46%	0.73%

volving the addition of multiple analytes to the specimen. Accuracy can be ascertained through the standard addition technique, in

which various standard solutions are introduced to the specimen, subsequently comparing the disparity between the specimen concentrations pre- and post-addition of the standard solution against the genuine concentration. The percentage of recovery achieved in this research was 104.7%, signifying acceptable analytical accuracy within the validation range. A recovery value within the 80-110% range is deemed acceptable. The complete data for analyte recovery determination is presented in Table 3.

Table 3. Determination of the % recovery of analyte samples.

Replication	Absorbance of Analyte Samples	Absorbance of Analyte Sample + Standard Solution
1	0.0469	0.0482
2	0.0473	0.0478
3	0.0469	0.0485
4	0.0472	0.0482
5	0.0468	0.0478
6	0.0469	0.0477
7	0.0473	0.0475
Average	0.0470	0.0480
% Recovery	-	104.7%

3.6.3 Limit of detection and limit of quantitation

Determination of the limit of detection (LOD) and limit of quantitation (LOQ) in this study is predicated on the analysis of a standard solution containing cadmium metal utilizing CuO-based optical sensor film measured by UV-Vis spectrophotometry. The absorbance results for LOD and LOQ can be observed in Table 4.

The findings of this research demonstrate significant outcomes, where LOD represents the minimum detectable concentration of an analyte that may not be precisely quantified. The LOD value of

Table 4. Determination of LOD and LOQ.

Repetition	Absorbance	Concentration
1	0.0469	
2	0.0473	
3	0.0469	
4	0.0472	
5	0.0468	
6	0.0469	
7	0.0473	
Average	0.0470	
Standard Deviation	0.0002	
LOD (Average + 3 SD)	0.047643	0.049926 ppm
LOQ (Average + 10 SD)	0.049043	0.051303 ppm

0.049926 ppm indicates the capability of the optical sensor film to detect low cadmium concentrations within the evaluated experimental conditions.

On the other hand, LOQ represents the minimum quantifiable concentration of an analyte with a satisfactory level of precision and accuracy. With an LOQ value of 0.051303 ppm, the optical sensor film enables quantitative analysis within the tested range. The LOD and LOQ values are presented to demonstrate the analytical sensitivity and quantification capability of the developed optical sensing system rather than regulatory compliance assessment.

3.6.4 Ruggedness and robustness

Ruggedness and robustness tests were conducted by comparing absorbance measurements of the optical sensor film at various time intervals, specifically at 5, 10, 15, 20, 25, 30, and 35 minutes. The variations in absorption values obtained with different measurement times are detailed in Table 5.

Based on the measurement results, the %RSD in the ruggedness and robustness tests was determined to be 0.7%. The

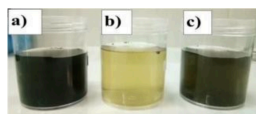
Table 5. Determination of LOD and LOQ.

Time Interval (Minute)	Analyte Absorbance	Spike Absorbance
5	0.0565	0.0592
10	0.0572	0.0578
15	0.0569	0.0585
20	0.0576	0.0582
25	0.0569	0.0578
30	0.0572	0.0577
35	0.0578	0.0577
Average	0.0572	0.0581
% RSD	0.7%	0.9 %

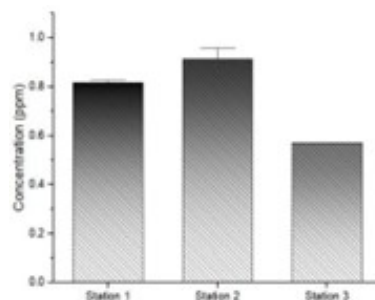
analytical method employed in the validation of cadmium detection using a UV–Vis optical sensor approach demonstrates commendable resilience and durability as it remains impervious to test variations encountered during measurement, meeting the stipulated criteria for the acceptable %RSD value.

3.6.5 Leachate test

Leachate samples taken at a point at the final waste disposal site are shown in Fig. 11, which illustrates leachate water collected from three different locations within the landfill area. These samples were analyzed using UV-Vis spectrophotometry in combination with the CuO-based optical sensor film to evaluate the absorption characteristics and corresponding concentrations of analytes present in the leachate. The results of this analysis are summarized in Table 6, which presents the absorption and concentration values in landfill leachate.

**Fig. 11.** Leachate water samples in three different locations.

By utilizing an optical sensor film calibration curve within standard cadmium

**Fig. 12.** The concentration of cadmium metal in the leachate of each station at the landfill.

metal solution with the equation $y = 0.5569x + 0.0195$, the concentrations of cadmium metal in the three leachate samples from stations 1, 2, and 3 were determined to be 0.818058756, 0.913985, and 0.570748 ppm, respectively (Fig. 12). These results demonstrate a proportional relationship between absorbance and concentration consistent with the Beer–Lambert law. The analysis of leachate samples was conducted to evaluate the practical detection capability and analytical response of the developed optical sensing film toward real environmental matrices.

The concentration of cadmium metal at the three research stations exceeded the threshold specified in the Decree of the State Minister of the Republic of Indonesia for the Environment No.51 of 2004, which is 0.001 ppm (refer to Fig. 12) [31].

4. Conclusion

This study entailed the fabrication of CuO nanostructures utilizing green chemistry, with red dragon fruit peel extract. The transition in color from green to dark green signifies the formation of copper oxide nanostructures. Different proportions of Cu^{2+} precursor and peel extract were employed, leading to color variations and the formation of precipitates. Various factors impacted the efficacy of CuO nanostruc-

Table 6. Absorption and concentration values in landfill leachate.

No	Absorbance			Concentration (ppm)		
	Station 1	Station 2	Station 3	Station 1	Station 2	Station 3
1	0.4790	0.5532	0.3344	0.8251	0.9583	0.5656
2	0.4586	0.5536	0.3359	0.7885	0.9591	0.5682
3	0.4699	0.5389	0.3369	0.8088	0.9326	0.5701
4	0.5017	0.5086	0.3375	0.8659	0.8783	0.5711
5	0.4659	0.4880	0.3418	0.8017	0.8413	0.5788
Average	0.4750	0.5284	0.3373	0.8181	0.9139	0.5707

ture synthesis, including precursors, extract concentration, pH, temperature, and duration. The pH plays a pivotal role, with an alkaline setting promoting the creation of smaller nanostructures, while acidity fosters the growth of larger particles due to diminished capping agent activity. Stability assessment via UV-Vis spectroscopy validated the generation of nanostructures associated with optical absorption characteristics of CuO nanostructures. The stability was affirmed by consistent absorption peaks within the 400-900 nm range in the synthesized nanostructure, indicating robust stability. Subsequent investigations delved into the development of optical sensor films by incorporating CuO nanostructures into a nata de coco matrix. The interaction between metal ions and cellulose in the matrix facilitated the successful deposition of CuO nanostructures, resulting in a yellowish-green optical sensor film. FTIR analysis identified distinct functional groups in red dragon fruit peel extract, bacterial cellulose-NP-CuO-DF composite film, and NP-CuO-DF powder, confirming the presence of diverse bonds and groups. Crystal size assessment via XRD revealed a characteristic peak at a specific angle (2θ value), signifying the existence of a copper oxide crystal structure. Furthermore, morphological analysis through SEM showcased the pore structure and particle size of copper oxide with varying ex-

tract ratios. Optical sensor evaluations were conducted to detect cadmium metal in standard solutions, demonstrating commendable precision (%RSD = 0.46%), satisfactory accuracy (% recovery = 104.7%), and a low LOD (0.049926 ppm), indicating the analytical capability of the developed sensing system. Leachate testing demonstrated that the developed optical sensing film can respond to cadmium presence in real environmental samples, supporting its practical analytical applicability.

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