Utilization and Characterization of the Cellulose Nanocrystalline Derived from Durian(Durio zibethinus) Husk as Potential Packaging Material

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Utilization and Characterization of the Cellulose Nanocrystalline Derived from Durian (*Durio zibethinus*) Husk as Potential Packaging Material

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Abstract: *Plastic packaging extends shelf life but often ends up in landfills due to limited recyclability. Cellulose-based alternatives, especially those using nanocellulose, are gaining attention. This study explores durian husk, an abundant biomass waste, for producing cellulose nanocrystalline (CNC) to reinforce biocomposite films at varying concentrations (0–7%). Films with 5 wt.% CNC showed the highest elongation at break. CNC extraction was confirmed via DLS (53 nm particle size) and FTIR. SEM-EDX revealed a rough surface structure due to starch and CNC. TGA showed thermal stability in the films. Biodegradability, mechanical properties, and water absorption were also analyzed. No antimicrobial activity was detected. Overall, durian husk-derived CNC is a promising reinforcement for sustainable packaging.*

Keywords: Durian Husk; Cellulose Nanocrystalline; Biocomposite Film; Packaging Material.

1. INTRODUCTION

Plastic production, waste generation, and environmental pollution are driven by the growing human population and the continuous demand for plastics and plastic products [1]. In 2015, humans produced 8.3 billion metric tons of plastic, of which 6.3 billion tons became waste. If current trends continue, the amount of plastic waste in landfills is projected to reach 12 billion metric tons [2]. To address this issue, eco-friendly alternatives to conventional packaging, emphasizing environmental sustainability, are necessary. Several studies have identified cellulose, proteins, and starch as promising materials for biodegradable packaging, with unique properties derived from plants and crop waste. Cellulose is a versatile industrial material due to its abundance, renewability, biodegradability, and ease of chemical modification [3]. It can be converted into nanocellulose using mechanical and chemical treatments [4]. Nanocellulose is increasingly important in food packaging due to its sustainable production from agricultural waste.

Durian husks, a major source of biomass waste, pose an environmental problem when discarded in landfills or burned [5, 6]. Thus, utilizing durian husk can help reduce its environmental impact. On Mindanao Island in the Philippines, durian production reaches approximately 54,700 metric tons annually, resulting in about 22,000 metric tons of waste, much of which ends up in landfills or decomposes along roadsides [7]. Durian husk contains 57–64% cellulose, making it a viable source for cellulose nanocrystals (CNC) extraction [8]. Romadhon and Sanjaya [8] noted that high cellulose content can produce environmentally friendly packaging films suitable for food and non-food applications. This study evaluates the use of CNC derived from whole durian husk as a reinforcing agent in five biocomposite film formulations with different CNC concentrations (0%, 1%, 3%, 5%, and 7%). The films were prepared using the solution casting method and assessed for mechanical, morphological, thermal, and physical properties, as well as biodegradability and antibacterial activity. A previous study by Romadhon and Sanjaya [9] extracted CNC only from the white part of the durian husk. However, to the best of the authors' knowledge, no study has yet used the entire durian husk for CNC extraction. Therefore, this study aims to utilize and characterize CNC extracted from the whole durian husk as a potential reinforcing agent for biocomposite film production.

2. MATERIALS AND METHODS

2.1 Material and Sample Preparation

Durian husk waste was collected from Langihan Market, Butuan City, Philippines. Chemicals were purchased from Dalkem Corporation, while other materials were obtained from the post-harvest laboratory at the Caraga State university (CSU)-Main Campus. For sample preparation, the durian husk was cut into smaller pieces, washed under running tap water, and soaked in water for 24 hours to remove impurities. The cleaned husk was then oven-dried at 120 °C for four hours. After drying, the husk was ground into powder, sieved using a 200-mesh screen, and stored in a desiccator until use.

2.2 Chemical Treatment

The durian husk powder was pre-treated with a mixture of 90% acetic acid and 10% sulfuric acid at 100 °C for 4 hours. The sample was then filtered and washed four times to remove residual chemicals. Next, it underwent alkaline treatment using 10% (w/v) sodium hydroxide at a 1:10 ratio, stirred and heated at 100 °C for 2 hours, followed by filtration and repeated washing with distilled water. For bleaching, the sample was treated with 200 mL of sodium hypochlorite in 1000 mL of distilled water at 100 °C for 4 hours, then filtered and washed. A second bleaching was performed using 400 mL of concentrated sodium hypochlorite under the same conditions. The resulting insoluble residue was filtered, washed thoroughly, and adjusted to pH 7.

2.3 CNC Extraction and Biocomposite Production

CNC was extracted following Romadhon & Sanjaya [9], with slight modifications. Acid hydrolysis was carried out using 45% (w/w) sulfuric acid at 50 °C for 2 hours. Twenty grams of cellulose were added to 100 mL of sulfuric acid and stirred vigorously at 1500 rpm. The reaction was stopped by diluting the suspension tenfold with cold distilled water, followed by centrifugation at 4500 rpm for 10 minutes. The residue was rinsed and dialyzed with cold distilled water until neutral pH was achieved. The suspension was then ultrasonicated for 10 minutes at 50 kHz using a Qsonica Sonicator Q55 and stored at 5 °C. Biocomposite films were produced via the solution casting method by mixing chitosan, starch, acetic acid, glycerin, and CNC at varying concentrations (0%, 1%, 3%, 5%, and 7%). The mixture was stirred at 300 rpm for 50 minutes at 170 °C, poured into a 6×6 in acrylic mold (30 mm thick), and oven-dried at 50 °C for 4 hours. Films were then removed and stored for further analysis.

2.4 CNC Characterization and Biocomposite Film Evaluation

The extracted cellulose nanocrystals (CNC) were characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Dynamic Light Scattering (DLS). FTIR analysis was conducted at the Chemistry Analytical and Research Laboratory, Ateneo de Davao University, while DLS was performed at the NanoCentre, Chemistry Department, CSU-Main Campus. The developed film samples were evaluated for elongation at break, Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX) analysis, thermal stability, water absorption, biodegradability, and antibacterial activity to assess their potential for food packaging. Elongation-at-break testing was conducted using a hydraulic testing machine at Equi-Parco Construction Company in Butuan City. SEM-EDX analysis was performed at Ateneo de Davao University, and thermogravimetric analysis (TGA) was carried out at the University of the Immaculate Conception Science Resource Center in Davao City. Biodegradation was monitored over 12 days in both normal and organic soils. For the water absorption test, based on a modified ASTM D570-98 method, film samples (2-in diameter) were oven-dried for 24 hours, then soaked and weighed at 1-hour intervals for 6 hours. Antibacterial testing was conducted at the CSU-Main Campus Biology Laboratory.

3. RESULTS AND DISCUSSION

3.1 Characterization of Cellulose Nanocrystalline

3.1.1 Dynamic Light Scattering (DLS) Analysis

Dynamic Light Scattering (DLS) is used to measure particle sizes in the sub-micron range. As shown in Figure 1, the extracted CNC had a particle diameter of 53 nm and a Polydispersity Index (PI) of 0.2, confirming its nanoscale size (<100 nm in one dimension) [10]. According to Thilak et al. [11], the PI indicates the size distribution of particles in a sample: values <0.05 suggest a monodisperse system, while values >0.7 indicate high polydispersity. In simpler terms, a higher PI reflects less uniformity in particle size [12].

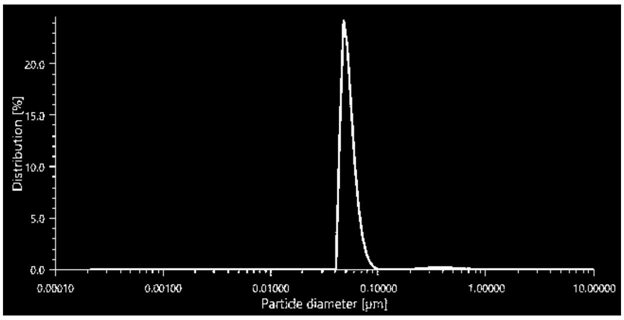

Fig. 1. DLS of Cellulose Nanocrystalline of Durian Husk.

3.1.2 FTIR Analysis

FTIR is a spectroscopic technique used to analyze the chemical structure of powdered durian husk during pre-treatment, alkaline treatment, bleaching, and acid hydrolysis. As shown in Figure 2, the spectra from each extraction stage display a broad absorption band between 3440–3475 cm^{-1}, indicating hydrogen bonding. This corresponds to O–H stretching vibrations, characteristic of cellulose [13,14]. The presence of this band confirms the successful removal of lignin and hemicellulose while preserving cellulose [15].

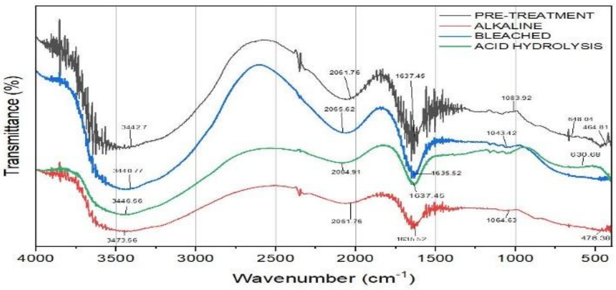

Fig. 2. FT-IR Spectra of the Samples: (a) Pre-treatment, (b) Alkaline, (c) Bleaching, (d) Acid Hydrolysis.

3.2 Composite Film Evaluation

3.2.1 Mechanical Property – Elongation at Break

Elongation at break (EAB) measures the maximum length change before a film breaks, reflecting its average flexibility [16]. Figure 3 shows that the biocomposite film with 5% CNC exhibited the highest EAB. The EAB increased as CNC concentration increased, but at 7% CNC, it decreased, though still higher than at 0% CNC. Romadhan and Sanjaya [9] attributed similar EAB decreases to chitosan, which reduces the intermolecular bond distance. However, in this study, the chitosan amount was constant across all treatments. The decrease in EAB at 7% CNC may be due to increased intermolecular interactions between starch and CNC, which created a more rigid structure. This excess CNC limited the deformability of the biocomposite, reducing its EAB. Khan et al. [19] noted that excessive CNC incorporation restricts matrix motion, decreasing EAB. Talebi et al. [20] and Iriani et al. [18] also found that 5% CNC loading optimizes EAB, improving stress transfer and distribution in the film.

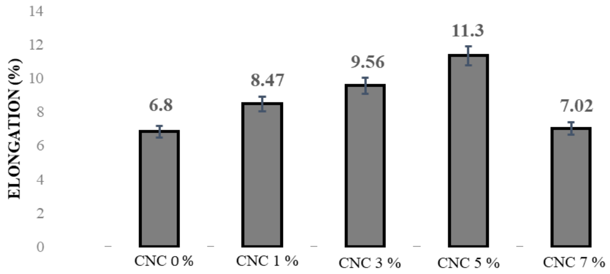

Fig. 3. Elongation at Break (EAB) of the Biocomposite film with different concentrations of Durian Husk CNC.

3.2.2 SEM-EDX Analysis

SEM analysis reveals the morphological changes and effects of biopolymer combinations on the surface microstructure of the composite film. The film with the best mechanical properties, incorporating 5% CNC, was selected for SEM analysis. Figure 4 displays the film's surface at different magnifications. At lower magnification (Figure 4, top), the surface shows roughness and randomly distributed small bubbles, which result from the addition of cassava starch and chitosan. This finding aligns with Sangian et al. [21], who observed a similar rough surface when using cassava starch. CNC appears as light crystalline shapes, randomly distributed across the surface. The addition of chitosan and CNC alters morphology, creating an uneven surface with visible CNC lumps, as noted in previous studies by Romadhon and Sanjaya [9]. Johar and Amad [22] also observed randomly distributed white domains in films containing CNC. Talebi et al. [20] reported CNC as white dots, indicating strong linkages within the film's texture. Higher magnification (Figure 4d) reveals more visible CNC crystals. Several studies, including Talebi et al. [20], confirm that CNC's excellent dispersion and compatibility with chitosan enhance the film's mechanical properties, as nanocellulose forms strong interactions with chitosan powder [23].

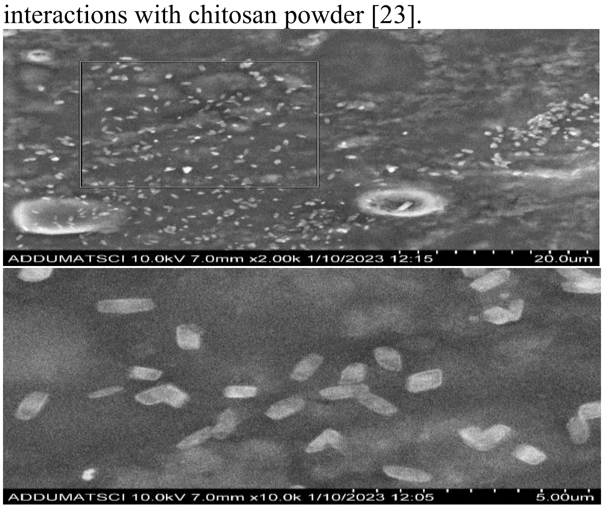

Fig. 4. SEM micrographs for the surface area of biocomposite film with 1000x (top) and 10000x (bottom) magnification levels.

3.2.3 Energy Disperse X-ray (EDX) Analysis

EDX analysis identifies the chemical elements and their concentrations in the material [24]. The results show that carbon (46.08%) and oxygen (53.92%) are the primary components, as seen in the EDX spectrum (Figure 5). These elements are common in the natural polymers of durian shells. Similar findings were reported by Taer et al. [25], who used durian shells as activated carbon, and by Kusrini et al. [26], who identified C (60.31%), O (28.08%), and N (3.06%) in durian husk. The detected elemental composition confirms the presence of polymer material [27]. Malhotra et al. [28] highlighted cellulose as a natural polymer used in film production, making it a promising source for biodegradable packaging. These results suggest that CNC derived from durian husk is a viable material for packaging. Additionally, the letter "K" refers to the shell energy level in electron configuration.

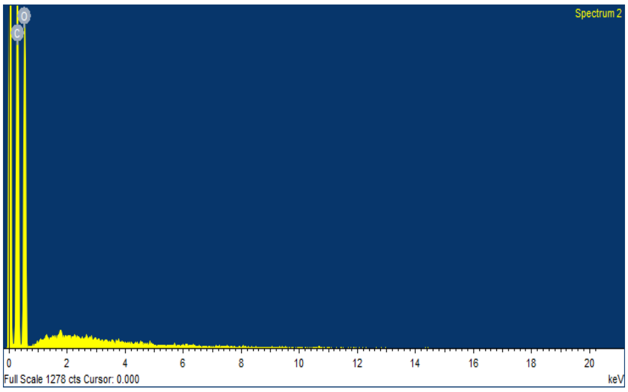

Fig. 5. Energy Disperse X-ray Spectrum of the biocomposite film CNC 5%.

3.2.4 Thermogravimetric Analysis

Thermogravimetric (TG) analysis assessed the thermal stability and degradation profile of the biocomposite film [23]. This technique measures mass changes due to temperature-induced effects such as evaporation, oxidation, and degradation. The film with 5% CNC, chosen for its superior mechanical properties, was analyzed. As seen in the TGA curve (Figure 6), three thermal degradation stages occurred between 20 °C and 600 °C for 77 minutes. The first stage (below 100 °C) showed a 6.26% mass loss, mainly due to the elimination of water molecules [29]. The second stage (100–500 °C) experienced the highest degradation (54.11% mass loss), likely linked to the breakdown of chitosan, starch, glycerol, and CNC [29]. Xu et al. [30] noted CNC decomposition occurs between 240–335 °C, chitosan between 140–310°C, and glycerol between 160–200 °C [31]. The third stage (500–600 °C) showed a 13.65% mass loss, attributed to carbonaceous material breakdown and gas production [32,33]. These results suggest that the film maintains thermal stability, making it suitable for packaging applications.

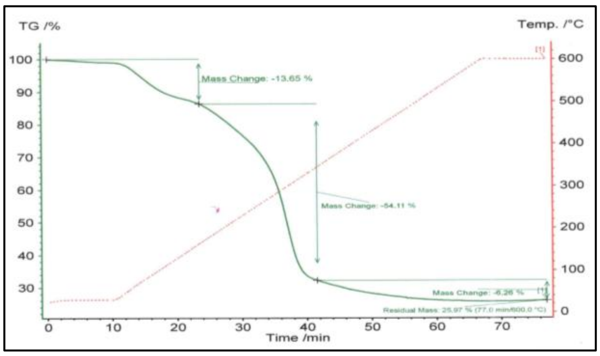

Fig. 6. TGA curve of the biocomposite film CNC 5%.

3.2.5 Water Absorption

Water absorption refers to the percentage of water absorbed by a material at a specific time and temperature [34]. This property is crucial for packaging, as water plays a significant role in deteriorative reactions [35]. Materials with high water absorption tend to biodegrade faster [36] but are also more prone to mechanical failures [37]. In this study, the water absorption of biocomposite films was evaluated, and the results are shown in Figure 7. The films were treated with varying concentrations of CNC (0%, 1%, 3%, 5%, and 7%). Water absorption was measured over 1 to 6 hours, with CNC 0 absorbing more water than the other treatments.

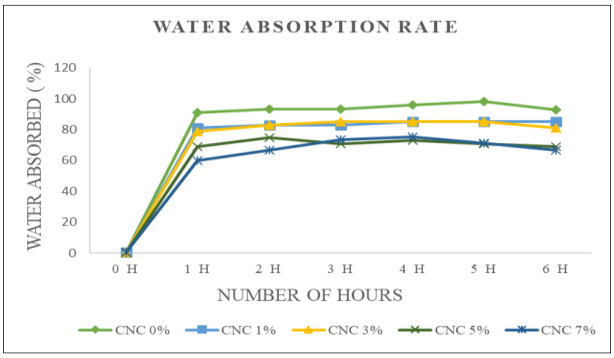

Fig. 7. Water Absorption rate of films at different concentration of CNC from 1 to 6 hours of test.

The presence of cellulose nanocrystals (CNC) in films significantly reduces water absorption. Despite cellulose's many hydroxyl groups, it doesn't easily dissolve in water due to strong hydrogen bonding between its units. As more CNC is added, water absorption decreases. The low water absorption in films with higher CNC concentrations suggests that CNC is a good reinforcement agent for developing packaging films with reduced water absorption. Halimatul et al. [38] noted that factors like increased crystallinity and good compatibility between the matrix and reinforcement contribute to reduced solubility and water absorption. Thus, CNC derived from durian husk shows strong potential as a reinforcement agent for packaging films.

3.2.6 Soil Biodegradation

The biodegradability test, following ASTM D5988 standards (Soil Burial Degradation Test), assessed the degradation of biocomposite films in different soil types. Results revealed that longer burial times led to higher degradation rates, with weight loss correlating directly to deterioration. Films with 0% and 1% CNC degraded faster in both organic soil (9 days) and normal soil (12 days) than other treatments, suggesting they absorbed more water. According to Ilyas et al. [39], faster degradation is due to higher water absorption in films with less CNC, making them more vulnerable to microbial attack [40]. This finding aligns with Ilyas et al.'s study, where sugar palm starch films degraded faster than bio-nanocomposite films, attributed to water absorption and crystallinity. Additionally, biocomposite films in this study showed a mass reduction of over 50% after 6 days of burial, indicating they are easily digested by microbes [41]. Films in organic soil degraded more rapidly, likely due to a higher concentration of microorganisms. As noted by Maran et al. [43], starch and glycerol are more susceptible to microbial degradation due to their hydrophilic nature.

3.2.7 Antibacterial Activity Testing

The antibacterial activity was evaluated by measuring the inhibition zone diameter around the sample. Figure 8 shows the lack of inhibition growth on Petri dishes with both gram-positive and gram-negative bacteria after incubation with the sample films. Only positive control exhibited an inhibition zone. This indicates that the biocomposite film does not exhibit antimicrobial properties. Some studies suggest that the lack of antimicrobial effect is due to the concentration and type of chitosan used. While Chitosan alone has strong antimicrobial activity, its effectiveness is reduced when incorporated into a film matrix, as the interaction between the Chitosan and microbial cell walls is diminished [44].

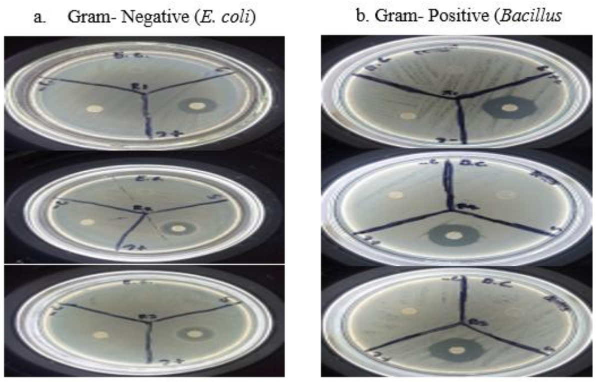

Fig. 8. Antibacterial Activity of the biocomposite film using the Disk Diffusion Method in (a) Negative gram bacteria (*E. coli*) (b) Positive gram bacteria (*Bacillus cereus*).

Siripatrawan and Vitchayakitt [45] reported a lack of antimicrobial activity in various Chitosan films, showing no inhibition against S. aureus, E. coli, or Pseudomonas aeruginosa. Similar findings were noted by Foster and Butt [44], where Chitosan films did not inhibit E. coli, S. aureus, or S. epidermidis. Sondari et al. [46] also found that films with cassava starch and chitosan showed no inhibition after 24 and 48 hours against both gram-positive and gram-negative bacteria. Jannatyha et al. [47] highlighted that chitosan exhibits different antibacterial mechanisms for gram-positive and gram-negative bacteria. Additionally, Agustin & Padmawijaya [48] noted that glycerol reduces the antimicrobial activity of chitosan films. While antimicrobial properties in packaging are desirable to extend shelf life and prevent microbial growth [49, 50], the present study found no inhibition, suggesting that not all food packaging materials require antibacterial properties.

4. CONCLUSION & RECOMMENDATIONS

This study successfully isolated CNC from durian husk, confirmed by DLS and FTIR analyses. The extracted CNC was used as a reinforcing agent in biocomposite films. The film with the best mechanical properties was further analyzed using SEM-EDX and TGA. SEM revealed changes in morphology and CNC distribution, while EDX confirmed the biopolymers used in film development. TGA showed the film's thermal stability, indicating its potential as packaging material. Water absorption significantly impacted the films' biodegradability and mechanical properties; films with higher absorption rates degraded faster and were more prone to mechanical failure. The films also exhibited consistent color properties. While the films showed promising potential as packaging material, they lacked antibacterial properties. Moreover, Alvarado et al. [51] suggested that for nanocellulose extraction and innovation studies, the researchers must consider assessing the economic feasibility of the technology by employing a technoeconomic analysis (TEA). With this consideration, agricultural by-products will be more valorized properly and will provide baseline data for their marketability [52].

Hence, the study recommends: (a) using a Textural Profile Analyzer to assess mechanical properties; (b) selecting an appropriate method to examine the films' color properties; (c) exploring additives that provide antibacterial activity; (d) investigating techniques to prevent bubbles in the film; and (e) evaluating additional parameters for packaging materials.

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