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Application of Carbon Nanodots from Cocoa Husk Waste to Improve the Mechanical Properties of Cellulose-Carrageenan-Based Biodegradable Films

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Abstract

Biodegradable films made from natural materials such as cellulose and carrageenan are easily degradable but exhibit poor mechanical properties. Incorporating carbon nanodots (CDs) from cocoa husk waste has the potential to improve their mechanical properties. This study aimed to characterize CDs synthesized from cocoa husk waste and evaluate their mechanical properties of cellulose-carrageenan-based biodegradable films. The research stages include the isolation of cocoa husk cellulose, CDs synthesis, CDs characterization, biodegradable film preparation, and characterization of the mechanical properties of biodegradable films. The experimental design used was a Completely Randomized Design (CRD) with the treatment factor of CDs addition at six levels (0%, 1%, 3%, 5%, 7%, 9%) with three replicates. The results showed that the average size of cocoa husk CDs was 997 nm, and the absorbance peak was detected at 298 nm, validating that the CDs had been successfully synthesized with blue light emission. Incorporating CDs significantly affected the mechanical properties of the biodegradable film. Biodegradable cellulose-carrageenan-based films with the addition of CDs from cocoa husks had a thickness of 0.35–0.64 mm, tensile strength of 2.94–4.20 MPa, elongation of 36.51–63.28%, and elasticity of 6.00–9.45 MPa. The tensile strength and elongation values meet the JIS Z 1707 standard. Higher concentrations of CDs significantly improved tensile strength and elasticity, while reducing the thickness and elongation.

Keywords: biodegradable film; carbon nanodots; carrageenan; cellulose.

INTRODUCTION

Plastic has become the most widely used material across all levels of society. According to the latest 2024 report from the Ministry of Environment and Forestry of Indonesia (KLHK), the country generates approximately 33 million tons of waste annually from daily human activities. Within this total, plastic accounts for 19.76% of waste composition, making it the second largest contributor after food waste. More than 40% of this waste is poorly managed, creating a significant risk of environmental pollution. The accumulation of unprocessed plastic waste, in particular, poses serious threats to ecosystems (Putra et al., 2025). Efforts to mitigate waste accumulation include reducing single-use plastic consumption, promoting recycling initiatives, and substituting conventional plastics with biodegradable materials (Sari et al., 2023).

Biodegradable films are thin layers derived from natural polymers that can be decomposed by microorganisms into simpler products such as CO_2, CH_4, H_2O, and energy. The degradation process involves water molecule penetration into the material, cleavage of covalent bonds, and the enzymatic breakdown of cellulose and hemicellulose (Safira & Purbasari, 2022). These films can serve as edible coatings for products such as sausages, meat, and fruits, or as light protective layers for various applications (Rachmawati et al., 2023). The base materials commonly used in biodegradable films are polysaccharides, including starch, cellulose, and lignin, selected depending on the intended application. Among these, cellulose-based films are preferred due to their superior film-forming ability. To enhance the performance of biodegradable films, additional materials such as alginate, chitosan, and carrageenan are often incorporated during the thickening process. Carrageenan is widely applied as an emulsifier, thickener, stabilizer, and food coating material. When dissolved in water, carrageenan forms gels and exhibits strong thickening properties, thereby facilitating biodegradable film

production (Zanjabila et al., 2023). However, despite these advantages, cellulose- and carrageenan-based biodegradable films have limitations such as low elasticity and poor water resistance. Reinforcing agents are therefore required to improve their mechanical properties.

Carbon nanodots (CDs) are carbon-based nanomaterials known for their biocompatibility, strong fluorescence, water solubility, low toxicity, and favorable reflective properties. Incorporating CDs into biodegradable films has been shown to significantly enhance their mechanical performance. For example, Millah and Dwandaru (2024) reported that the addition of CDs synthesized from corn husk waste increased tensile strength by up to 217% and improved the water resistance of corn starch-based edible films. Similarly, Sari et al. (2023) demonstrated that CDs enhance thermal stability and prevent the degradation of organic components in films. CDs can be produced through heat treatment of organic feedstocks, including empty fruit bunches of palm oil, peanut shells, and sugar (Koshi et al., 2021). A promising source of organic feedstock for CDs is agro-industrial waste, particularly cocoa husks.

Cocoa fruit generally consists of 75% husk, 22% bean shell, and 3% placenta. The large proportion of cocoa husk presents an opportunity for its valorization into sustainable materials such as CDs. Cocoa husks are rich in lignocellulosic components, containing approximately 35.4% cellulose, 37% hemicellulose, and 14.7% lignin (Sena et al., 2021). Lignocellulose derived from agricultural residues serves as an abundant carbon source and an effective precursor for CDs synthesis (Lee & Ko, 2025). Furthermore, lignocellulosic components are known to enhance the strength of polymer-based materials. Thus, the utilization of cellulose from cocoa husks represents a promising pathway for CDs production.

Research on the use of cocoa husks as a precursor for CDs remains limited, and their application in improving the mechanical properties of biodegradable films has not been extensively explored (Nazibudin, 2022; Norman et al., 2022). Given the high lignocellulose content of cocoa husks, they are an ideal candidate for CDs synthesis. Therefore, the objective of this study was to characterize CDs synthesized from cocoa husk waste and to investigate the effect of CDs incorporation on the mechanical properties of cellulose-carrageenan-based biodegradable films.

MATERIAL AND METHODS

Materials and Equipments

Cocoa husk waste was obtained from cocoa farmers in Sukoharjo 1 village, Pringsewu district, Lampung. Additional materials included distilled water, deionized water, NaOH, H_2O_2, carrageenan (INDOgum), ethanol, acetic acid, and glycerol. The equipment used comprised standard chemical glassware, Petri dishes, a grinder, an oven (Memmert), an 80-mesh sieve, a 250-mesh nylon filter, a hydrothermal autoclave, an ultrasonic machine, a centrifuge, a particle size analyzer (Beckman Coulter), an X-ray diffractometer (PANalytical), a Genesys 10 UV-Vis spectrophotometer, a digital micrometer (RoHS), and a Universal Testing Machine (Zwick Roel).

Experimental Design

The experimental procedure consisted of cellulose isolation from cocoa husk, synthesis and characterization of carbon nanodots, preparation of biodegradable films, and evaluation of their mechanical properties, including thickness, tensile strength, elongation, and elasticity. The study employed a completely randomized design with a single treatment factor, namely the addition of carbon nanodots at six levels (0%, 1%, 3%, 5%, 7%, and 9%), each replicated three times.

Isolation Celluse of Cocoa Husk

Cellulose isolation from cocoa husk waste was carried out following the method of Sari et al. (2023). One kilogram of cocoa husk was washed with water, sliced into approximately 1 cm pieces, and dried in an oven at 65°C for 3 h. The dried husks were then ground for 5 minutes and sieved through an 80-mesh sieve to obtain fine powder. The powder was subjected to delignification by heating in 1 L of 12% NaOH solution for 2 hours. The mixture was filtered, and the residue was washed thoroughly to yield crude cellulose. Bleaching was performed by heating the cellulose in 300 mL of 3% H_2O_2 at 100°C for 1 h. The product was then filtered, washed, and dried at 65°C for 3 hours, resulting in cellulose powder from cocoa husk waste.

Synthesis of Carbon Nanodots

Carbon nanodots were synthesized following Marphongahtun et al. (2023) with modification of the carbon source. One gram of cocoa husk powder was homogenized with 50 mL of deionized water and transferred into a hydrothermal autoclave, where it was processed for 6 h at 200°C. The resulting product was centrifuged at 3000 rpm for 30 min. The black precipitate was separated, and the supernatant was filtered and stored for further characterization. The supernatant contained the synthesized carbon nanodots, which were characterized using particle size analysis (PSA), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and UV-Vis absorbance spectroscopy.

Characterization of Carbon Nanodots

The synthesized carbon nanodots were analyzed using PSA to determine particle size distribution and average diameter at the nanometer scale. FTIR spectra were used to identify functional groups based on characteristic absorption peaks. XRD was employed to determine diffraction patterns, phase characteristics, and

crystallinity. UV-Vis absorbance spectroscopy was conducted in the range of 200–600 nm to evaluate light absorption properties.

Preparation of Biodegradable film

Biodegradable films were prepared using a solution casting method adapted from Koshy et al. (2021). Six grams of cellulose, previously isolated from cocoa pod husk, were dissolved in 45 mL of acetic acid at 60°C for 25 minutes. The mixture was then combined with 100 mL of distilled water, 5 mL of glycerol, 4 g of carrageenan (kappa), and carbon nanodots at the designated treatment levels. The solution was stirred at 70°C for 25 minutes, poured into glass plates, and air-dried at room temperature for 2 days, followed by oven drying at 60°C for 16 hours. The resulting films were peeled off and subjected to mechanical testing, including thickness, tensile strength, elongation, and elasticity (ISO, 2019**).**

Characterization of the Mechanical Properties of the Biodegradable Film

Mechanical properties of the films were characterized according to ISO (2019). Thickness was measured at five different points using a digital micrometer, and the results were expressed in millimeters. Tensile strength, elongation, and elasticity were determined using a Universal Testing Machine with 2 × 7 cm film specimens. Tensile strength was expressed in MPa, elongation in percentage, and elasticity was calculated as the ratio of tensile strength to elongation, expressed in MPa.

Data Analysis

Data obtained from this study were analyzed both statistically and descriptively, depending on the parameter. Mechanical property data were expressed as mean values with standard deviations and analyzed statistically using one-way ANOVA, followed by Duncan's multiple range test at a significance level of α = 0.05. Statistical analysis was performed with SPSS version 25. Characterization data from PSA, FTIR, XRD, and UV-Vis spectroscopy were analyzed descriptively.

RESULTS AND DISCUSSION

Particle Size Analysis (PSA)

Particle size analysis was conducted to determine the distribution of particle sizes in the carbon nanodots (CDs) samples. This technique can be performed using various approaches, including laser diffraction, microscopy, and sedimentation (Putri, 2023). In this study, 2 mL of CDs suspension was analyzed, and the numerical data were visualized in the form of a particle size distribution graph, as shown in Figure 1. The analysis revealed that the average particle size was 997 nm, with a range from 811.1 nm to 3125 nm and a polydispersity index (PI) of 0.401. These values indicate that the product did not fall within the conventional size range of carbon nanodots. According to Khan et al. (2021), CDs are typically defined as carbon nanoparticles with average sizes below 10 nm and exhibiting either spherical or sheet-like morphology. Similar findings were reported by Kristian & Wahyuni (2022), who observed particle sizes between 38.75 nm and 51.8 nm for CDs synthesized from tangerine peel waste, which likewise exceeded the conventional sub-10 nm definition. Particle size in CDs is strongly influenced by factors such as the synthesis approach (top-down or bottom-up), precursor type, and reaction conditions including temperature, duration, and pH. Generally, top-down techniques tend to yield smaller nanoparticles, whereas precursor composition and reaction parameters play a critical role in determining final CDs dimensions (Mocci et al., 2022).

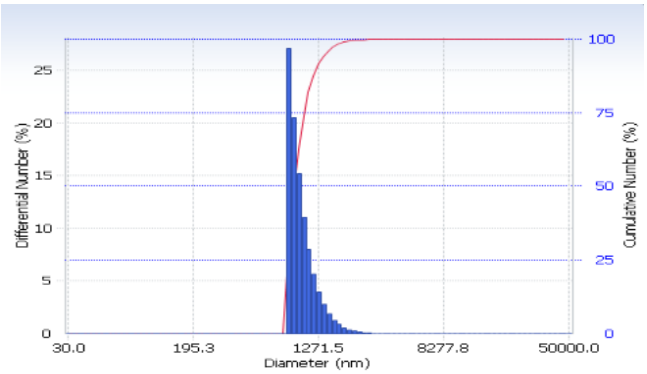

Figure 1. Particle Size Analysis test results.

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was employed to identify molecular vibrations and predict the chemical structure of the CDs samples (Sanjiwani et al., 2020). The FTIR analysis revealed three major absorption peaks at 3257 cm^{-1}, 2190 cm^{-1}, and 1640 cm^{-1} (Figure 2). The broad peak at 3257 cm^{-1} corresponds to O–H stretching vibrations, confirming the presence of hydroxyl groups in the molecular structure (Adu et al., 2022). The peak at 2190 cm^{-1} is attributed to triple-bond vibrations (C≡C or C≡N), indicating the presence of alkynes or nitrile groups. The band at 1640 cm^{-1} is associated with C=C or C=O stretching, suggesting the existence of carbon–carbon or carbon–oxygen double bonds, which are characteristic of carbon-based nanostructures. These results are consistent with those reported by Yudhanto & Dwandaru (2024), who identified similar functional groups, including O–H, C=C, and C≡N, in CDs derived from jackfruit seed waste. The agreement in functional group distribution confirms the successful formation of characteristic CDs structures in this study.

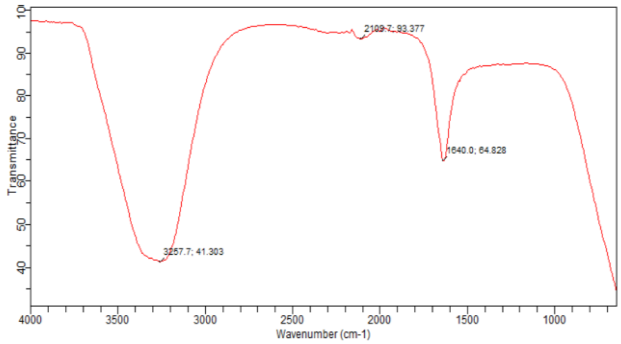

Figure 2. Fourier Transform Infra-Red test results.

X-ray Diffraction (XRD) Analysis

XRD analysis was conducted to investigate the crystalline structure and lattice parameters of CDs synthesized from cocoa husk (Millah & Dwandaru, 2024). The diffraction pattern obtained is shown in Figure 3, which presents the relationship between diffraction angle (2θ) and intensity. The diffraction pattern exhibited several peaks, with a broad main peak appearing around 2θ = 24°, which is typically associated with the interlayer spacing of graphitic structures. Additional peaks at approximately 2θ = 43° and 54° were also observed, which may correspond to crystalline phases originating from impurities or secondary structures within the sample. The broad nature of the primary peak indicates the presence of small crystallites with a predominantly amorphous structure. The crystallinity degree of the CDs sample was calculated as 11.42%, confirming that the material is primarily amorphous to semi-amorphous in nature. These findings are consistent with those of Millah & Dwandaru (2024), who reported that CDs derived from corn husk waste exhibited an amorphous structure, with broad diffraction peaks between 10° and 35° and no sharp crystalline peaks. Thus, the XRD results support the conclusion that CDs synthesized from cocoa husk predominantly display amorphous structural characteristics.

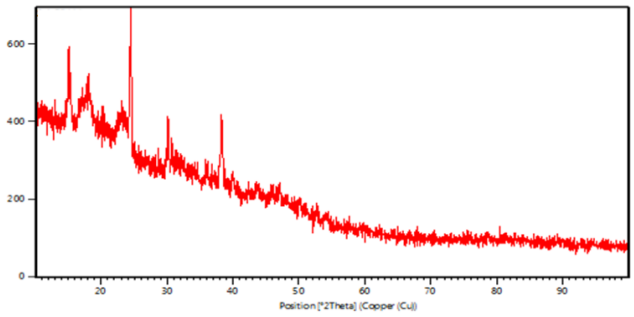

Figure 3. X-Ray Diffraction test result.

UV-Vis Spectrophotometer Testing

UV-Vis spectroscopy was carried out to determine the absorbance of the synthesized CDs in the ultraviolet (UV) and visible (Vis) regions at specific wavelengths (Dwandaru et al., 2020). The test was performed over a wavelength range of 200–600 nm, and the results are presented in Figure 4. The CDs synthesized from cocoa husk exhibited absorption peaks at 298, 310, 324, and 328 nm, with the highest absorbance recorded at 298 nm. The absorbance values were primarily observed at UV wavelengths below 380 nm. This finding is consistent with Sari et al. (2020), who reported that CDs synthesized from betel leaf exhibited absorption peaks between 260–360 nm. The red shift observed in cocoa husk CDs, indicated by the absorption peak shifting toward longer wavelengths, suggests an increase in particle size. A higher absorbance intensity also reflects the successful formation of CDs (Elfino & Dwandaru, 2022). Dwandaru et al. (2020) reported that CDs derived from namnam fruit displayed absorption peaks between 228–268 nm, while Sari et al. (2020) observed peaks between 257–320 nm for betel leaf CDs. These findings support the conclusion that the optical properties of CDs vary depending on precursor materials and synthesis conditions.

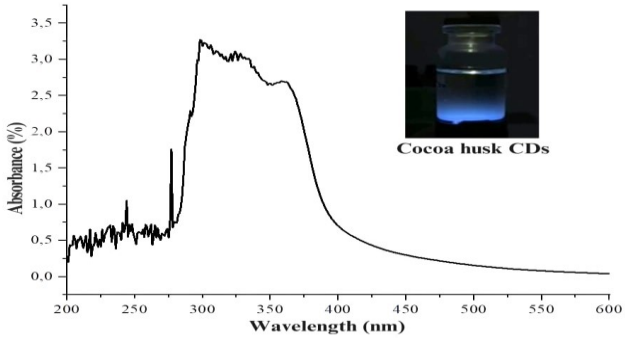

Figure 4. Spektrofotometer UV-Vis test result.

Mechanical Properties of the Biodegradable Film

The mechanical properties evaluated for the cellulose–carrageenan-based biodegradable film included thickness, tensile strength, elongation at break, and Young's modulus. The results are presented in Table 1.

Table 1. Mechanical characteristics of biodegradable film.

CDs concentration	Thickness (mm)	Tensile strength (MPa)	Elongation (%)	Young's modulus (MPa)
0%	0.63±0.04 a	3.59±0.06 b	63.28±2.42 a	5.67±0.41 a
1%	0.64±0.03 a	3.68±0.08 b	61.44±1.46 a	6.00±0.19 a
3%	0.35±0.02 d	4.20±0.13 a	44.55±2.01 c	9.45±0.59 b
5%	0.54±0.03 b	2.94±0.35 c	36.51±2.32 c	8.12±1.24 b
7%	0.43±0.04 c	3.51±0.06 b	41.11±1.00 b	8.53±0.12 b
9%	0.44±0.02 c	3.47±0.04 b	39.43±1.83 b	8.80±0.35 b

Note: Different letters following the numbers within the same column indicate statistically significant differences according to Duncan's test at the 5% significance level.

Film thickness is a critical parameter for determining the structural integrity and quality of biodegradable films in packaging applications (Khotimah et al., 2022). Table 1 shows that the maximum thickness (0.64 mm) was obtained with 1% CDs, while the minimum thickness (0.35 mm) was observed with 3% CDs. These results indicate that increasing CDs concentrations tends to reduce film thickness. Zaky et al. (2021) noted that film thickness can also be influenced by the addition of plasticizers such as carrageenan and glycerol. However, the thickness values obtained in this study exceeded the JIS Z 1707 standard, which specifies a maximum of 0.25 mm for packaging materials (Japanese Standards Association, 2019).

The tensile strength values in Table 1 demonstrate a significant effect of CDs incorporation. The addition of 3% CDs yielded the highest tensile strength (4.20 MPa), significantly higher than the control (3.59 MPa). Although the tensile strength decreased slightly at 5% CDs (2.94 MPa), an increase was again observed at 7% CDs (3.51 MPa), which remained significantly higher than at 5%. This suggests that CD incorporation up to 3% strongly reinforces the polymer matrix, while higher concentrations may cause aggregation before partially stabilizing again. These findings align with Millah & Dwandaru (2024), who reported increased tensile strength with CDs addition. Suheti (2022) attributed such improvements to enhanced stress transfer between polymer chains and CDs nanofillers. The tensile strength values obtained in this study satisfy the JIS requirement for packaging materials, which sets a minimum tensile strength of 0.392 MPa.

Elongation at break reflects the flexibility of films under tensile stress. The results (Table 1) show that the highest elongation (63.28%) occurred in the control film, while the lowest (36.68%) was recorded for 5% CDs. This inverse relationship between tensile strength and elongation indicates that CDs incorporation increases rigidity while reducing flexibility. Similar trends were reported by Millah & Dwandaru (2024), who observed that CDs enhanced tensile strength but reduced elongation. Changes in the mechanical properties of the biodegradable film occur due to molecular-level interactions between the surface of nanofillers and the polymer matrix (Roy & Rhim, 2021). According to JIS standards, elongation values above 50% are considered good, while values below 10% are considered poor. Based on this criterion, the elongation results in this study can be considered satisfactory.

Young's modulus, which represents film stiffness, also varied significantly with CDs concentration. The elasticity and strength of a material can be observed through the stress-strain curve or table obtained by performing tensile testing on the material (Sakti et al., 2023). The results that showed at the Table 1, highest value (9.45 MPa) was recorded at 3% CDs, while the control film exhibited the lowest value (5.67 MPa). These results confirm that CDs incorporation enhances film stiffness, with higher modulus values corresponding to reduced elasticity. This finding is consistent with Suheti (2022), who reported that CDs nanofillers improve mechanical rigidity by strengthening interactions between fillers and polymer chains. A similar trend was observed by Millah & Dwandaru (2024), where the addition of CDs from corn husk waste increased the modulus of edible films from 11.40 MPa to 22.53 MPa.

CONCLUSIONS

Carbon nanodots (CDs) were successfully synthesized from cocoa pod husks, as evidenced by their characteristic blue fluorescence, an average particle size of 997 nm, the presence of –OH, C=C, and C≡N functional groups, and a main XRD diffraction peak at 2θ = 24°. The incorporation of these CDs into cellulose–carrageenan biodegradable films produced thin layers with thickness values ranging from 0.35 to 0.64 mm, tensile strength of 2.94–4.20 MPa, elongation at break of 36.51–63.28%, and Young's modulus of 6.00–9.45 MPa. Increasing CDs concentration generally enhanced the tensile strength and Young's modulus of the films, demonstrating their potential as functional nanofillers for improving the mechanical performance of biodegradable packaging materials.

Authors' Contributions: Esa Ghanim Fadhallah, Ahmad Sapta Zuidar, and Teguh Setiawan designed the study.

Fitri Nuraini Fadila, Jihan Allya Syahar, Hersan Pratama Ashari Precillia Regitha and Fathan Khanifadin carried out the laboratory work and analyzed the data. Gustaf Triyoga, Wulan Nur Aisyah, Dea Meranda, and Zaskia Rahma Andini wrote the manuscript in Bahasa. Fitri Nuraini and Gustaf Triyoga translate to English. Esa Ghanim Fadhallah proofread the manuscript. All authors read and approved the final version of the manuscript.

Competing Interests: The authors declare that there are no competing interests.

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