BIO Web of Conferences **165**, 09002 (2025) https://doi.org/10.1051/bioconf/202516509002
*ICGAB 2024*

# Preparation and Characterization of Pectin-based Biopolymer from Combination of Coffee and Cocoa Husks as Agroindustrial Waste Utilization

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**Abstract.** Coffee and cocoa are among the largest commodities in Indonesia. During the processing, high amounts of these husks are generated as agricultural waste that contains pectin, which applicable to be used for biopolymers. The research aimed to 1) extract pectin from coffee and cocoa husks and 2) synthesize and characterize pectin-based biopolymers from both husks. Pectin extraction was done based on alcoholic precipitation methods. About 6.82% and 4.91% of pectin were extracted per 100 g of dried coffee and cocoa husk powder, respectively. FTIR has confirmed the extracted pectin from both husks. This study applied two compositional variations: pectin coffee biopolymer (CB) with a 1:0 ratio of coffee pectin to cocoa pectin, and combined pectin biopolymer from coffee and cocoa (CCB) with a 1:1 ratio. Thickness, lightness, and tensile strength of biopolymer variation CB were obtained at 0.16 mm; 28.76; and 0.00012 MPa, respectively. The thickness, lightness, and tensile strength of biopolymer variation CCB were 0.24 mm; 37.41; and 0.00014 MPa. The soil burial degradation test over 3 days, CB and CCB biopolymers showed respective biodegradability of 99.58% and 99.63%. These findings confirm pectin from coffee and cocoa husks can create valuable biopolymers, with further development required to expand applications

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# 1 Introduction

Indonesia is one of the top cocoa producers in the global with a production volume of 688,210 tons in 2021. This high productivity led to a significant export value of cocoa that reached up to 1.21 billion USD, highlighting its important contribution to the global cocoa trade [1]. Based on the composition, 75% of the whole fruit consists of cocoa pod shells or husks, while the rest is the beans as the most utilized part [2]. This could affect a significant amount of cocoa husk waste generated after the cocoa processing. Improper waste management practices of cocoa husks potentially cause environmental concerns, such as unpleasant odor [3]. Meanwhile, the cocoa husks are composed of valuable compounds, such as cellulose (35.4%), hemicellulose (37%), and lignin (14.7%) [4].

Indonesia is also one of the world's leading coffee producers with production reaching up to 786,190 tons in 2021 [5]. During the processing, a significant amount of coffee husk will be generated approximately 50 – 60% of the total production [3]. If the coffee husk is not properly utilized, this by-product can lead to environmental challenges. Meanwhile, the coffee husk is composed of cellulose (15.38%), hemicellulose (6.34%), and lignin (33.79%), as determined by chemical analysis [6].

These circumstances highlight that the cocoa and coffee husks have the potential to be further utilized. Pectin can become a thickener, emulsifier, and gel formation agent [7]. A former study has employed significant work in developing biopolymers by utilizing pectin from durian rind waste [8]. Despite the rising trend of biopolymer research from agricultural waste, there is still a lack of innovation in integrating and combining two natural resources derived from by-product processing for developing biopolymers. Thus, the objectives of this study were 1) to extract pectin from coffee and cocoa husks and 2) to synthesize and characterize pectin-based biopolymers from coffee and its combination with cocoa husks. This can be one of the promising alternative solutions to address plastic pollution issues in Indonesia that urgently require further attention. Since plastics are non-degradable materials and are quite challenging in natural decomposition and environmental pollution, this innovation highlights the urgency for substituting plastic materials with other biodegradable alternatives and environmentally friendly solutions.

# 2 Materials and methods

## 2.1. Sample preparation

Robusta coffee (*Coffea canephora*) husk samples were collected two weeks after harvesting from the local farmers in Temanggung Regency – Jawa Tengah. Meanwhile, Cocoa (*Theobroma cacao*) husk samples were collected three days after harvesting from the local farmers in Gunung Kidul Regency, Special Region of Yogyakarta, Indonesia. Due to limited condition and the time constrains of harvesting commodities, sample collection period could not be conducted simultaneously. About 15 kg of each cocoa and coffee husks were collected from the areas. Afterward, both samples were cut into smaller pieces and dried on a cabinet dryer at 50ºC for 24 h. Then, the dried coffee and cocoa husks were ground into powder and sieved (Ø 40 mesh). Only the dried powder that passed the sieve was used for the next step.

## 2.2 Pectin extraction from coffee husks

Pectin extraction from the coffee husks was done by a modified method of [8]. A total of 100 g of dried coffee powder was mixed with 900 mL 1 M HCl. The solution was incubated in a water bath at 85 °C for 4 h. Then, the solution was filtered using cheesecloth and the filtrate was allowed to settle at room temperature for a while. Afterward, the filtrate was mixed with acidified ethanol (a mixed solution of 4% HCl and 96% ethanol in a ratio of 1:4) with a ratio of 1:1. The mixture was precipitated for 24 h and filtered through filter paper. The residues were washed twice with 96% ethanol. Then, the solution was precipitated and filtered through filter paper. The extracted pectin was obtained in the precipitate of the mixture and dried at 55 °C for 24 h. The percentage of pectin yield (%) can be determined by Equation 1.

$$Pectin\ yield\ (\%) = \frac{dried\ extracted\ \ pectin\ (g)}{initial\ weight\ of\ dried\ powder\ (g)}\ x\ 100\% \qquad (1)$$

## 2.3 Pectin extraction from cocoa husks

Pectin extraction from cocoa husks was done by a modified method of [9]. A total of 50 g of dried powder mixed with 4% citric acid solution. The solution was stirred at 95°C for 95 minutes and precipitated at room temperature for 24 h. After the precipitate was formed, it was filtered with filter paper. Then, the filtrate was mixed with 96% ethanol at a ratio of 1:2 and refiltered with filter paper. The obtained residue was heated at 55°C for 24 h. Pectin yield percentage was determined by Equation 1.

## 2.4 Fourier Transform Infrared (FTIR) Analysis

The dried extracted pectin was tested using FTIR Spectroscopy (Thermo Scientific Nicolet iS10) to identify its main characteristics by functional groups. This was meant as a qualitative analysis to confirm the matched spectra between the extracted pectin to the database. FTIR test was carried out at the Integrated Laboratory for Research and Testing Universitas Gadjah Mada.

## 2.5 Biopolymer synthesis

Biopolymer synthesis was done by following the method of Lestari et al. [8] with modifications. Two compositional variations were set to be applied in this study: 1) pectin coffee biopolymer (CB) with a 1:0 ratio of coffee pectin to cocoa pectin, and (2) combined pectin biopolymer from coffee and cocoa (CCB) with a 1:1 ratio, with two replications. For biopolymer synthesis of variation CB, a total of 0.3 g of pectin-based coffee husk was diluted in 10 mL of distilled water. The solution was heated and stirred with a magnetic stirrer at 75 °C for 30 mins. Meanwhile, 2% chitosan solution was prepared by dissolving 0.2 g of chitosan with 10 mL acetic acid and heating at 65 °C. Then, the pectin solution was mixed with 10 mL of chitosan solution, 0.4 mL of ethylene glycol, and 0.1 mL of glycerol. The mixture was stirred and heated at 75°C for 5 mins. Next, the mixture was poured into an acrylic molding and heated in an oven at 40°C for 24 h. The biopolymer sheet was removed from the mold and placed in a desiccator for 10 mins. Similar stages of biopolymer synthesis were applied to form variation CCB with the difference in compositional weight of dried pectin-based coffee and cocoa husks at 1:1 ratio.

### 2.6 Biopolymer characterization

The biopolymers from both variations were characterized by physical (thickness, color), mechanical (tensile strength), and biodegradability characteristics. The thickness and color were measured by using a thickness gauge and chromameter, respectively. The tensile strength test was performed according to the ASTM D882 method using a Universal Testing Machine. The soil burial degradation test was applied using a modified method [10] to measure biodegradability potential. Biopolymer samples (2.5 cm x 15 cm in size) were buried in humus soil at a depth of 10 cm using polybag media and placed at room temperature (25°C) for 7 days with sampling times every 3 days. Weight loss as a biodegradability potential indicator can be calculated using Equation 2. All characterization results were then compared with the Indonesian National Standard (SNI) 7188.7:2016 (Ecolabel Criteria - Part 7: Product Category Plastic Shopping Bags and Biodegradable Bioplastics).

$$Weight\ loss(\%) = \frac{initial\ weight\ (g) - final\ weight(g)}{initial\ weight(g)} \times 100\% \quad (2)$$

## 3 Result and Discussion

### 3.1. Pectin extraction

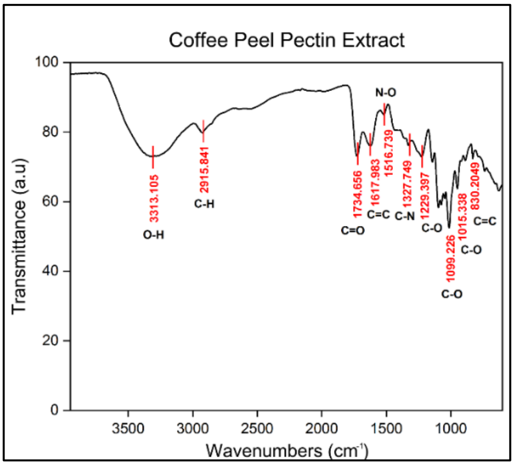
Pectin yield from coffee and cocoa husks was obtained 6.82% and 4.91% on average, respectively (Table 1). The results showed a difference in pectin yield percentage, while the pectin-based coffee husks showed a higher yield percentage than the cocoa ones. This condition could be affected by the water content from the raw material. Lower water content in the material could cause the extraction process to become more difficult as the solids are not dissolved in the water. Based on the results, the pectin from coffee husks can be obtained in dried powder. Meanwhile, the pectin from cocoa husks was still in the form of clumpy solids. This might be caused by lower water content in the cocoa powder than in the coffee. This becomes challenges during the extraction process which need more solvent and longer extraction time to obtain a higher yield of pectin.

**Table 1.** Results of pectin extracted from coffee and cocoa husks

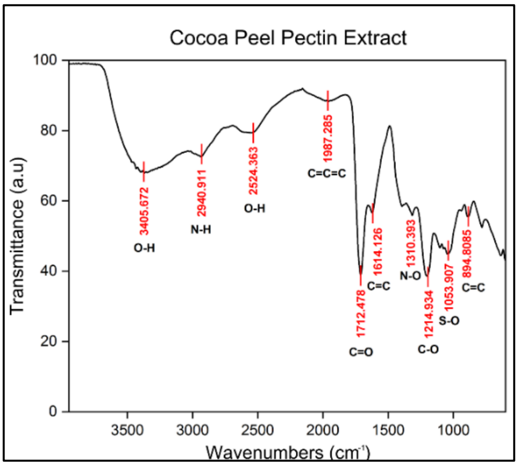
| Material | Repetition | Dry weight (g) | | Pectin yield (%) | Average pectin yield (%) |
|---|---|---|---|---|---|
| | | Dried husk powder | Extracted pectin | | |
| Coffee Husk | 1 | 100 | 6.13 | 6.13 | 6.82 ± 0.69 |
| | 2 | 100 | 7.51 | 7.51 | |
| Cocoa Husk | 1 | 100 | 5.70 | 5.70 | 4.91 ± 0.79 |
| | 2 | 100 | 4.12 | 4.12 | |

In addition, the extracted pectin from coffee and cocoa husks was identified using FTIR spectra (Figure 1). For the pectin-based coffee husk, the spectra showed intense peaks at 3313 $cm^{-1}$ (Figure 1a). Meanwhile, for the pectin-based cocoa husk, the spectra also showed intense peaks at 3405 $cm^{-1}$ (Figure 1b). These results are quietly similar to commercial pectin of cocoa husk, which has an absorption peak at wavelengths of 3323-3417 $cm^{-1}$ [11]. According to Hevira et al. [12], the presence of intense peaks at the wavelength ranges of 3200 – 3600 $cm^{-1}$ showed O – H functional groups. This indicated that there was strong bonding and vibration from the O - H functional groups from the extracted pectin spectra

(Figure 1), which indicated the presence of the pyranose ring as the main characteristic of pectin [13]. In addition, coffee and cocoa husk pectin peaks at wavelengths of 1735 $cm^{-1}$ and 1712 $cm^{-1}$, respectively, indicated the presence of carboxyl ester groups (C=O), while in the commercial pectin, a peak at a wavelength of 1747 $cm^{-1}$ was also obtained and indicated the presence of C=O [11].




(a)




(b)

**Fig. 1.** FTIR results of the extracted pectin from coffee (a) and cocoa husks.

## 3.2. Biopolymer characteristics

### *3.2.1 Physical and mechanical characteristics*

The results of physical and mechanical characteristics of biopolymers CB and CCB can be seen in Table 2. The appearance of biopolymer from pectin-based coffee husks and its combination with pectin-based cocoa husks can be seen in Figure 2. The thickness of the biopolymer combination of CCB (0.24 mm) was higher than the CB (0.16 mm), although both values met the standards (≤ 0.25 mm). Thickness is one of the most important properties of biopolymers due to their effectiveness as a product coating material. The thickness of bioplastics can affect other properties like tensile strength, elongation, and water resistance. The tensile strength of the biopolymer increases as it becomes thicker, but its elongation decreases [14]. In accordance, the water resistance of the biopolymer is expected to increase in correlation with the thickness and density of the sheet [15].

(a) (b)

**Fig. 2.** (a) Biopolymer CB (Pectin-based biopolymer from coffee husk) and (b) biopolymer CCB (the combination)

**Table 2.** Physical and mechanical characteristics of biopolymers

| Parameter | Unit | Standard* | Biopolymer | |
|---|---|---|---|---|
| | | | CB | CCB |
| Thickness | mm | ≤ 25 | 0.16 | 0.24 |
| Color: | - | - | - | - |
| L* (lightness) | - | - | 28.76 | 37.41 |
| a* (redness) | - | - | 9.07 | 13.18 |
| b* (yellowness) | - | - | 9.12 | 14.63 |
| Tensile strength | MPa | 24.7 - 302 | 0.00012 | 0.00014 |

*Note: * SNI 7188.7: 2016*

The higher thickness obtained at biopolymer CCB may result in a stronger and more water-resistant sheet than the CB. Meanwhile, the CB has higher elongation than the CCB due to its differences in thickness. The physical appearance of both polymers also showed color differences, as analyzed by lightness, redness, and yellowness (Figure 2, Table 2). The lightness value shows changes in the brightness with a range of values from 0 (black) to 100 (white) [16]. The lightness at biopolymer CCB was higher than the CB, which showed that the CCB was quite brighter. This could be affected by the pectin color produced from both sources. The color analysis results from a* and b* values showed a higher number in the biopolymer CCB than in the CB. This can be seen as the visual appearance of biopolymer CCB is brighter and more transparent than the CB, which showed better appearance (Fig. 2).

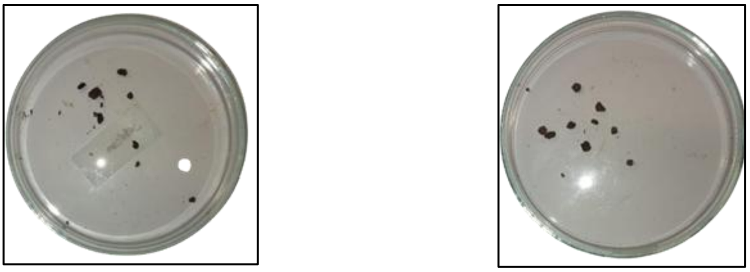
In addition, the tensile strength of the biopolymer CCB ($1.4 \times 10^{-5}$ MPa) was higher than the CB ($1.2 \times 10^{-5}$ MPa) (Table 2). This indicated that the combination of pectin may influence the tensile strength of the biopolymer. However, both tensile strength values did not meet the SNI standard (24.7–302 MPa). Former research by Dao et al. [17] demonstrated that biopolymers made from pectin-based coffee with the addition of microcrystalline cellulose (MCC) exhibited a tensile strength of 3 MPa. This may highlight that the presence of MCC as a texturizing agent, stabilizer, and filler may contribute to improving the mechanical characteristics of pectin-based biopolymers. Therefore, the tensile strength of CB and CCB biopolymers in this study could be enhanced by incorporating other additive agents, such as MCC. Meanwhile, CB and CCB biopolymers exhibit properties that make them promising candidates for use as edible coatings to extend the postharvest shelf life of fresh products. Additionally, the potency of bio-based packaging materials is gaining significant attention as sustainable alternatives compared to petroleum-based polymers [18].

### *3.2.2 Biodegradability*

The biodegradability for both biopolymer variations was tested using a soil burial degradation test to obtain gradual weight loss [19]. During the test, the samples were buried and placed in the soil layers to simulate environmental degradation [19]. Over three days, the biopolymer mass from both variations was decreased significantly (Table 3, Figure 3). In general, both pectin-based biopolymers showed biodegradability potency as the samples were able to be decomposed within 3 to 4 days under conditions of 50% relative humidity (RH) and a temperature of 25°C. This condition could be influenced by the principle of coffee and cocoa husks as biodegradable biomass content and can be decomposed in nature.

**Table 3**. Soil burial degradation test of the biopolymers

| Biopolymer | Initial Weight (g) | Weight loss at day 3 (g) | Biodegradability (%) |
|---|---|---|---|
| CB | 17.59 | 0.07 ± 0.36 | 99.58 |
| CCB | 17.26 | 0.06 ± 0.22 | 99.63 |



(a) (b)

**Fig. 3.** Results of soil burial degradation test over 3 days at (a) biopolymer CB and (b) biopolymer CCB

## 3.3. Comparison with previous studies

This research found that higher pectin yield was obtained from coffee husks (6.82%) than from cocoa husks (4.91%). This condition could be attributed to the differences in the method extraction process. Based on former research by Barrios-Rodriguez et al. [20], the pectin yiels varied for each method: water (2.0 - 23.3%), acid (1.3 - 11.73%), enzymatic (10.23%), ultrasound (8.3%), and microwave-assisted (1.93 - 42.0%). This variability may cause changes in pH, temperature, time, raw/solvent ratio, acid, enzyme concentration during the extraction.

In addition, the combination of both materials in the biopolymer CCB demonstrated physical properties enhancement compared to CB, including improved thickness (0.24 mm) and better visual characteristics with higher lightness values, which were potential advantages for certain applications that need better appearance and material integrity. Said et al. [21] stated that the combined pectin from different sources can lead to improving functional properties and enhanced viscosity and stability. Although the tensile strength of the biopolymers in this study still does not yet meet the standard requirements, the remarkable biodegradability performance (>99.5% within 3 days) showed promising potency for environmentally friendly products-based agricultural waste utilization, with future development.

## 4 Conclusion

Pectin yield from coffee and cocoa husks was obtained at 4.91% and 6.82%, respectively. Physical and mechanical characteristics of the biopolymers were obtained in thickness (0.16-0.24 mm), lightness (28–37), redness (9.07 – 13.18), yellowness (9.12 – 14.63), and tensile strength (1.2 - 1.4 × $10^{-5}$ MPa). Biodegradability showed 99.58 – 99.63% over three days test. The combination of pectin from coffee and cocoa husks enhanced the thickness, visual appearance, and tensile strength of the biopolymers. Future continuous development is needed to particularly improve the mechanical characteristics.

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