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Preparation and Extraction of Pectin from Robusta Coffee (*Coffea canephora*) Peel Waste as a Sustainable Agriculture Biomass

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Abstract. Indonesia is one of the top global coffee producers. In 2023, Indonesia produced high coffee productivity up to 760.2 thousand tons, primarily dominated by robusta coffee (*Coffea canephora*). However, this situation may raise significant environmental concerns regarding the disposal of coffee peel waste. About 0.18 tons of coffee peel waste is estimated to be generated per 1 ton of processed coffee beans, which needs proper waste management. Meanwhile, the coffee peel contains a valuable component of pectin that can be used in various applications to support circular economy and environmental sustainability. This study aimed to investigate the composition and characteristics of pectin extract from robusta coffee peel waste. Coffee peel sample was collected from Sambak Village, Magelang Regency, Central Java, Indonesia. The acid extraction method was applied to obtain pectin extract using a solvent of HCl (1 M) and alcoholic precipitation with ethanol (96%). About 6.23 ± 2.43 % of pectin yield on average was extracted per 100 g of dried coffee peel waste. FTIR test results confirmed the presence of functional groups of pectin characteristics at the peak points of 3320.46; 1731.69; and 164.92 cm^{-1}. These findings showed that the extracted pectin from coffee peel waste is one of the promising and sustainable ways to utilize agricultural biomass.

1 Introduction

High agricultural productivity is needed to address the increasing public demand for food supply. This practice has led to an increase in agricultural products, while also generating a significant amount of by-product waste [1]. Improper waste management could affect environmental concerns, including unpleasant odors, gas emissions from open combustion, and decaying compounds that may pollute the surrounding environment [2, 3]. Meanwhile, agricultural waste in the form of by-products of fruits, bark, roots, stems, and leaves can potentially be used as biomass resources [4].

Coffee is one of the significant commodities in Indonesia. In 2023, coffee production in Indonesia has reached 760.2 thousand tons, mainly produced in robusta coffee [5, 6]. However, during the processing, this may generate a significant amount of coffee peel waste, which accounts for 12% of the overall coffee beans. It was estimated that about 0.18 tons of coffee peel waste was generated per 1 ton of processed coffee beans [7]. The application and utilization of coffee peel waste are still limited to livestock feed and plant fertilizers directly without proper treatment [8]. Meanwhile, the composition of coffee peel waste comprised of cellulose (65.9%), hemicellulose (24.95%), lignin (0.21%), pectin (0.42%), protein (0.81%), tannins (1.05%), caffeine (0.09%), and polyphenols (0.81%) [9]. This highlighted the potential of coffee peel waste for supporting sustainable agriculture and circular economy.

Waste valorization is one of the strategic approaches to converting waste into added-value products [10]. Former studies have revealed some potential products from coffee peel waste valorization, such as biofuel, briquettes, fertilizers, and biosorbents [11]. In addition, the composition of coffee husk waste can be further utilized through the extraction process to create economically valuable products, such as biopolymers [12].

Pectin can be extracted from coffee peel waste, which is a potential and valuable polysaccharide compound [9]. Pectin is widely used as a stabilizer, thickener, gelling agent, and emulsifier in food processing up to the health sector [13]. This was due to pectin's superiority as a non-toxic compound, degradable capability, and gel formation in a simple mechanism [13, 14]. The main component of pectin is D-galacturonic acid polymer with a bond of α-1,4-glucoside and is widely found in the middle lamella of plant cells. Galacturonic acid comprises carboxyl groups that bind Mg^{2+} or Ca^{2+} ions leading to gel formation [15, 16]. The pectin is located in the primary cell wall of plants, which is between the cellulose and hemicellulose gaps causing an extraction process is needed to obtain pectin compounds and separate them from the other compositions [17].

Former studies of pectin extraction were generally conducted by acid method using an incubation process with acid solutions, such as HCl [18], HNO_3 [19], ammonium oxalate [20], or citric acid [21], which were then afterward purified with ethanol [19, 22]. However, only a few studies have provided limited information regarding pectin extraction from coffee peel waste using HCl as the primary solvent during the incubation process, meanwhile, the application of HCl has been extensively used in the pectin extraction from other biomass resources, such as durian rind [22], dragon fruit [18], and sweet potato [23]. Therefore, this study aimed to investigate the composition and characteristics of pectin extract from coffee peel waste as a potential biomass resource, which was focused on the extraction using HCl. This study is crucial as the extracted pectin from coffee peel waste can be applied as one of the promising solutions to solve improper agricultural waste management while also adding value to support circular economic development.

2 Materials and Methods

2.1 Sample preparation

Coffee peel sample was collected from Sambak Village, Kajoran District, Magelang Regency, Central Java, Indonesia. The sample was collected in dried condition after two months of harvesting and in the type of robusta coffee peel (*Coffea canephora*). The fresh sample condition could not be obtained at the time. First, the sample was dried using a cabinet dryer at 45ºC for 24 hours [24]. Afterward, the dried sample was ground and sieved (Ø 50 mesh) to obtain dried coffee peel powder (Fig. 1). Six repetitions were done to obtain the dried sieved powder (Table 1), which was then stored in a closed container.

(a) (b) (c)

Fig. 1. Sample preparation of coffee peel dried powder during a) drying, b) grinding, and c) sieving

2.2 Pectin extraction

Pectin extraction was carried out using modified acid methods of [22, 25] and combined with the experimental steps performed in this study (Fig. 2). First, a total of 25 g of the dried powder was mixed with 225 mL HCl (1 M), then incubated in a water bath for 4 h at 85ºC. Afterward, the results from incubation were cooled down at room temperature for a while and continued to be filtered using cheesecloth. Then, the filtrate was added to an acidic ethanol solution in a ratio of 1:1 for 24 h. Acidic ethanol is a mixture of HCl (4%, 1 M) and ethanol (96%) in a ratio of 1:24. After obtaining the precipitate from the solution, it was filtered and purified again using ethanol (96%) . Hereafter, the extracted pectin in solid form was separated and dried in an oven at 45ºC for 24 h. The percentage of pectin yield was calculated according to Equation 1 [26].

For process efficiency in this study, pectin extraction was conducted in six trials, using 100 g of dried powder for each trial and adjusting chemical solution volumes according to the methods of [22, 25]. The results from six extraction trials were sufficient to provide a representative depiction of the pectin content in the coffee peel.

$$Y\ (\%) = \frac{DP}{DC} \qquad (1)$$

Note:
Y = pectin yield (%)
DP = dried pectin (g)
DC = initial weight of dried coffee peel powder (g)

(a) (b)

Fig. 2. Pectin extraction during incubation process (a) and after alcoholic precipitation (b)

2.3 Fourier transform infrared (FTIR) analysis

FTIR spectroscopy is a method used to identify a compound based on its functional group [27]. The extracted pectin in dried solid condition was tested using FTIR Spectroscopy (Thermo Scientific Nicolet iS10) as a qualitative analysis to confirm that the transmittance of the extracted pectin corresponds with the pectin spectra in the standard database. FTIR test was done at the Integrated Laboratory for Research and Testing Universitas Gadjah Mada. In addition, the spectra results in this study were comparable to the former study which used standard pectin from Sigma-Aldrich [21].

3 Results and Discussions

3.1 Pectin extraction of coffee peel waste

Pectin extraction is meant to separate the active components from the coffee peel waste and obtain pure pectin in solid form [28]. Results of the extracted pectin were obtained in dark brown color, which derived from the color of the coffee skin used (Fig. 3). About a total of 37.4 g of extracted pectin was obtained from 600 g of dried coffee peel powder (50 mesh) (Table 1). The results indicated that, on average, about 6.23 ± 2.43 % of pectin was successfully extracted from every 100 g of dried coffee peel powder.

Fig. 3. Pectin extraction of coffee husk waste

Table 1. Results of pectin yield extraction.

Experiment	Dry Weight (g)			Pectin Yield (%)
	Dried coffee peel before sieving	Dried coffee peel powder passed 50 mesh	Extracted pectin	
1	300	100	10.5	10.50
2	300	100	4.2	4.20
3	300	100	4.5	4.50
4	300	100	4.8	4.80
5	300	100	5.8	5.80
6	300	100	7.6	7.60
Total	**1800**	**600**	**37.4**	**6.23 ± 2.43** [a]

Note: [a] *pectin yield percentage on average ± SD*

The average percentage of pectin yield (6.23 ± 2.43 %) from robusta coffee peel waste in this study (Table 1) was quite comparable to the results of former studies with different acid solutions as solvents (Table 2). The study of [29] conducted pectin extraction from coffee peel using citric acid / $HOC(CH_2CO_2H)_2$ with the addition of HCl and reported a pectin yield of 6.63%. Meanwhile, other studies applied HNO_3 [30] and $(NH_4)_2C_2O_4$ [20] obtained pectin yield of 8.65 ± 3.85 % and 21.87 ± 0.52 %, respectively. This condition might indicate the different acid solutions applied in the extraction process could affect the pectin yield results.

Table 2. Comparison of pectin yield results

No	Solvent	Pectin Yield (%)	Reference
1	HCl 1 M	6.23 ± 2.43	This study
2	$HOC(CH_2CO_2H)_2$	6.63	[29]
3	HNO_3	8.65 ± 3.85	[30]
4	$(NH_4)_2C_2O_4$	21.87 ± 0.52	[20]

In addition, the results of extracted pectin can also be affected by the solution's temperature, time, and pH during extraction [31]. Pectin acquisition during the extraction is initially triggered with a hydrolysis process by H^+ ions, separating protopectin with other compounds and then converted into pectin compounds [29]. On the other hand, the hydrolysis process that takes too much time can lead to pectin conversion to pectic acid [32]. In addition, the condition of the coffee peel obtained after 2 months of harvest can also affect the pectin extract yield. The storage period may influence the pectin composition in the coffee peel. During storage, pectin can undergo degradation through depolymerization and de-esterification processes, which may be attributed to enzymatic activity and changes in the chemical structure of pectin [33].

3.2 FTIR results of coffee peel extraction

FTIR Spectroscopy test aimed to validate the extracted pectin by characterizing the functional groups. The spectra result of the extracted pectin can be seen in Fig. 4, which showed important peaks at 3320.46, 1731.69, and 164.92 cm^{-1}. The peaks between 3296-3363 cm^{-1} may indicate the presence of an OH group from the pyranose ring, which is the main characteristic of pectin compounds [34]. The peaks between 1750-1638 cm^{-1} may correspond to the vibration of the stretch of the carbonyl functional group (C=O) in the methyl esterified carboxylate group ($-COOCH_3$) and the free carboxyl group (COO-). These peaks can indicate the degree of esterification in the pectin [35].

Former study showed similar spectra images from the extracted pectin of coffee peel waste and its comparation to the standard procured from Sigma-Aldrich [21]. The spectra images from [21] was quite aligned with the results in this study. Thus, the extracted pectin obtained in this study may valid as the pectin compound. Meanwhile, for further comparation, another study reported that coffee pectin consists of ester carbonyl groups (C=O), methoxyl groups, and glucose unit (OH, CH, CH_2) [36]. The chemical structure further illustrates the presence of that functional groups showed in Fig. 5 [29]. Additionally, the molecular weight (M_w) of extracted pectin was determined to be 3.921 x 10^5 g/mol [19].

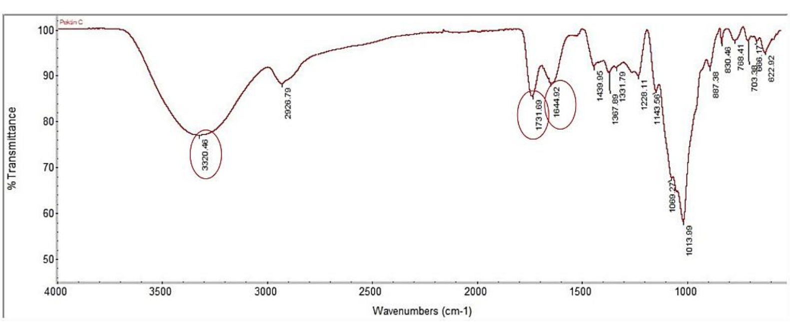

Fig. 4. Results of FTIR spectra from the extracted pectin of coffee peel waste in this study.

4 Conclusions

Pectin extraction from robusta coffee peel waste obtained an average yield of 6.23 ± 2.43 % per 100 g of dried powder, with results ranging from 4.20 to 10.50 g. These results may be influenced by several factors, such as differences in solvent application, extraction methods, and the condition of the materials used. FTIR test results confirmed the presence of significant functional groups of pectin, indicated by the peak points at 3320.46, 1731.69, and 164.92 cm^{-1}. These findings revealed that pectin extraction from coffee peel waste can effectively be performed using HCl solution, which was quite affordable and often readily available. However, the pectin yield obtained is still lower compared to other acidic extraction methods.

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