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## OPTIMIZATION OF PECTIN YIELD FROM KEPOK BANANA PEEL (*Musa Balbisiana* BBB) USING ULTRASONIC EXTRACTION WITH ACETIC ACID AND SULFURIC ACID SOLVENTS

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### Abstract

In Indonesia, banana fruit production occupies the first position compared to other fruits. The peel constitutes approximately 40% of the total weight of the fruit, thus producing a large amount of waste. The white part of the banana peel (mesocarp) has the potential to serve as a source of pectin, which primarily functions as a thickening and gel-forming agent. Pectin can be extracted from fruit peel using acidic solvents. This research was performed using the ultrasound-assisted extraction (UAE) method. This study utilized banana peel waste produced by banana chips traders in the city of Bandar Lampung and aims to determine the effect of temperature, time, and type of extraction solvent on the yield of pectin extract from kepok banana peels (*Musa balbisiana* BBB), and to identify the molecular functional groups of extracted pectin with the highest yield. Based on this research, it is known that the yield increases with increasing temperature and extraction time. The optimum condition for pectin extraction from kepok banana peel (*Musa balbisiana* BBB) was obtained at a temperature of 70 °C with a time of 90 minutes for both acetic acid and sulfuric acid solvents, yielding 12.63 and 14.09%, respectively. FTIR spectroscopy analysis reveals that the pectin extract exhibits functional groups with characteristic absorption bands in the specific wavelength regions consistent with pectin's structure. These include O–H stretching vibrations, –CH<sub>3</sub> groups in the methoxyl (COOCH<sub>3</sub>) branch, C–H bonds, C=O carbonyl groups, C–O bonds, and ether (C–O–C) linkages.

**Keywords:** Banana; Pectin; Solvent; Ultrasound-assisted extraction; Yield

### Abstrak

Di Indonesia, produksi buah pisang menempati posisi pertama dibandingkan buah-buahan lainnya. Dengan bobot kulit mencapai sekitar 40% dari total berat buah, kulit pisang menghasilkan limbah dalam jumlah yang besar. Bagian kulit pisang yang berwarna putih (mesocarp) berpotensi sebagai sumber pektin yang memiliki fungsi utama sebagai bahan pengental dan pembentuk gel. Pektin dapat diekstraksi dari kulit buah menggunakan pelarut asam. Penelitian ini dilakukan dengan ekstraksi menggunakan metode ultrasound-assisted extraction (UAE). Studi ini memanfaatkan limbah kulit pisang hasil produksi pedagang keripik pisang di kota Bandar Lampung dan bertujuan untuk mengetahui pengaruh suhu, waktu dan jenis pelarut ekstraksi terhadap nilai rendemen ekstrak pektin dari kulit pisang kepok (*Musa balbisiana* BBB), serta mengidentifikasi gugus fungsi molekuler pektin dengan nilai rendemen ekstrak terbaik. Berdasarkan penelitian ini diketahui bahwa nilai rendemen meningkat seiring dengan meningkatnya suhu dan waktu ekstraksi. Nilai optimum ekstraksi pektin dari kulit pisang kepok (*Musa balbisiana* BBB) yaitu pada suhu 70 °C dengan waktu 90 menit untuk pelarut asam asetat dan asam sulfat, menghasilkan yield sebesar 12,63 dan 14,09%. Berdasarkan hasil spektroskopi FTIR, ekstrak pektin menunjukkan keberadaan gugus-gugus fungsional dengan serapan pada daerah panjang gelombang yang sesuai dengan struktur khas pektin. Hal tersebut ditandai dengan terdapatnya vibrasi O-H, ikatan –CH<sub>3</sub> pada cabang metoksil COOCH<sub>3</sub>, ikatan –C–H, gugus karbonil –C=O, ikatan C–O dan gugus eter C–O–C.

**Kata Kunci:** Pektin; Pelarut; Pisang; Rendemen; Ultrasound-assisted extraction

## 1. INTRODUCTION

Banana production in Indonesia reached 9.24 million tons, accounting for approximately 33% of the national fruit production (Badan Pusat Statistik, 2022). Bananas are very popular as fresh fruit or processed products. Bananas have numerous applications in the community, including their leaves, stems, flowers, and fruit, and peel, which are highly useful and reusable. However, kepok banana peels (*Musa balbisiana* BBB) are not utilized; they are typically discarded as organic waste or used as feed for livestock such as goats, cows, and buffalo (May et al., 2019). The weight of banana peels reaches 40% of the fruit, making banana peels generate large amounts of waste. This waste can still be used in products that have economic value if used correctly and optimally (Megawati & Machsunah, 2016). Banana peels contain various compounds such as water, sucrose, starch, crude protein, crude fat, crude fiber, protopectin, and pectin, which are present in large amounts in banana peels (Timang & Sabang, 2019).

The chemical composition of banana peels still contains many carbohydrates, as much as 40.74% (Christy, 2017) and a pectin content of 22.4% (Tuhuloula et al., 2013). Owing to its thickening, gelling, and emulsifying properties, pectin is widely utilized in the food industry. It is also used in biomedical and biomaterial applications because of its possible immunomodulatory and anti-inflammatory effects, biodegradability, and biocompatibility. Another soluble dietary fiber with several advantageous physiological and gastrointestinal benefits is pectin (Roman-Benn et al., 2023). The need for pectin in Indonesia has increased from year to year, and to meet domestic pectin needs, in 2021, pectin imports reached 455,035 tons with an import value of US\$ 6,635,609, so one of the solutions to overcome the increasing need for pectin is to use banana peel waste as a raw material for pectin production (Badan Pusat Statistik, 2022). Pectin is included in the class of carbohydrate biopolymer compounds consisting of  $\alpha$ -D-galacturonic acid, which contains methyl esters and can be extracted from fruit peel using inorganic acid solvents (HCl,  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ) or organic acids ( $\text{C}_6\text{H}_8\text{O}_7$ ,  $\text{CH}_3\text{COOH}$ ) (Febriyanti et al., 2018). Acid solvents can release the pectin contained in plant cell walls by breaking the bonds between protopectin and materials contained in the fruit peel (Devianti et al., 2020).

Several methods can be used for pectin extraction, including conventional and non-conventional methods. Conventional methods such as maceration and soxhlet extraction are considered less effective in extracting because they require a long time (Damanik & Pandia, 2019). Susanti et al. (2021) has conducted research with hydrochloric acid, reaching the highest yield obtained in the ultrasonic method, which is 20.0008 grams, with a yield of 25.59%, while the conventional method can only get 18.3%. The advantages of the ultrasonic extraction are that the working procedure is more straightforward, the production process is more efficient because the extraction time is faster, the yield is greater, produces better product quality, and reduces

the use of solvents and energy. This study aims to determine the yield of pectin extract from green banana peel with variations in acid solvents, namely sulfuric acid and acetic acid, to compare the effects of strong versus weak acids, extraction temperature, and extraction time using the Ultrasonic Assisted Extraction (UAE) method.

## 2. MATERIALS AND METHODS

Kepok banana peel was taken from the local market, and 98% glacial acetic acid, 99% sulfuric acid, and 96% ethanol were purchased from a local chemical store.

### 2.1 Pretreatment of Banana Peel

The green kepok banana peel (*Musa balbisiana* BBB) was cleaned with running water and cut into small pieces using a knife. After being cut, it was placed in an oven (Advance AOV-500) at 50 °C until the weight remained constant and the moisture content was below 10%. Once dried, it was pureed using a blender and then strained using a 50-mesh sieve until a fine powder of kepok banana peel was obtained.

### 2.2 Pectin Extraction

A total of 20 grams of peel powder was placed into a two-neck flask. Then, 400 mL of acetic acid solution was added as the extraction solvent, and the mixture was stirred. Extraction was carried out using the 14 L 80 kHz Digital Ultrasonic Cleaner as shown in the Figure 1. The temperature was set at 50, 60, and 70 °C, while the extraction time was set at 30, 60, and 90 minutes. After extraction, the sample was filtered using Whatman filter paper grade 42, 125 mm. The experimental steps are then repeated using sulfuric acid as the extraction solvent.



**Figure 1.** Ultrasonic-assisted extraction setup

### 2.3 Pectin Deposition

The pectin filtrate was mixed with 96% ethanol in a 1:1 v/v ratio. The mixture was stirred and left to stand for 2 days. Afterward, the precipitate was separated from the solution using Whatman filter paper.

#### 2.3.1 Pectin wash

The pectin precipitate was stirred with 96% ethanol. Filtration was carried out using filter paper. Washing was repeated until the filtrate became clear and colorless. A pH test using indicator paper showed a neutral pH, confirming the absence of residual acid.

### 2.3.2 Pectin drying

The pectin was dried in an oven at 50 °C until it reached a constant weight. The dried pectin was then ground and prepared for analysis.

### 2.3.3 Extract analysis

After the pectin was dried, it was weighed, and the yield for each variation was calculated. The pectin obtained was then analyzed using an FTIR spectrophotometric analysis (using the Agilent Cary 630 spectrometer) to identify the functional groups and chemical structures. The FTIR spectrum was determined using the Agilent Technologies spectroscopy tool from the UPT Integrated Laboratory, University of Lampung. The FTIR spectrum of the extracted pectin was compared to the spectrum of commercial Cargill® and Danisco® pectin used as a standard.

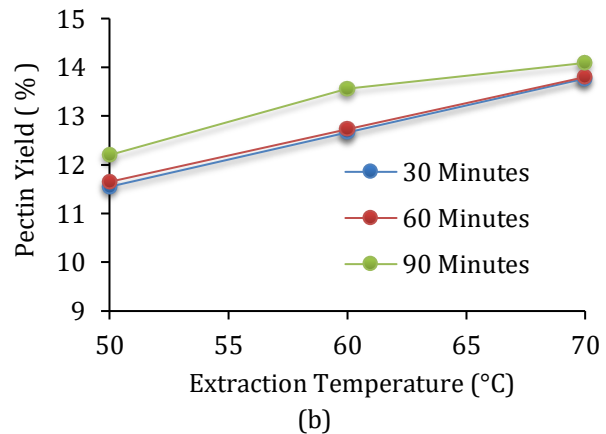
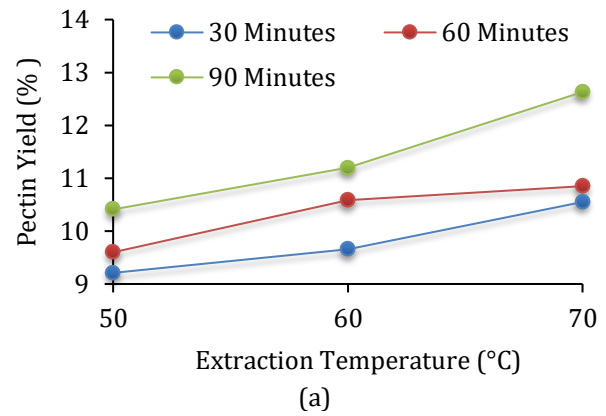
## 3. RESULTS AND DISCUSSION

### 3.1 Effect of Extraction Temperature and Time on the Pectin Yield (%)

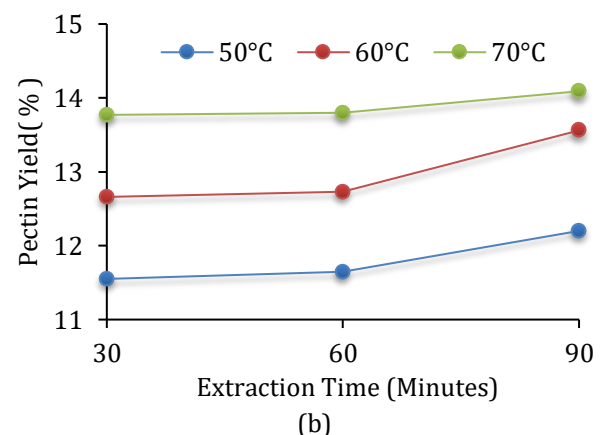
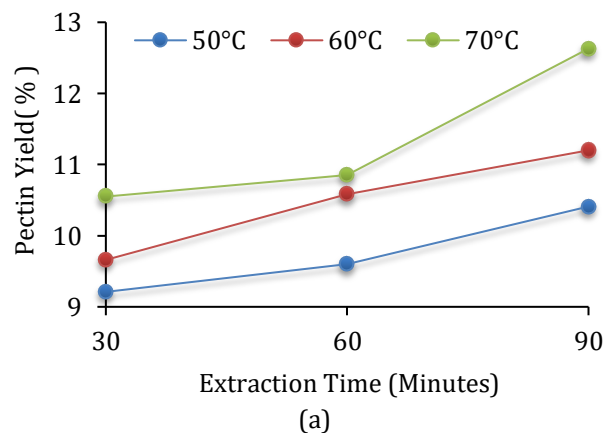
Figure 2 compares extraction temperature and pectin yield using acetic and sulfuric acid. The results show that increasing the extraction temperature increases pectin yield. The highest pectin yield was obtained at an extraction temperature of 70 °C.

Pectin extraction relies on the conversion of insoluble protopectin into soluble pectin by acid or enzymatic hydrolysis (Hanum et al., 2012b). Acid hydrolysis is influenced by temperature, acid concentration, and the material to be hydrolyzed. The effect of temperature on reaction speed follows the Arrhenius equation: the higher the temperature, the faster the reaction. The speed of the hydrolysis reaction will increase nearly twofold for every 10 °C increase in temperature. According to Figure 2, increasing the extraction temperature results in a greater yield.

This study shows that more extended extraction periods lead to higher yields, as shown in Figure 3. Extraction time was directly proportional to the pectin yield. High temperature and long extraction time increase the kinetic energy of the solution, increase solvent diffusion into tissue cells, and increase contact between particles (Shen et al., 2023). This causes pectin to be released from tissue cells, and the resulting yield becomes higher. Notably, an extraction time of 90 minutes resulted in the highest yield. A similar pattern was observed in a previous study by Hanum et al. (2012), where at an extraction temperature of 90 °C and time of 80 minutes, the highest pectin yield was 52.1% (Hanum et al., 2012a). However, according to Girma & Worku (2016), pectin yield decreases after 120 minutes. This decline occurs because, beyond 105 minutes, the high temperature can cause the pectin to degrade, resulting in a lower yield.



**Figure 2.** Effect of extraction temperature on pectin yield using (a) acetic acid and (b) sulfuric acid as solvents



**Figure 3.** Effect of increasing extraction time using different solvents: (a) acetic acid; (b) sulfuric acid

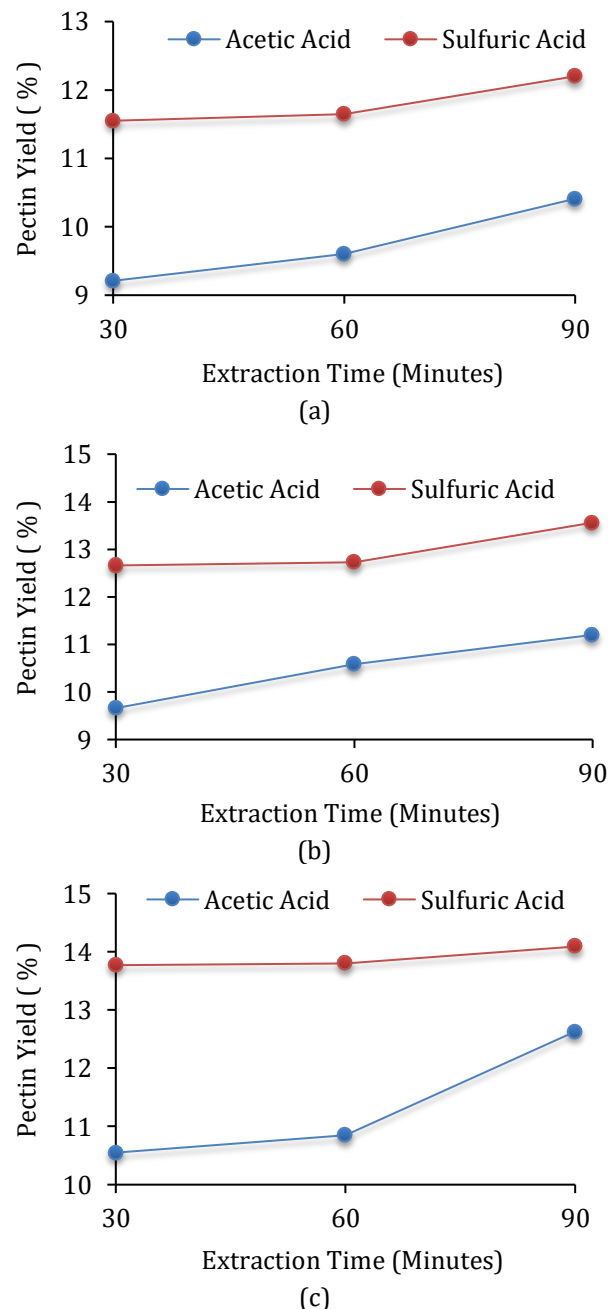
### 3.2 Effect of Solvent Type on the Yield (%) of Kepok Banana Peel Pectin Extract

This study extracted pectin using two different solvents: acetic and sulfuric acid. The pectin obtained is considered safe and suitable for pharmaceutical and food applications. To ensure its purity, the pectin extract is treated with ethanol, which helps dissolve any remaining acid or impurities. Additionally, the pectin is washed repeatedly until the filtrate is clear and colorless, and a pH test confirms that it is neutral, indicating the absence of acid residues (Febriyanti et al., 2018).

In Figure 4, it can be seen that pectin extraction using sulfuric acid produces a higher yield compared to acetic acid at various temperatures and times. This is due to the superior solvent ability of sulfuric acid in dissolving the pectin from the kepok banana peels. Sulfuric acid has higher acidity compared to acetic acid. This acidity affects the acid's ability to remove pectin from plant cells. Although high acidity can increase extraction efficiency, too high acidity can also damage the pectin structure. Sulfuric acid is also more reactive toward specific plant cell components, whereas acetic acid requires more specific reaction conditions (Hanum et al., 2012b).

Sulfuric acid produces a higher yield percentage compared to acetic acid, because sulfuric acid has more  $H^+$  ions than acetic acid, which functions to break protopectin bonds with other compounds in plant cell walls, and can unite one pectin molecule to another. Thus, forming a network that can trap water. Dissolving protopectin into pectin is due to the replacement of calcium and magnesium ions by hydrogen ions or breaking the bonds between pectin and cellulose. The higher the concentration of hydrogen ions, the higher the ability to replace calcium and magnesium ions, and the ability to break the bonds of pectin with cellulose will be higher, so that the dissolved pectin will increase (Nantika, 2022). The strength of the acid will increase in proportion to the increase in electronegativity of the central atom of sulfuric acid, so that the influence of electronegativity can influence the strength of the acid, and the destruction process takes place optimally.

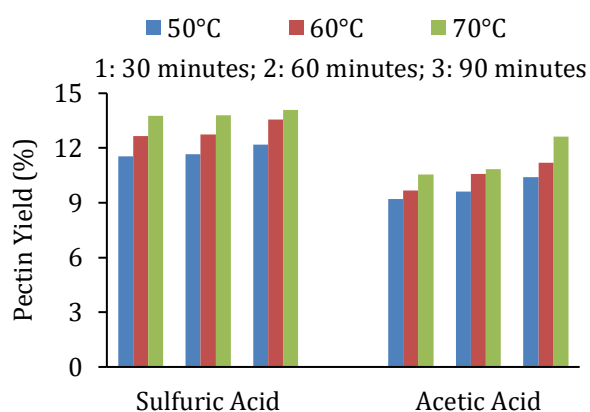
The yield of pectin produced from Kepok banana peel waste ranges from 9.21 to 14.09%. The highest yield in the acetic acid solvent was achieved at a temperature of 70 °C and 90 minutes, 12.63%, while in the sulfuric acid solvent, the highest pectin yield was achieved at the same temperature and the same time, which was 14.09%. In this case, the highest yield obtained was higher when compared to the results of research conducted by Fitria (2013), which stated that the highest pectin yield was obtained using sulfuric acid solvent of 9.67% with the soxhlet method (Tuhuloula et al., 2013). This proves the UAE method can produce a higher yield than conventional methods, such as soxhlet extraction.



**Figure 4.** Effect of solvent type on pectin yield at extraction temperatures of (a) 50 °C, (b) 60 °C, and (c) 70 °C

### 3.3 Simultaneous Comparison of Temperature, Extraction Time, and Solvent Type on the Yield (%)

Figure 5 shows that the yield value (%) will increase if the extraction temperature and time are greater. The highest yield was produced using a sulfuric acid solvent at 70 °C, with 90 minutes, as much as 14.09%.



**Figure 5.** Pectin yield at different solvents, times, and temperatures

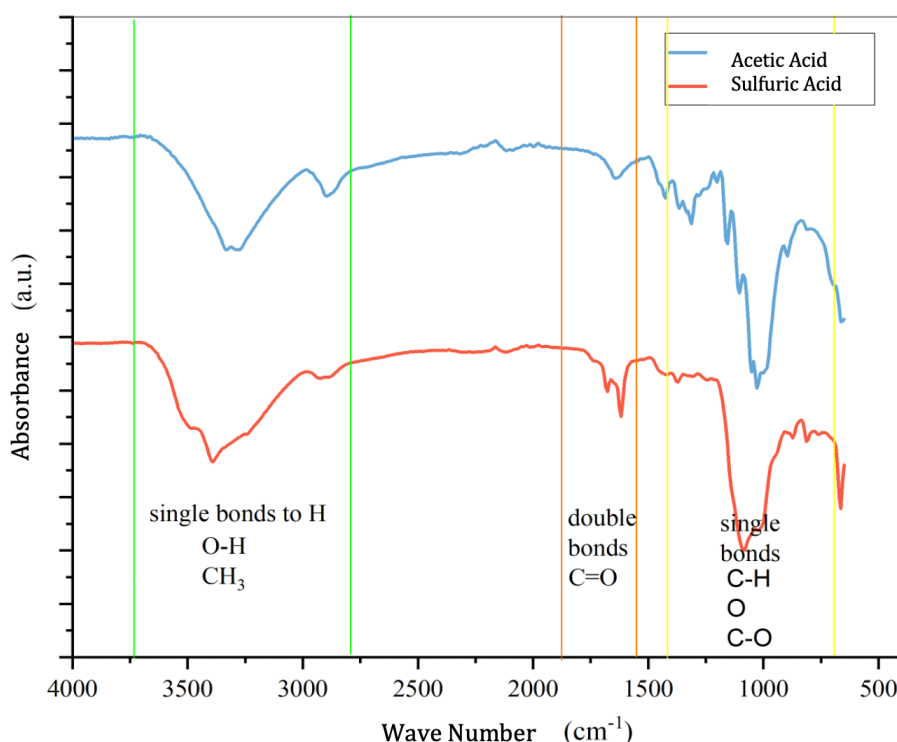
### 3.4 Fourier Transform Infrared (FTIR) Analysis

Of the 18 research samples, one best sample was selected from each type of solvent to be tested using FTIR. The selected samples were those extracted at 70 °C for 90 minutes.

Figure 6 shows that the main functional group in pectin is located in the wavelength area of 1000-2000  $\text{cm}^{-1}$  (Kalapathy & Proctor, 2001). The carboxyl bond is at 1630-1650  $\text{cm}^{-1}$  for the free carboxyl group and

1740-1760  $\text{cm}^{-1}$  for the esterified carboxyl group (Gnanasambandam & Proctor, 2000). Increasing the degree of esterification will also increase the intensity and area of esterified carboxyl groups. Pectin with high  $\text{COOH}$  (low degree of esterification) has better metal ion binding ability and is suitable for pharmaceutical applications (Sunthongjeen et al., 2004). While Pectin with high  $\text{COOCH}_3$  (high degree of esterification) is suitable for food applications that require gel formation in acidic conditions and high sugar content, such as jam and jelly (Kameshwar & Qin, 2018). The pectin in this study can be applied in the pharmaceutical field because it has a more dominant carboxyl group ( $-\text{COOH}$ ) than the methoxyl group ( $-\text{OCH}_3$ ). At wavelengths between 1100  $\text{cm}^{-1}$  and 1200  $\text{cm}^{-1}$ , it shows ether bonds ( $\text{R-O-R}$ ) and cyclic  $\text{C-C}$  bonds in the ring structure of the pectin molecule. The broad spectrum at 2400-3600  $\text{cm}^{-1}$  is moist in the absorbed pectin (Fitria, 2013).

Table 1 presents FTIR spectrum data for commercial pectin and extracted pectin. At specific wavelengths, absorption peaks indicate the presence of functional groups, such as  $\text{OH}$ ,  $\text{CH}_3$ ,  $\text{C=O}$ ,  $\text{C-H}$ ,  $\text{C-O}$ , and  $-\text{O}-$ . This spectrum depicts the stretching vibrations of the hydroxyl  $\text{OH}$  groups in the bonds between cellulose,



**Figure 6.** FTIR spectrum of pectin extract with acetic acid and sulfuric acid solvent

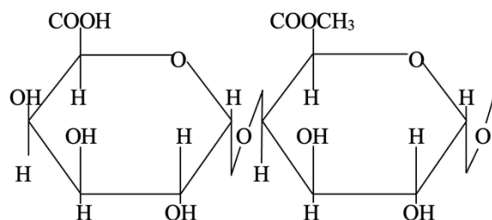
**Table 1.** Commercial pectin & extracted pectin FTIR spectrum data

Pectin	Area / Wave Number Description ( $\text{cm}^{-1}$ )					
	$-\text{OH}$	$-\text{CH}_3$	$-\text{C=O}$	$-\text{C-H}$	$-\text{C-O}$	$-\text{O}-$
Danisco®	3366,8	2939,6	1665,6	1441,8	1236,4	1010,7
Cargill®	3340,8	2936,7	1616,42	1443,17	1232,5	1143,84
Sulfuric Acid, 70 °C, 90 minutes	3391,9	2922,2	1677,3	1371,7	1088,4	812,6
Acetic Acid, 70 °C, 90 minutes	3382,2	2892,4	1640,0	1431,3	1312,0	1028,7



hemicellulose, and lignin. In addition, aliphatic CH bonds are absorbed from hydrocarbons in cellulose.

The pectin structure measured via FTIR spectroscopy includes functional groups such as OH vibrations,  $-\text{CH}_3$  bonds in the methoxyl branch ( $\text{COOCH}_3$ ), aliphatic  $-\text{C}-\text{H}$  bonds, carbonyl groups ( $-\text{C}=\text{O}$ ), and ether groups ( $-\text{O}-$ ).



**Figure 7.** Pectin structure (Krisnayanti & Syamsudin, 2013)

Figure 7 shows the structure of pectin with absorption in certain wavelength areas characterized by the presence of OH vibrations,  $-\text{CH}_3$  bonds in the methoxyl branch ( $\text{COOCH}_3$ ), aliphatic  $-\text{C}-\text{H}$  bonds, carbonyl groups ( $-\text{C}=\text{O}$ ), and ether groups ( $-\text{O}-$ ), which is in accordance with the FTIR spectrum results. Thus, FTIR analysis confirmed the structural components of pectin extracted from kepok banana peel waste.

#### 4. CONCLUSION

This research concluded that the higher the temperature and the longer the extraction time, the greater the yield of pectin extract. The choice of acid solvent significantly impacts the yield of pectin extract; pectin extraction using sulfuric acid solvent produces a higher yield percentage than acetic acid solvent. At a temperature of 70 °C and an extraction time of 90 minutes with sulfuric acid solvent, the best pectin was produced with a yield of 14.09%. FTIR spectrum analysis showed similarities between commercial and extracted pectin, indicating that pectin from kepok banana peels has similar structures and functional groups. The FTIR spectrum depicts the vibration of OH, the ( $-\text{CH}_3$ ) bond in the methoxyl branch ( $\text{COOCH}_3$ ), the aliphatic ( $-\text{C}-\text{H}$ ) bond, the carbonyl group ( $-\text{C}=\text{O}$ ), the (C-O) bond, and the ether group ( $-\text{O}-$ ) according to the structure of pectin.

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