Isolation of Pectin from coffee pulp Arabica Gayo for the development of matrices membrane

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Abstract. Pectin from Arabica Gayo coffee pulp has been successfully isolated using citric acid as a solvent. The pectin extracted from the coffee pulp is determined by the extraction conditions. This study aims to produce pectin from dried coffee extract using citric acid using two factors, namely the difference factor of citric acid (1: 5 and 1: 20 b/v) and extraction time 75 and 150 minutes. The optimum ratio of Arabica Gayo coffee pulp with citric acid was 1:20 b/v with rendemen 7.8% with 125 minute extraction time and at the temperature of 80 °C and pH 4. The methoxyl content of isolated pectin was 12.71% and has been determined as high methoxyl pectin (HMP). The result of analysis with FTIR is known that the extract produced is pectin with comparison with pectin standard. The pectin has been used as a matrix membrane and showed its smooth surface gel form and smaller diameter from scanning electron microscopy (SEM).

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1. Introduction

Coffee is one of the most important food commodities in the world and ranks second after crude oil. These plants can usually grow in tropical and subtropical regions [1]. A number of countries are known as coffee producers including Indonesia which is the fourth largest global coffee producer with an annual production of 686,763 tons (in 2007). Arabica and Robusta coffee (Coffee canephora) are the most famous coffee produced in Indonesia. Both coffee possess distinct taste and caffeine content and are cultivated in different areas. Arabica coffee, the most popular coffee are usually grown in the highlands. This coffee is well known for its low caffeine content and favourable non acidic taste. In Indonesia, this coffee is known as Gayo Arabica Coffee and are grown central Aceh. Similarly, Arabica coffee is also grown in Central America like Mexico, Guatemala, Costa Rica, Dominica, Jamaica.

It is common knowledge that the seeds from the coffee plant are processed into coffee beans which can be consumed directly afterwards. During this process, 55% to 60% useless material are produced from the pulp (part mesocarp), skin (eksokarp part), mucilage and parchment (endocarp part) [2]. Commonly, it is used for animal feeding or starting material for organic fertilizer. Carbohydrate forms the largest composition of coffee pulp which consists of pectin (35%), protein (5.2%), fiber (30.8%) and minerals (10.7%) while the mucilage portion contains water (84.2%), protein (8.9%), sugar (4.1%) and ash (0.7%) [2]. Therefore, based on these composition, coffee pulp becomes a potential material for pectin recourse.

Pectin is a natural anionic polysaccharide which is located in plant cell walls and are highly biodegradable [5]. It is a high value functional food which is widely used in gel and polymer formation [6]. With the availability of carboxylic groups, it was predicted that pectin can be utilized as matrices for biomolecule attachment. Pectin that is commonly found in the industry is a type of high methoxyl pectin (HMP). The HMP which minimally contains 7% of methoxyl and low methoxyl pectin (LMP) which maximally contains 7%, of methoxyl. It has the ability to form gel with sugar and acid. otherwise it will be able to form gel in the presence of calcium ions [3]. The formation of gel from pectin with high degree of methylation is also affected by pectin concentration, percentage of sugar and pH. The greater the concentration of pectin, the easy the gel will be formed. The formation of high methoxyl pectin gel takes place through hydrogen bonds between free carboxyl groups and hydroxyl groups [4].

This work is aimed to isolate pectin from Gayo Arabica coffee using organic acid solvent (citric acid). onto the best of our knowledge, there is no previous report about the use of citric acid as a solvent to extract pectin. citric acid is a more environmentally friendly option than strong acid [7] It is a weak acid that has lower dissociation than strong acid. In addition, the use of weak acid can prevent depolymerization of protons from pectin [8]. In order to confirm the formation of pectin, the isolated pectin will be characterized using Fourier Transform Infra Red Spectroscopy 400FT-IR/FT-NIR while the morphology gel matrix membrane will be evaluated using scanning electron microscopy

2. Materials and Methods

2.1 Material and Instrument

The main material used in this work was Gayo Arabica coffee pulp obtained from smallholder farmers in Takengon Central of Aceh, Indonesia and was dried in a green house for one month. 96% Ethanol was obtained from Emsure and citric acid, phenophthaline were purchased from Fisons. Sodium hydroxide, hydrochloric acid (HCl) were obtained from Merck. The isolated pectin was characterized using 400 FT-IR/FT-NIR Spectrophotometer and Scanning Electron Microscopy (SEM) Fesem Merlin.

2.2 Methodology

Gayo Arabica Coffee was obtained from Takengon, Central Aceh of Indonesia. The first layer of coffee seed was then dried for one month in a green house. Dried coffee peel was crushed using a
blender followed by pectin extraction using various citric acid concentration (w/v) (1:5, 1:10, 1:15, 1:20) In order to find the optimum yield of pectin, different length of extractions (75, 100, 125, 150 minutes) were investigated at temperature 85°C [9].

The extracts were filtered and cooled. Next, these filtrates were precipitated using 96% ethanol. The precipitate was washed with 30 mL of 96% ethanol for three times through filtration process and dried at 60°C. The yield of extracted pectin was determined. In addition, the degree of methoxylolation (DM) was calculated using acid base titration method and specific functional groups of pectin were evaluated based on FTIR spectra.

3. Result and Discussion

3.1 Effect of Pulp and Solvent Ratio on Yield Percentage

The yield of pectin obtained from extraction depends on the type of solvent and method used. Figure 1 presents a profile of rendement percentage on ratio of solvent and time of extraction.

![Figure 1: Effect of coffee pulp to solvent ratio at various extraction times on rendemen percentage.](image)

Percentage of pectin rendemen was influenced by ratio of solvent and extraction time. Figure 1 shows that the percentage of rendement has increased after 100 minutes of extraction time and the optimum rendement was reached at 125 minutes extraction time for all pulp to solvent ratio. The highest result, 7.8%, was obtained when the extraction was conducted at ratio 1:20 (w/V) pulp to solvent. This is due to the greater volume of solvents used. After 150 minutes of extraction time, the rendemen becomes stable because the ability of acid to hydrolize protopectin to pectin has reached its maximum capacity. When the coffee pulp to solvent ratio is increased against a longer extraction time a spread distribution of particles and expansion of surface contact was observed. previous studies using different starting materials such as banana peels, apples, and lemon byproducts reported that pulp to solvent ratio and a longer time extraction will affect the spread distribution of particles and expand the surface contact of particles and solvents. This will increase the production yield of pectin [10,11]. In this experiment the best ratio obtained was 1:20 after 125 minutes extraction time, with the percentage of rendements becoming stable. Therefore it can be concluded that the optimum extraction time was reached at 125 minutes.

Higher pectin content is caused by the decomposition of protopectin (cell wall) into smaller pectin particles due to partial acid hydrolysis during extraction, increased temperature and time. Pectin consists of α (1-4) related units of galactic acid or methyl ester [12]. Glycosidic bond is one type of ether bond, so it can pass the hydrolysis reaction under the right conditions. In this case, it is assumed that the higher polymer hydrolysis of the pectin molecule to the lower polymer causes increased
solubility in water and allows for easier extraction. At low temperatures, it may not be sufficient to allow the hydrolysis of protopectin by acid, this results in lower pectin yields[13].

3.2 Metoxyl Content Analysis
Metoxyl content in pectin is used to classify types of pectin like low metoxyl or high metoxyl pectin. In this work, the metoxyl content is in the range of 7.75% to 12.71%. The optimum result was found at pulp to solvent ratio of 1:20 and at 125 minutes extraction time. The lowest metoxyl content of 7.75% occurred at the lowest experimental condition of ratio 1:5 with 75 minutes extraction time. Figure 2 shows that metoxyl content was not affected by extraction time but is highly influenced by the pulp to solvent ratio. The metoxyl content also affects the solubility of pectin in water and will determine its application. The isolated pectin in this work was indicated to be high metoxyl pectin. Based on the FOOD Chemical Codex (1996) standard, pectin methoxyl content > 7% is classified as high and <7% is considered as low.

![Figure 2](image)

**Figure 2.** The Effect of coffee pulp to solvent ratio and extraction time on the percentage of methoxyl.

3.3 FTIR Analysis and SEM
Isolated pectin from Gayo Arabica coffee pulp was analyzed using FTIR and the morphology of its surface was characterized by SEM. Figures 3 and 4 depict the characteristics of pectin functional group. The FTIR spectrum in Figure 3 shows the wave number range of 950 and 1200 cm\(^{-1}\) as the 'finger print' area which identifies polysaccharides as carbohydrates. It has been confirmed by the similarity of finger print pectin standard as reported by Černá et al. 2003[14]. Degree of esterification (DE) of pectin can be determined by the relationship between peak area of the free carboxyl functional group (1650 cm\(^{-1}\)) and the ester group (1750 cm\(^{-1}\))[15].
The morphology of isolated pectin surface was characterized using a Scanning Electron Spectroscopy (SEM). The pectin surface image was taken at 2 μm magnification. As seen in Figure 3, pectin in gel form has a smooth area and homogenous surface. This characteristic indicates that pectin can be applied as a matrix membrane.

4. Conclusion
Pectin from Arabica Gayo coffee pulp has been successfully extracted using acid solvent and has been categorized as high metoxyl content. FTIR analysis has confirmed the functional group as pectin. In addition, the isolated pectin can be formed into gel and has homogeneous surface area that appropriate used as matrix membrane.
5. References

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