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EXTRACTION AND CHARACTERIZATION OF PECTIN FROM PASSION FRUIT (*PASSIFLORA EDULIS* L.) USING CHLORIDE ACID SOLUTION

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ABSTRACT

Pectin is a natural substance found in most food crops. In general, pectin extraction uses mineral and organic acids, such as sodium hexametaphosphate acid, sulfuric acid, hydrochloric acid, acetic acid, nit citric, and citric acid. The study aimed to obtain the concentration of HCl, which gave the best value for the physical and chemical properties of passion fruit pectin. This study was a completely randomized design. The tested factor was 400 ml HCl solution with five treatment levels of HCl concentration: 1 N; 1.5 N; 2 N; 2.5 N; and 3 N. Each treatment was repeated three times, and therefore 15 experimental units were tested. The variables observed were pectin yield, methoxyl content, galacturonic content, esterification degree, clarity, water content, and ash content. The results showed that the 2.5 N HCl concentration resulted a yield value of 27.47%, methoxyl content 5.20%, galacturonic content 121.62%, esterification degree 24.26%, clarity 14.65%, water content 4.37 %, and 0.54% ash content, so the 2.5 N HCl concentration was the HCl concentration that gave the best effect on the physical and chemical properties of passion fruit pectin extraction.

Keywords*:* Pectin, Passion fruit, Hydrochloric acid.

INTRODUCTION

Pectin is a naturally occurring substance found in most food crops. Pectin is a structural element in tissue growth and the major component of the middle lamella in plants. It also works as an adhesive and maintains the integrity of plant tissues and cells. In the presence of acids and sugars, pectin can gel. Pectin is most commonly used as an adhesive or thickening (Fitria, 2013).

Pectin is used as a gelling agent and thickening in fruit juice, jam, jelly, marmalade, emulsifiers, emulsion stabilizers, and medicinal drugs in the food industry. Pectin is used as a mixing agent in the pharmaceutical industry to make ointments, emulsion pastes, pills, and tablets. In contrast, pectin is used as an adjuvant in manufacturing soap, hair oil, and pastes (Delvia et al., 2019).

The need for pectin in Indonesia has increased from year to year, in 2007, which was 183,050 kg/year, until 2013, which was 240,792 kg/year. In 2020, it was estimated that the demand for pectin in Indonesia would reach 1,320 tons/year.

Pectin is widely used in the food and non-food industries. The need for pectin in Indonesia has been increased every year until now. There are not many companies that provide pectin for industrial needs. Therefore, Indonesia still imports pectin from abroad. The increasing value of pectin imports evidences this. The need for pectin has increased by 10-15% every year.

Passion fruit is widely grown in tropical and sub-tropical areas. Passion fruit is rich in vitamin C and minerals such as phosphorus. The passion fruit rind contains high pectin, namely 14%, so it is suitable as a source of pectin (Laga, 2000).

Chloric acid is the most commonly used acid in pectin extraction. Chloric acid is a cheap mineral acid and is widely used in industry, including the pectin-producing industry (Kesuma et al., 2018). Chloric acid is a stronger acid that is less dangerous to handle than other strong acids. This compound is widely used in the food industry (Ramdhan and Phaza, 2010).

By utilizing passion fruit as a basic ingredient for pectin production, it will reduce pectin imports, which can save the country's foreign exchange. Extraction was carried out using a concentration of 1N, 1.5N, 2N, 2.5N, and 3N HCl solutions.

MATERIALS AND METHODS

Research design. This research was conducted at the Agroindustry Laboratory and Agronomy Laboratory, Faculty of Agriculture, Tadulako University from November to December 2020.

The passion fruit (Passiflora edulis L.) was obtained from Baiya Village, Tawaeli District, Central Sulawesi. Fruit age ± 85 days after the flowers bloom, picked in the rainy season. The supporting materials used were HCl, 99% ethanol, distilled water, pectin samples, phenolphthalein (PP) indicator, NaCl, and NaOH.

The tools used in this study include a blender, digital scale, knife, analytical balance, furnace, petri dish, oven, dropper, measuring pipette, filter cloth, thermometer, plastic tray, plastic spoon, funnel, stirring rod, glass, 250 and 1000 ml Becker glass, 100 and 1000 ml measuring glass, 100 and 1000 ml volumetric flasks, stopwatch, refrigerator, titration equipment, 250 and 1000 ml Erlenmeyer, stopwatch, iron filter, camera, and stationery.

This study was one factor completely randomized design. The tested factor was 400 ml HCl solution using a concentration consisting of five treatments levels of HCl concentration: 1 N; 1.5 N; 2 N; 2.5 N, and 3 N. The analytical parameters observed were pectin yield, methoxyl content, galacturonic content, esterification degree, clarity, water content, and ash content.

Pectin Extraction. The extraction process was based on Ahmad (2017) method after modification. The first step is to wash the passion fruit thoroughly, then chop it with a size of ± 0.5 – 1.5 cm. Furthermore, the passion fruit was weighed as much as 100 g, and the passion fruit blended with 400 ml of HCl solution. Subsequently, blended fruits then filtered to separate the passion fruit pulp. The results are then heated in a frying pan using a hot plate at a temperature of 60 C for 30 minutes, then 99% ethanol was added and then precipitated. Then, separating the pectin from the solution was carried out in plastic packaging and stored in a refrigerator for analysis.

Observation Parameter

Pectin Yield. To determine the yield of pectin obtained, that is, by weighing the dry pectin and then doing a comparison with the weight of the sample.

Methoxyl levels. Methoxyl content was determined by dissolving 0.25 g of pectin with 50 ml of distilled water. After that, 6 drops of phenolphthalein were added, then titrated with 0.1 N NaOH. A color change indicated the equivalence point from brownish white to pinkish. The volume of NaOH required was recorded. After that, six drops of 0.1 N HCl solution were added and shaken. Then the solution was allowed to stand for 15 minutes (Abdillah, 2006; Akhmalludin, 2009).

Galacturonic levels. The solution that resulted in the determination of the methoxyl content was then shaken until the pink color disappeared, and 6 drops of phenolphthalein were added and titrated with 0.1 N NaOH until a pink color appeared. The degree of esterification (DE) of pectin can be obtained by comparing methoxyl and galacturonic levels. Degree of Esterification (Schultz, 1965).

The degree of esterification determines the properties of pectin, especially solubility and gel formation. The degree of esterification was calculated from the methoxyl content and galacturonic concentration obtained and calculated using the following formula:
Degree of esterification = (Methoxyl content x 176)/(Galacturonic concentration x 31) x 100 (Akmalludin and Arie, 2009)

Degree of Esterification. Pectin properties, particularly solubility and gel formation, are determined by the degree of esterification. The degree of esterification was determined using the following formula based on the methoxyl and galacturonic content obtained (Schultz, 1965):

Degree of Esterification =
$$\frac{\text{methoxil level x 176}}{\text{galacturonic level x 31}} \text{ x 100}$$

Clarity. Measuring the level of clarity of pectin was carried out using a spectrophotometer with a wavelength of 570 nm. Prepare a sample solution with several concentrations, connect the spectrophotometer to the socket and turn it on, wait until the term "initialization" appears and all tool functions have been checked automatically, wait until the menu selection display appears, select no.1 (photometric). Determine the wavelength to be used and the absorbance or transmittance.

Dissolve the pectin sample in 10 ml of distilled water. Stir until homogeneous, then filtered. Prepare a clean cuvette tube fill it with distilled water or sample solution to the mark on the top of the cuvette. Insert the cuvette containing the sample solution into the cuvette chamber properly and close the cuvette chamber. Wait until the absorbance or transmittance of the sample solution appears and record it. Clean the cuvette that has been used for the sample solution using distilled water and dry it with tissue paper until it is free of water and fat (Slamet et al, 1984).

Water Content. A total of 0.3 g of pectin samples were dried in an oven at a temperature of 105oC for 4 hours using a porcelain dish with a known empty weight. It is then cooled in a desiccator and weighed until the correct weight was obtained (Pardede et al., 2013).

Ash Content. The porcelain dish was dried in a kiln at a temperature of 650 C, then cooled in a desiccator and weighed as the weight of the container. A total of 0.5 g of pectin samples were added to a porcelain dish whose weight was known and then placed in a kiln at 650oC for 4 hours. Then cooled in a desiccator and weighed until a constant weight was obtained (Ranggana, 2000).

RESULTS AND DISCUSSION

Pectin Yield. The yield of pectin obtained in Figure 1 shows that the yield of pectin at a concentration of 1 N HCl was 16.85% then at a concentration of 1.5 N HCl decreased to 14.26%, then at a concentration of 2 N HCl, 2.5 N, and 3 N increased to 26.58%, 27.47%, and 39.01%.

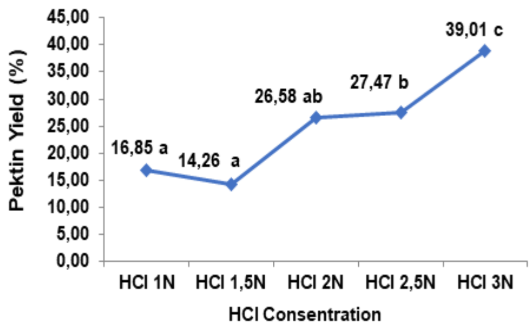

Figure 1. Passion Fruit Pectin Yield at Various Concentrations of HCl . Solution

Pectin extraction from different types of materials, solution concentrations, and methods will affect the yield of the extracted pectin. The pectin yield in passion fruit ranged from 16.85% to 39.01%. The optimum concentration is found in the concentration of 3 N HCl solution with a yield value of 39.01%. This was due to the influence of the concentration of acid used, indicating that at a concentration of 3 N HCl, the pectin substance in passion fruit was absorbed entirely, resulting in the highest yield.

According to Gatot et al. (2016), the yield of isolated pectin is strongly influenced by the extraction time and HCl concentration used. The higher the concentration of HCl used in the extraction of pectin will increase the yield of pectin obtained. It takes a long time to get a high pectin yield with a low HCl concentration. On the other hand, it takes a short time for high concentrations. The higher the concentration of HCl with the longer time it will decrease the yield of pectin obtained.

Kesuma et al. (2018) stated that the yield of pectin produced in the extraction using hydrochloric acid was higher than that of citric acid. Hydrochloric acid is a mineral acid that has a higher (K) balance than citric acid as an organic acid. The value (K) for hydrochloric acid is 10^7 while for citric acid, it is 7.21×10^4.

Methoxyl Level. The results of the methoxyl content (Figure 2) showed that the concentration of HCl 1 N was 1.97%, then it increased successively at concentrations of 1.5 N, 2 N, 2.5 N, and 3 N to 2.99%, 3, 82%, 5.20%, and 5.36%.

According to Fakhrizal et al. (2015), methoxyl content is defined as the number of moles of ethanol contained in 100 ml of galacturonic acid. Pectin can be called high methoxyl if it has a methoxyl content value equal to or more than 7%, less than 7% is called low methoxyl pectin (Fitria, 2013).

Based on the value of the methoxyl content, the pectin produced in this study is classified as pectin with low methoxyl content. Low methoxyl pectin can be isolated in small quantities from plants with relatively limited sources, including those extracted from cashew nut flesh, dragon fruit peel, sweet orange peel, papaya peel, jackfruit skin, Japanese plums, passion fruit skin, grape dollar fruit seeds, canola cake, and sunflowers (Liew et al., 2014).

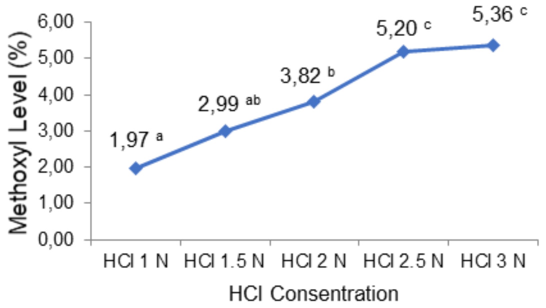

Figure 2. Methoxyl Pectin Levels of Passion Fruit at Various Concentrations of HCl . Solution.

Galacturonic Level. The results of galacturonate levels (Figure 3) show that the concentration of 1 N HCl was 52.00%, then it increased successively at the concentrations of 1.5 N, 2 N, 2.5 N, and 3 N to 72.61%, 91, 64%, 121.62%, and 126.39%.

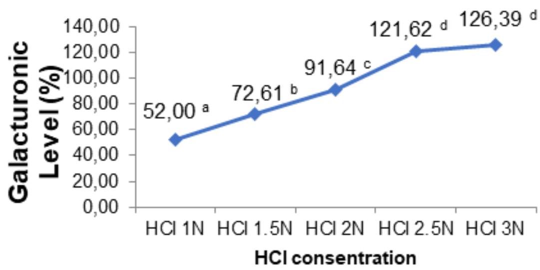

Figure 3. Galacturonic Acid Pectin of Passion Fruit at Various Concentrations of HCl . Solution.

The higher the concentration, the higher the galacturonic concentration. The higher the concentration, the more H+ ions where the H+ ion functions and breaking protopectin bonds with compounds in the passion fruit cell wall. Also, uniting one pectin molecule with other pectin molecules so that a network is formed that can trap water so that the texture of pectin is formed are bound to each other (Constenla and Lozano, 2003).

The hydrolysis reaction of protopectin into pectin causes the tendency for galacturonic levels to increase with increasing temperature and time. The essential component is D-galacturonic acid (Budiyanto and Yulianingsih, 2008). The increase in galacturonic acid occurs due to breaking the bond between the hemicellulose component and the polygalacturonic acid component of pectin due to heating with an acid solution (Shaha et al., 2013).

Esterification Degree. The results of the degree of esterification (Figure 4) show that the concentration of HCl 1 N was 21.94%, then it increased successively at concentrations of 1.5 N, 2 N, 2.5 N, and 3 N to 23.42%, 23, 66%, 24.26%, and 24.05%.

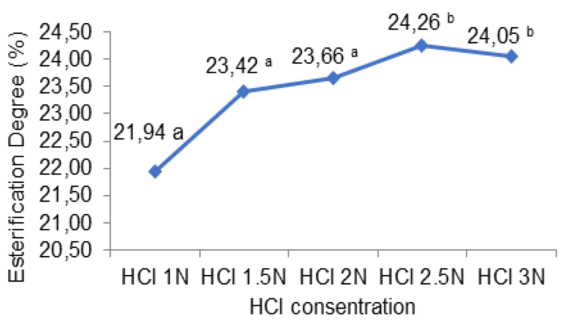

Figure 4. Degree of Esterification of Passion Fruit Pectin at Various Concentrations of HCl Solution.

According to Prabowo (2017), the higher the extraction temperature and time up to a certain point cause the esterification degree value to reach the optimum condition. However, there is a decrease in the esterification degree with increasing temperature and time.

Because pectin is a galacturonic acid with an esterified carboxyl group, increasing the amount of pectin extracted increases the esterification degree value. However, due to pectin degradation, the value of the esterification degree decreased at a given temperature and time.

This degradation is mainly due to the depolymerization mechanism of the galacturonic pectin chain (Liew et al., 2015). In addition, the high temperature and duration of extraction caused the de-esterification of pectin, so the value of the degree of esterification decreased (Hariyati, 2006).

Clarity. The clarity results (Figure 5) show that the concentration of 1 N HCl was 0.86%, then it increased successively at the concentrations of 1.5 N, 2 N, 2.5 N, and 3 N to 1.22%, 5.01 %, 14.65%, and 16.73%.

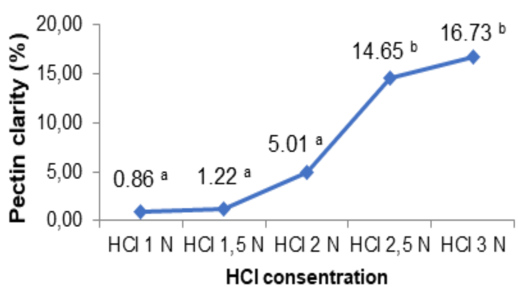

Figure 5. Clarity of Passion Fruit Pectin at Various Concentrations of HCl . Solution

According to Fitriani (2003), the clarity of pectin is strongly influenced by ethanol washing. If washing does not remove acid, the clarity of pectin will be low. The wet pectin obtained was found to contain ingredients other than pectin still. Due to the use of acid solvents that remain in the pectin precipitate after washing with ethanol, these components are most likely present throughout the extraction and agglomeration process. Extraction for too long will result in pectin that is not clear, hazy jelly, and a decrease in jelly strength (Kurniawati, 2013).

Based on Ahmad's research (2017), the clarity of the best cocoa pod husk pectin based on the results of extraction using hydrochloric acid was 71.22%. The HCl solution used in the extraction process did not affect the color of the ipod husky cocoa pectin because it was acidic. Ethanol is a clear liquid material that can provide clarity of pectin so that if it is added it will not change the color of the pectin packaging produced.

Water content. The results of the water content (Figure 6) show that the concentration of 1 N HCl was 2.46%, then increased successively at the concentrations of 1.5 N, 2 N, 2.5 N, and 3 N to 2.82%, 3.88%, 4.37%, and 6.76%.

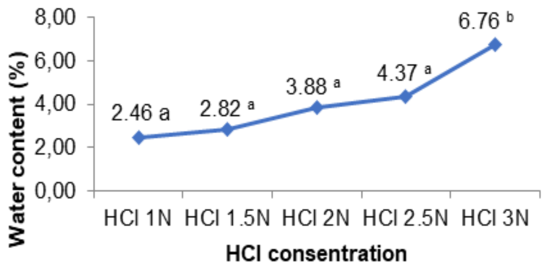

Figure 6. Passion Fruit Pectin Moisture Content at Various Concentrations of HCl Solution.

Based on these results, the water content of pectin produced in this study ranged from 2.46% to 6.76%. According to the quality standard of pectin (Table 2), pectin is declared of good quality if it contains water with a moisture content of <12%. Referring to the description, it can be stated that the pectin water content of passion fruit as a result of this study has met the quality standard.

The resulting water content can be affected by the yield of pectin. The higher the pectin yield, the higher the water content produced (Lumbantoruan et al., 2014).

Water content that is too high can be affected by the degree of drying and storage conditions of the pectin. The absorption of water by pectin during the extraction process depends on the free –OH group of the pectin molecule. The process of water absorption by pectin occurs during drying. The process of drying water from pectin can be divided into several stages. The first step is breaking hydrogen bonds between water molecules, which are the bonds with the lowest energy. Some of the water escapes, and the pectin surfaces approach each other. The hydrogen bonds between water and pectin are split, and hydrogen bonds are formed between the pectin surfaces (Pardede et al., 2013).

Ash Level. The results of the ash content (Figure 7) show that the 1 N HCl concentration was 0.47%, then decreased at 1.5 N HCl concentration to 0.34%, but at 2 N and 2.5 N HCl concentrations, it increased to 0 .38% and 0.54%, then decreased in 3 N HCl to 0.47%. The results showed that the passion fruit pectin ash content ranged from 0.47%-0.54%, following the quality standard of pectin ash content set by IPPA (International Pectin Producers Association) in Table 3, which is a maximum of 10%.

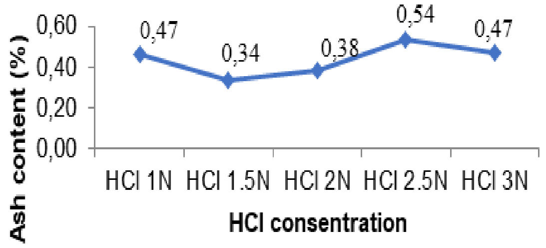

Figure 7. Ash Content of Passion Fruit Pectin at Various Concentrations of HCl . Solution

According to Castillo-Israel et al. (2015) in Delvia et al. (2019), the ash content indicates that there are still inorganic components left in the pectin. The lower the ash content, the better the pectin purity. The best quality pectin has an ash content of 0%.

According to Maulana (2015) in Delvia et al. (2019), treatment carried out using acid can result in hydrolysis of pectin from calcium and magnesium bonds. The components of Ca^{2+} and Mg^{2+} in the extraction solution increased as the hydrolysis reaction of protopectin increased.

CONCLUSION

Based on the research results that have been done, it can be concluded that the concentration of 2.5 N HCl is the concentration of HCl that gives the best effect on the physical and chemical properties of passion fruit pectin.

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