

## Extraction and Characterization of Pectin from Snake Fruit Peel via Maceration

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### ABSTRACT

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The high consumption of fruit flesh from the snake fruit is not in line with the utilization of fruit peels, which are often considered organic waste that goes to waste. The lack of optimal utilization of fruit peels as organic waste can open up opportunities to develop other alternatives, such as using fruit peels as a source of pectin. The extraction of pectin from fruit peels is carried out using the maceration extraction method with citric acid as the solvent. This research aims to determine the effect of the solvent material ratio and solvent concentration, as well as to understand the characteristics of the resulting pectin product. The results of this study indicate that among the variations in the ratio of materials and solvents, the highest %yield was achieved at a ratio of 1:40 (gr/mL) with a %yield value of 13.15%. In the variations of solvent concentration, the highest pectin %yield was at a concentration of 10% with a %yield value of 17.5%. The characteristics of the produced pectin were the highest equivalent weight of 114.94 mg, the highest methoxyl content of 9.92%, and the highest galacturonic acid content of 1063.04%. Functional group analysis using FTIR showed the presence of the O-H functional group at a wavelength of  $3255\text{ cm}^{-1}$ , the aliphatic C-H functional group at a wavelength of  $2808\text{ cm}^{-1}$ , the carboxyl (C=O) functional group at  $1838\text{ cm}^{-1}$ , and the methyl C-H functional group at a wavelength of  $1442\text{ cm}^{-1}$ . Therefore, the best research results were found at 1:40 and a solvent concentration of 10% with the highest %yield, equivalent weight, methoxyl content, and galacturonic acid content.

**Keywords:** pondoh snake fruit peel, pectin, maceration extraction, citric acid

## 1. INTRODUCTION

Indonesia is an agrarian country with various types of fruits. Fruit consumption in Indonesia has a very high value, reaching more than 20 million tons each year [1]. One of the fruits that is often consumed is snake fruit. Its very low price makes it popular among the public and adds to its appeal because it is rich in benefits such as vitamin C, fiber, and antioxidants [1], [2]. The production of salak fruit in Indonesia has always been above 1 million tons each year [1]. In 2021, the production of salak fruit reached 1,120,242 tons according to data from the Central Statistics Agency (BPS), increased to 1,147,473 tons in 2022, and returned to 1,120,739 tons in 2023 [1]. The high consumption of fruit flesh is not in line with the utilization of fruit peels, which are often considered organic waste that goes to waste [3]. Most fruit peels account for 30-35% of the total weight of the fruit, which tends to be underutilized [4]. The lack of optimal utilization of fruit peels as organic waste can open up

opportunities to develop other alternatives, such as using fruit peels as a source of pectin. Because pectin from fruits is generally found in the skin of the fruit [3], [5], [6]

Pectin is a complex anionic polysaccharide found in the primary cell walls and intercellular spaces of higher plant cells [7] [9]. Generally, the pectin found in natural materials is located in the primary cell walls, particularly between cellulose and hemicellulose [7]. Pectin is used as a functional component in the food industry due to its ability to form a gel and stabilize proteins [8]. One of the separation methods to produce pectin is through the extraction method [9], [10].

Extraction is the process of separating components from raw materials in liquid or solid form with the help of a solvent [11], [12]. Generally, pectin extraction is carried out using acidic solvents, both mineral acids and organic acids. The role of acid in pectin extraction is to separate bivalent ions,

break the bond between pectic acid and cellulose, and hydrolyze protopectin into water-soluble pectin. Common solvents used for extraction include sulfuric acid, hydrochloric acid, acetic acid, nitric acid, and citric acid [13], [14]. In addition, the characteristics and physical properties of pectin are also influenced by the conditions of pectin extraction [13]. The extraction conditions that affect the characteristics and physical properties of pectin include contact time, the ratio of material to solvent, solvent concentration, extraction temperature, and the type of solvent used during extraction [5], [12], [13], [15].

In the study, citric acid ( $C_6H_8O_7$ ) solvent is needed to help extract pectin from the skin of Pondoh snake fruit. The use of this solvent is since organic acid solvents can minimize the degradation of pectin molecules into pectic acid. The selection of organic acids is considered more strategic than mineral acids because they have lower toxic properties. In this study, experiments will be conducted using the batch maceration method.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Snake fruit peel, citric acid, 96% ethanol, 1 N NaOH, 0.25 N NaOH, 0.25 HCl, sodium chloride, potassium bromide, distilled water, PP indicator (phenolphthalein), aluminum foil, and filter paper.

### 2.2 Experimental procedure

Preliminary treatment was carried out to eliminate bacteria in the sample. Then, drying was performed using an analytical oven to remove the moisture content from the skin. Next, the skin was ground with the help of a copper blender until it became a fine powder. The powder is sifted through a wire mesh with a size of 0.25 mm, resulting in small particles that can accelerate the extraction process.

The fine powder is weighed at 20 grams and then mixed with a citric acid solution with an initial concentration (8%). This initial concentration is taken from the middle variable in the solvent concentration. Reacted with the variable ratio of sample to solvent (g/mL). Pectin extraction was carried out using an analytical oven at a temperature of 95°C and a duration of 150 minutes, with stirring every 30 minutes. The heated mixture is immediately filtered using filter paper and a vacuum filter. The filtrate obtained from the filtration is allowed to stand until room temperature. After the temperature decreases, 96% ethanol is added in a 1:1 ratio to precipitate. The mixture is stirred until homogeneous. After the

precipitate forms in the shape of bubbles, it is filtered again. After filtration, the precipitate is washed with 96% ethanol. The result is placed in an oven at 50°C for 24 hours. The drying result is a pectin product. The highest yield in this variable is used for the next treatment, which is varied with the solvent concentration variable.

### 2.3 Methods of analysis

**Table 1.** Standard quality of pectin based on SNI pectin and International Pectin Procedures Association [14].

Quality Factor	Content		
Gel strength	Min 150 grade		
Metoxyl content:			
• High methoxyl pectin	>7.12%	2.5-7.12%	
• Low methoxyl pectin			
Galacturonic acid content	Min 35%		
Moisture content	Maks 12%		
Ash content	Maks 10%		
Degree of esterification:			
• High ester pectin	Min 50%		
• Low ester pectin	Maks 50%		
Acetyl number	0.15 – 0.45%		
Equivalent weight	600 – 800 mg		

Commercial pectin must meet high-quality standards, which should be by the International Pectin Producers Association (IPPA) and the established SNI Pectin (01-2238-1991). The analysis of pectin in this study includes equivalent weight analysis, methoxyl content, and galacturonic acid content [10].

#### 2.3.1 Analysis of Equivalent Weight

Equivalent weight indicates the type of methoxyl content present in pectin, which can affect the quality of pectin in gel formation. The equivalent weight is determined through the weight of the sample titrated with normality and the volume of titrant obtained during the titration.

$$\text{Equivalent weight} = \frac{\text{Sample weight (gr)} \times 1000}{\text{Vol. titran (mL)} \times \text{N Titran}} \quad \dots \quad (1)$$

#### 2.3.2 Analysis of Methoxyl and galacturonic acid content

The analysis of methoxyl content serves as a benchmark for measuring the amount of alcohol still attached to pectin. Meanwhile, the galacturonic acid content can be used as a reference to indicate the amount of degraded pectin. The methoxyl content and galacturonic acid content are determined by performing back titration on the equivalent weight analysis results, which are added

with 0.25 N NaOH and 0.25 N HCl, each with a volume of 25 mL [10], [16].

$$\% \text{ Metoksil} = \frac{\text{vol.titran (mL)} \times \text{N titran} \times 31}{\text{gr pektin} \times 1000} \times 100\% \quad \dots \dots \dots (2)$$

% Galacturonic acid =

$$\frac{\text{mEq NaOH (Equivalent weight +methoxyl)} \times 176}{\text{gr pectin} \times 1000} \times 100\% \quad \dots \dots \dots (3)$$

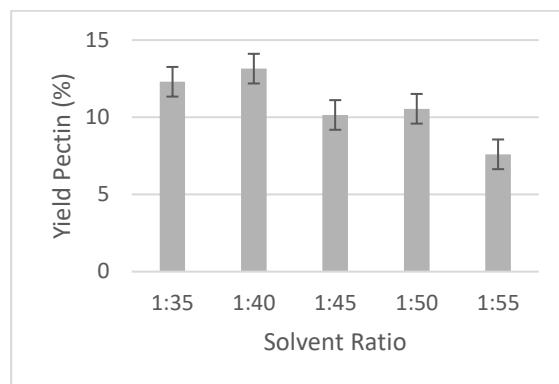
### 2.3.3 Analysis of functional group of pectin with FTIR spectrum

The functional groups in pectin were identified using FT-IR spectroscopy. Pectin samples were prepared by drying them at 40°C for 24 hours to remove moisture. The dried samples were ground into fine powders using a mortar and pestle. Approximately 2 mg of the powdered pectin sample was mixed thoroughly with 200 mg of spectroscopic-grade potassium bromide (KBr) and pressed into a thin, transparent pellet using a hydraulic press under a pressure of 10,000 psi. Alternatively, for ATR-FTIR, a small amount of the powdered pectin was directly placed on the ATR crystal. The FT-IR spectra were recorded using a PerkinElmer Spectrum Two FT-IR Spectrometer equipped with an ATR accessory. Spectra were collected in the mid-infrared range of 4000–400 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup> and 32 scans per sample to ensure a high signal-to-noise ratio. The spectra were collected after performing a background scan to eliminate interference from atmospheric water vapor and CO<sub>2</sub>. The pectin sample was analyzed at room temperature under ambient conditions.

## 3. RESULTS AND DISCUSSION

In this study, the yield and characteristics of the obtained pectin showed varying results. The treatment was conducted using 20 samples, which were limited by two variables: the ratio of material to solvent and the solvent concentration variable. The pectin results are shown in **Figure 1**.

### 3.1 The Effect of Solvent Ratio on the Yield of Pectin



**Figure 2.** Pectin yield at various material to solvent ratios

Figure 2 shows the results obtained, the yield of pectin decreases as the amount of solvent used increases. This is because as the solvent ratio increases, the effective acid concentration for releasing pectin from the cell walls decreases, thereby reducing the extraction efficiency [5]. Large volume of solvent causes the dissolved pectin to become too dilute, making the precipitation or concentration process less efficient, which in turn reduces the yield of pectin [13]. From the data, the highest pectin yield was obtained at a ratio of 1:40 with a yield of 13.5%. In banana peel waste was used as the raw material, with the highest yield obtained at a material-to-solvent ratio of 1:50, yielding 10.68%. Meanwhile, at a ratio of 1:40, a pectin yield of 9.88% was obtained.

ANOVA analysis was conducted with a homogeneity test aimed at ensuring that the data groups come from populations with the same variance. (homogen). Statistical analysis in this study was conducted using one-way ANOVA. Decision-making in ANOVA statistical analysis can be based on the following criteria. If the Sig value > 0.05, then the data distribution is homogeneous. Whereas, if the Sig value < 0.05, then the data distribution is not homogeneous (Table 2). Based on the results of the ANOVA analysis, a significant value of 0.931 was obtained, which means the Sig value > 0.05. Therefore, based on this homogeneity test analysis, it can be said that the data used is homogeneous.

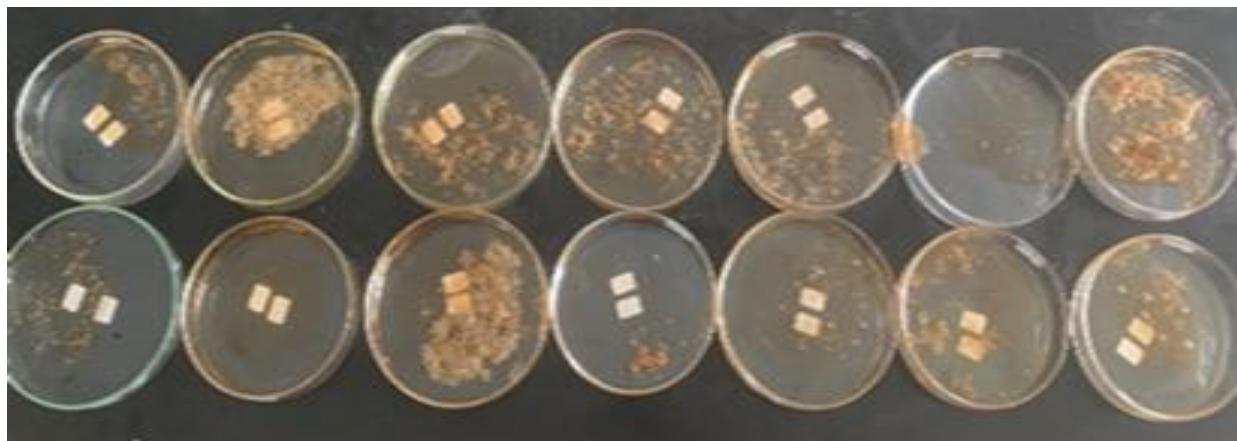


Figure 1. Pectin product

Table 2. ANOVA analysis of homogeneity test for the ratio of material to solvent variable

Test of Homogeneity of Variances					
		Levene Statistic	df1	df2	Sig.
Yield pectin (%)	Based on mean	0.008	1	8	0.931
	Based on median	0.015	1	8	0.905
	Based on median and with adjusted df	0.015	1	7.850	0.905
	Based on trimmed mean	0.004	1	8	0.948
ANOVA					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	10.920	1	10.920	2.678	.140
Within Groups	32.622	8	4.078		
Total	43.542	9			

### 3.2 The Effect of Concentration on the Yield of Pectin

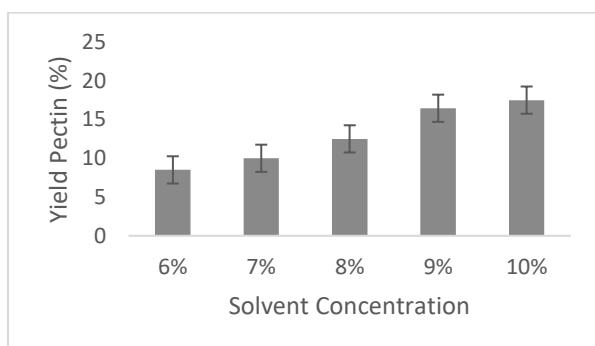


Figure 3. Pectin yield on solvent concentration variables

Based on Figure 3, it is stated that the higher the solvent concentration, the higher the yield obtained. Higher solvent concentrations, especially in terms of acid, can enhance the solvent's ability to break down plant cell walls. The cell walls of the raw materials usually consist of cellulose, hemicellulose, and lignin, which bind pectin. With the increasing concentration of acid, the hydrolysis

process becomes more effective, facilitating the release of pectin from the cell walls, thereby increasing the pectin yield [5].

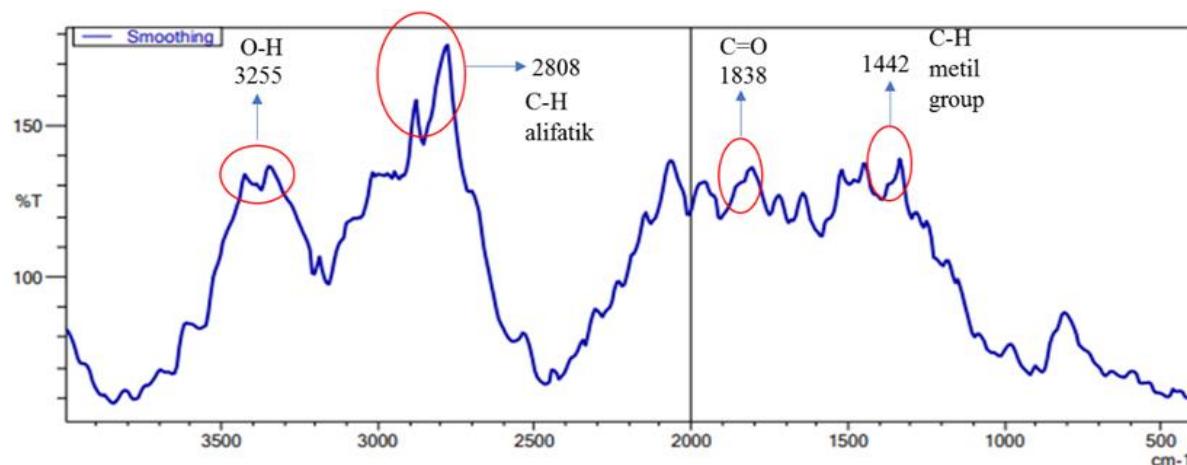
The best pectin yield was obtained at a solvent concentration of 10% with a yield percentage of 17.5%. However, the increase in pectin yield from a concentration of 9% to 10% was not very significant. Therefore, it is possible that after the 10% concentration, there will be a decrease in pectin yield. As in the study which used 4 variations of citric acid concentrations, namely 5%, 7%, 9%, and 11%, it was stated that the highest yield was obtained at a concentration of 9% amounting to 35.67%, while during the experiment at a concentration of 11%, there was a decrease in pectin yield. The yields obtained at each citric acid concentration were 32.11%, 33.56%, 35.67%, and 32.6%. The decrease in pectin yield was caused by the solution being too concentrated, leading to the degradation of pectin into pectic acid [9].

**Table 3.** ANOVA analysis homogeneity test of solvent concentration variable

		Test of Homogeneity of Variances		
		Levene Statistic	df1	df2
Yield pectin (%)	Based on mean	5.948	1	8
	Based on median	1.119	1	8
	Based on median and with adjusted df	1.119	1	4.025
	Based on trimmed mean	4.357	1	8
ANOVA				
	Sum of Squares	df	Mean Square	F
Between Groups	256320.100	1	256320.100	1/059
Within Groups	1936716.800	8	242089.600	
Total	2193036.900	9		

**Table 4.** Characteristics of pectin on the variable ratio of material to solvent

c	Equivalent Weight (mg)	Methoxyl Content (%)	Galacturonic Content (%)
1:35	114,94	6,51	760,32
1:40	97,09	9,3	936,32
1:45	86,96	8,06	992,64
1:50	84,03	9,92	1063,04
1:55	90,91	8,68	971,52



**Figure 4.** FTIR spectrum of pectin

Based on the results of the ANOVA homogeneity test, a significant value of 0.041 was obtained, which means the Sig value  $< 0.05$ . Therefore, from this homogeneity test analysis, the data on the solvent concentration variable can be said to be non-homogeneous (Table 3).

### 3.3 The Effect of Solvent Ratio on Pectin Characteristics

In the pectin product with a 1:35 ratio, the analysis results showed an equivalent weight yield of 114.94 mg. The methoxyl analysis yielded 6.51%,

classifying it as low-methoxyl pectin, and the galacturonic acid yield was 760.32%. In the pectin product with a 1:40 ratio, the equivalent weight yield was 97.09 mg. The methoxyl analysis yielded 9.3%, indicating that the pectin is classified as high-methoxyl pectin. The galacturonic acid analysis yielded 936.32% (Table 4)

The pectin product with a 1:45 ratio has been treated to determine its characteristics through analysis of equivalent weight, methoxyl content, and galacturonic acid content. Each

analysis yielded results of 86.96 mg for the equivalent weight analysis, 8.06% for the methoxyl content analysis, and 992.64% for the galacturonic acid content analysis in the pectin product. The pectin product at a 1:50 ratio had an equivalent weight of 84.03 mg, which is still below the standard commercial pectin requirements. The methoxyl content at a 1:50 ratio was 9.92%, indicating high methoxyl pectin. The galacturonic acid content showed a result of 1063.04%. Meanwhile, the pectin product at a 1:55 ratio had an analysis result of 90.91 mg for the equivalent weight, 8.68% for the methoxyl content, which is classified as high methoxyl pectin, and 971.52% for the galacturonic acid content.

Pectin can form gels well if it has a relatively high molecular weight, methoxyl content, and polygalacturonic acid content. In the analysis of equivalent weight, the highest yield was obtained at a ratio of 1:40, but the standard equivalent weight of pectin is in the range of 600 mg to 800 mg. Meanwhile, in the variable solvent ratio, pectin with high methoxyl content was obtained because it has a content of more than 7.12%, except at the ratio of 1:35. The lower the methoxyl content in pectin, the more difficult it will be for the pectin to dissolve in water; conversely, the higher the methoxyl content in pectin, the easier it will be to dissolve in water.

### 3.4 The Effect of Solvent Concentration on Pectin Characteristics

**Table 5.** Pectin characteristics at solvent concentration variables

Vari able	Equivalent Weight (mg)	Methoxyl Content (%)	Galacturonic Content (%)
6%	94,34	4,03	837,76
7%	96,15	2,48	788,48
8%	95,24	2,79	802,56
9%	85,47	5,27	943,36
10%	81,97	6,82	1013,76

Based on **Table 5**, the pectin product at a solvent concentration of 6% has an equivalent weight of 94.34 mg. According to the analysis conducted, the yield of methoxyl and galacturonic acid in the pectin product at this concentration are 4.03% and 837.76%, respectively. The pectin product at a solvent concentration of 7% has been analyzed with an equivalent weight of 96.15 mg. It has a low methoxyl content of 2.48% and a very high galacturonic acid content of 788.48%.

The pectin product at a solvent concentration of 8% has an equivalent weight of 95.24 mg. Its methoxyl content is 2.79%, and the

galacturonic acid content of the pectin at this concentration is 802.56%. The pectin product at a solvent concentration of 9% has an equivalent weight of 85.47 mg, a low methoxyl content of 5.27%, and a galacturonic acid content of 943.36%. The pectin product at a solvent concentration of 10% has the lowest equivalent weight among the other solvent concentration variables, with an equivalent weight of 81.97 mg. It has a low methoxyl content of 6.82%. The pectin product at this concentration has a galacturonic acid content of 1013.76%.

Based on these results, all the pectin products obtained have an equivalent weight that is still far below the commercial standards [3]. The low degree of esterification is due to the deesterification process of pectin into pectic acid, which increases the number of free acid groups, thereby reducing the degree of esterification [13].

In this solvent concentration variable, all pectin products show low-methoxyl pectin. Low-methoxyl pectin does not have the ability to form gels in the presence of sugar and acid, but can form gels in the presence of polyvalent cations such as calcium [3]. In this variable, the highest methoxyl content was obtained at a solvent concentration of 10%. An increase in acid concentration will raise the methoxyl content of pectin because the esterified carboxyl groups increase[17].

The standard galacturonic acid content set by the International Pectin Producers Association is a minimum of 35%. Although all samples have met the established quality standards, the results are still too high and exceed the maximum percentage limit. The difference in galacturonic acid content in this study compared to other studies may be influenced by the source of raw materials, solvents, or extraction methods used. Pectin also contains other compounds such as neutral sugars like D-galactose, L-arabinose, and L-rhamnose. These non-uronate compounds can be carried over during the pectin coagulation process. These compounds can become impurities, thereby affecting the composition of the pectin compounds [9].

### 3.5 FTIR Spectrum Analysis

From **Figure 4**, it shows the presence of a hydroxyl group peak at a wavelength of  $3255\text{ cm}^{-1}$  from the salacca peel pectin. Meanwhile, the absorption at a wavelength of  $2808\text{ cm}^{-1}$  occurs due to the aliphatic C-H group. The absorption spectrum at a wavelength of  $1838\text{ cm}^{-1}$  indicates the presence of the carbonyl group ( $\text{C}=\text{O}$ ), and at a wavelength of  $1442\text{ cm}^{-1}$ , it indicates the presence of the methyl C-H group [28].

#### 4. CONCLUSION

The influence of the material-to-solvent ratio variable on the yield and characteristics of pectin showed significant results, where the highest pectin yield in this variable was obtained at a solvent ratio of 1:40 (g/mL) with a yield of 13.15%. The characteristics of the produced pectin were an equivalent weight of 97.09 mg, a methoxyl content of 9.3%, and a galacturonic acid content of 936.32%. The influence of the solvent concentration variable on the yield and characteristics of pectin also showed significant results, where the highest pectin yield in this variable was obtained at a solvent concentration of 10% with a yield of 17.5%. The characteristics of the produced pectin were an equivalent weight of 81.97 mg, a methoxyl content of 6.82%, and a galacturonic acid content of 1013.76%.

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#### CONFLICT OF INTEREST

No potential conflict of interest was reported by the author(s).

#### CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Dani Faizal:** Data curation, Formal analysis, Investigation, Writing an original draft.

**Anila Wirantika:** Investigation, Writing original draft.

**Neni Damajanti:** Conceptualization, Methodology, Investigation, Formal analysis, Writing original draft. Supervision.

All authors have read and agreed to the published version of the manuscript.

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