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Screening of factors influencing Pectin Extraction of Pomelo peels using 2-Level Factorial Design

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Abstract. A study was conducted to vary the parameters affecting the extraction of pectin from pomelo peels (*Citrus maxima*) using a 2-level factorial design (2LFD). Experimental design was carried out to screen for significant environmental factors for extraction results. The factors involved consisted of pH, temperature and extraction time. The results of the variance analysis (ANOVA) found three main factors that had a statistically significant effect on the results of pectin extraction, namely pH, temperature and extraction time. The following important factors are the interaction factors between pH and temperature, the interaction between temperature and extraction time, the interaction between pH and extraction time, the interaction between temperature and extraction time. Statistical analysis shows that the linear model is significant with R² value of 0.9715. It was found that pH, temperature and extraction time are the most important parameters that affect the results of pomelo peels pectin extraction.

1. Introduction

Pomelos's scientific name is *Citrus Maxima* in the Citrus group and Rutaceace. about 30% of the total fruit weight is pomelo peels (like white spongy flowers), is a potential source of pectin [1]. Pectin is a complex family Galacturonic acid polysaccharide present in the primary and middle cell walls lamella plant tissue. Pectin is widely used as a gelling agent and stabilizer in various food products, pharmaceuticals, cosmetics [2]. Pectin is usually extracted from some resources, like orange peel, pomace apple, lemon and sugar beet by inorganic acids such as HCl [3] or HNO₃ [4], and organic acids such as citric acid [5], in the pectin industry. It is well-known that pectin has a role as a polysaccharide structure in the main cell walls and middle lamellartall plants which contributes to the structural strength of a plant and also to various cell functions including [6].

One of the main components of the primary cell wall and is generally considered to be around one third of all primary cell wall macromolecules is Pectin. Pectin is a group of polysaccharides consisting mostly of D-galacturonat acid and some carboxylic groups of galacturonic acid molecules in the pectin chain are methyl esterified and the percentage of the group esterified is expressed as DE (degree of esterification). Pectin is divided into two majors depending on the level of esterification; pectin ester is higher with ED more than 50%, and low pectin ester with DE lower than 50% [7]. The results and quality of pectin depend largely on the source, as well as the method used for pectin extraction. The results of pectin extracted from sugar beet by acid (HCl or HNO₃) at varying pH (1-3), temperature



(75-90oC) and time (30-90 minutes) show galacturonic acid content and extraction time varies from 295 to 528 mg / g (dry weight) and 34% to 94%, respectively [8]. This work aims to extract pectin from pomelo peels (*Citrus maxima*)

2. Experimental

2.1. Material

The selected pomelo peels (*Citrus maxima*) from Pangkep district, South Sulawesi. Furthermore, the peels part used as the experimental material in this study. Pomelo peel is cut into small pieces and then washed and dried in an oven at 60°C for 5 days until the dry weight of the leaves is stable. This dry sample of pomelo peel is ground and filtered to get a small particle size using a 1 mm filter.

2.2. Pectin Extraction

Extraction methods based modification from [8] and [9]. A total of 10 g pomelo peels flour was added to aquadest with ratio of 1:20. Determination of pH 1.5 and 2.5 with 0.5 N HCl solvent, then incubated of temperature at 60°C and 100°C, extraction time of 60 minutes and 120 minutes. Stirring is carried out during incubation. After incubation, filtering is done to get the filtrate using filter paper. Furthermore, the filtrate was precipitated with 95% ethanol concentration for 24 hours. The precipitate is washed twice also with 95% ethanol. The obtained pectin is dried in an oven at 50°C until a constant weight is reached. The dried is calculated as the weight of dry pectin (g) per 100 g of dried pomelo peels.

2.3. Experimental Design and Statistical Analysis

The two-level factorial design made by software DESIGN EXPERT (State-Ease Inc., Statistics simplified, Minneapolis, MN, USA, Version 6.0.4) was used to filter out the factors that influence the extraction of pomelo peels pectin. There are 3 variables used, so this design contains a total of 24 experimental experiments. Each independent variable is investigated for duplicates at high (+1) and low (-1). The variables used were pH (A), temperature (B), extraction time variables coded -1 (low code) and +1 (high coded) (Table 1). Variables that had a large effect on the results of pomelo peels pectin extract were identified based on a confidence level above 95% ($P < 0.05$). Important factors and interactions identified from half-normal plot analysis were chosen to produce a first-order model for texture responses after effects and interactions were evaluated. The significance of the linear effect of the three variables was evaluated by analysis of variance (ANOVA). The coefficient of determination R^2 (pronounced R-Square) and the adjusted R^2 coefficient are used to evaluate the suitability of the model. The statistical significance of the second-order model equation is determined by the value of F which is significant and not significant-of-fit F-value [10].

Table 1. Range of variable and their coded levels independent

Factor	Name	Low Coded	High Coded
		(-1)	(+1)
A	pH	1.5	2.5
B	Temperature (°C)	60	100
C	Extraction Time (min)	60	120

3. Results and Discussions

3.1. Screening using the Two Level Factorial Design method

Screening to determine which of the three variables that affect the results of pomelo peels extract using the Two Level Factorial Design method. Variables or factors have the most significant influence on the results of pomelo peel extract. To find out the main ingredients that significantly affected the results of pomelo peels extraction, the relative significance of the three factors was investigated by a 2-level factorial design. The design consists of 24 experiments. Filtering design is used to detect factors or independent variables that have a higher impact on the response variable resulting from pomelo peels extraction. Table 2 shows the predicted level of pomelo peels extraction from experimental data. Half the normal plots can be used to determine the significant factors that affect the response. In a study of the extraction results of pomelo peels extract, the experimental design began with a screening experiment involving 3 factors which reviewed the literature having a high impact on pectin production. In this review, there are 6 factors that have been studied using 2LFD experiments as shown in Table 2 and the value of the results of pomelo peels extraction is predicted by mathematical models and experimental data.

Interpretation of research analyzed statistically using analysis of variance (ANOVA) and complete results is shown in Table 2. The probability values (P-values) of each term and interaction factors are listed in Table 3. P-values less than 0.005 mean that these factors are significant to the response under study. This is based on the confidence level set at 95%. This model is significant with a probability of <0,0001. This means that the regression model is produced to explain the correlation of the results of pomelo peels extraction with the factors tested correctly in the statistics. Table 2 Analysis of variance (ANOVA) for the results of pomelo peels extraction using a two-level factorial design. pH is one of the most important and very significant variables obtained from the results of statistical analysis through this study. Temperature and extraction time are also the next important factor identified in this statistical analysis. The importance of temperature in the development of biological processes is very clear because temperature can determine the effects of the extraction of pomelo peels.

Table 2. Analysis of the variance (ANOVA) for yield of pectin from pomelo peels using 2LFD

Source	Sum of squares	Degrees of freedom	Min square	Value > F	P>F
Model	191.14	6	31.86	96.53	<0.0001*
A	15.20	1	15.20	46.06	<0.0001
B	5.90	1	5.90	17.88	<0.0006
C	51.33	1	51.33	155.55	<0.0001
AB	54.90	1	54.90	166.36	<0.0001
AC	54.30	1	54.30	164.53	<0.0001
BC	9.50	1	9.50	28.79	<0.0001
Pure Error	5.60	16	0.35		
Cor Total	196.75	23			
R ² = 0.9715					
Adj R-Squared = 0.9614					
Std. Dev.= 0.57					

According to [11], showed that the results of extracting cocoa peels pectin with HCl solvent was the highest pectin results at pH 2.5. Likewise, the extraction temperature is very influential, where extraction temperatures of 50°C - 95°C using citric acid at pH 2.5 or 4.0 and hydrochloric acid at pH 2.5 or 4.0 increase significantly (p <0 , 05) from the results of pectin. Furthermore [12], showed that

the optimum conditions for extraction of pectin was found at a temperature of 100°C by 60 min on the base of pectin yield from lemon pomace.

Two-level factorial design studies have been used to filter out significant factors affecting the results of pectin extraction of pomelo peels. The selected and filtered factors include pH, temperature, and time of extraction. The performance of pectin extraction was studied in terms of pectin yield. The results of this experiment suggest that the proposed mathematical model can explain the capabilities of the factors studied with the determination coefficient at 0.9715 ($R^2 = 97.15\%$). Two variables tested were significant factors influencing pectin yield obtained 18.2% with P value less than 0.05 ($P < 0.05$). A half-normal plot is used to determine the significant factors affecting the response in this study. This plot determines the absolute range of factor impression and shows the symbols of factors far below the linear line is a significant factor in the response [13]. In Figure 1, a half-normal plot indicates that the factor symbols far from the linear line are a significant factor in the yields of pomelo peels pectin extract. The effect of A (pH), B (temperature), and C (extraction time) is clearly located far from the line and thus shows a strong signal. This decision is related to P-value < 0.0001 (Table 2). The next significant factor was the interaction factor between pH and temperature (AB factor), interaction between pH and extraction time (AC factor), and interaction between temperature and extraction time

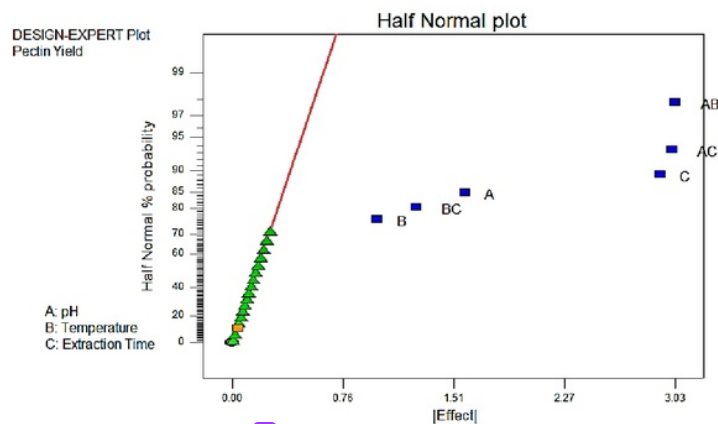


Figure 1. The plot is half normal for the effect of pH (A), temperature (B), extraction time (C) of pectin yield from pomelo peels.

3.2. Effect of Main Factors and Interaction Factors

Effect of Main Factors and Interaction Factors on the results of pomelo peels pectin extract. Responses to each of the main factors and also the interaction between factors were also examined statistically and also patented the response that these factors were also known to be definite in response. Fig.2, from the results of this study shows that there are 3 main factors that have a very statistically significant effect on the results of Pomelo peels extract, A (pH) and B (temperature). and factor C (extraction time) was found to be significant, that is, statistically giving an impression on the extraction process rather than the results of pomelo peels pectin extract in this study.

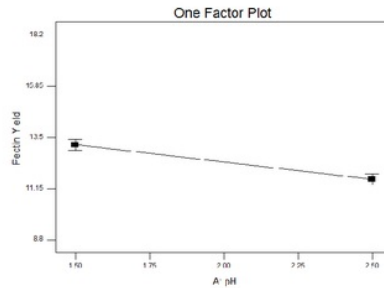


Fig. 2 a. Plot the main effect A (pH) on the results of pomelo peels pectin extract

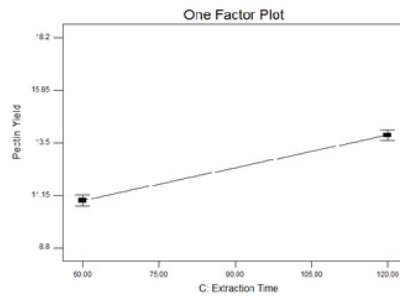


Fig. 2b. Plot the main effect B (Temperature °C) on the results of pomelo peels pectin extract

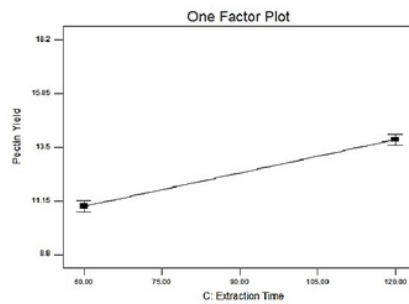


Fig.2c. Plot the main effect C (Extraction time) on the results of pomelo peels pectin extract

Figure 2 (a-c) also shows that this main factor is important in the extraction of pomelo pectin peels. Instead of Figure 2 (a-c), it can be seen that the increase in extraction activity factors in the ranking (-1 and +1) studied has had a significant effect on the results of pectin extract. Increasing the value for pH, temperature and extraction in the position studied has led to an increase in the results of pectin extract or is said to have given a significant impression of the response studied.

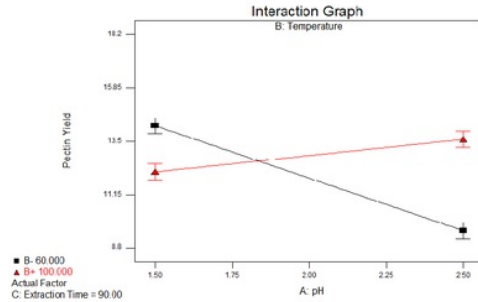


Fig. 3a. Plot significant interaction factors between pH and Temperature on the results of pomelo peels pectin extract (%)

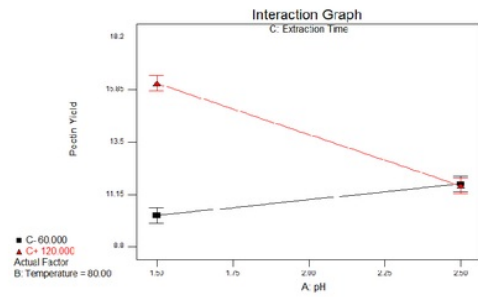


Fig. 3b. Plot significant interaction factor between pH and extraction time on yields of pomelo peels pectin extract (%)

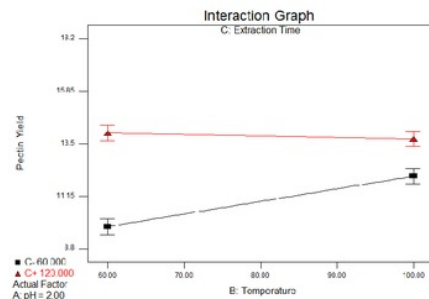


Fig. 3c. Plot significant interaction factor between temperature and extraction time on yields of pomelo peels pectin extract (%)

In addition to the impact of the main factors, the interaction between factors also shows different effects as a result of the statistical analysis that has been carried out. Figure 3a shows the interaction effect between 2 significant factors on pectin grapefruit skin extraction. The interaction of AB (pH

with temperature), interaction of AC (pH with extraction time) and interaction of BC (temperature with extraction time) had a significant effect on the responses studied with P-values <0,0001 smaller than 0.05.

Figure 3a shows the last interaction between two factors, pH (A) and temperature (B). The slope for high concentration B - (temperature of 60°C) and low concentration of B+ (temperature of 100°C) to both steep. This shows that the increase of the pH with the temperature (B -) showed the yield of pectin extract was higher.

Figure 3b shows the last interaction between two factors, pH (A) and extraction time (C). The slope for high concentration C+ (extraction time of 120 minutes) and low concentration of C- (extraction time of 60 minutes) to both steep. This shows that the increase of the temperature with the extraction time (C+) showed the yield of pectin extract was higher.

Figure 3a shows the first interaction between B (temperature) and C (extraction time). The interaction effect of BC has the interaction, where effect B is greater when B+ is at a temperature of 100°C. This plot shows that when the data is bound to B+, the effect is more flat than B -. However, in this study, the target is the result of pectin extract. Therefore, B + is the best choice based on the target. This interaction may occur because both factors contribute

4. Conclusion

The results of pectin from pomelo peels has been evaluated using a full two-level factorial Design. The effect of the operating parameters from experimental studies, the pH factor (A) and temperature (B) and extraction time (C) are identified as the most significant factor of pomelo peels pectin yield. Three interactions between AB, AC, and BC affect one factor to another in the pectin extraction process. The combination effect between A and C shows the highest percentage pectin yield contribution. Therefore, it can be concluded that the formation of pectin depends on this two factors. Therefore, pH, temperature and extraction time are those significant factors and are used in subsequent optimization experiments using a centralized composite design (CCD). The screening of this factor was aimed at identifying significant factors and subsequent research using RSM was to use these significant factors to obtain optimum pectin yields of pomelo peels in the studied stages.

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