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Optimization of Pasteurization Machine Level Settings with Heat Transfer Fluid and Pulsed Electric Field Technology Based on Randu Honey Quality Testing

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Abstract. *Randu honey is a type of monofloral honey produced by bees that consume nectar from randu flowers. Randu honey has a high water content because the flowering process occurs at night with high humidity. This research aims to determine the optimal parameter level settings in the pasteurization process of randu honey using a pasteurization machine regarding the parameters of heating temperature in the HTF, voltage at PEF, stirring speed in the tube, and PEF process time. Experiments were carried out using Response Surface Methodology (RSM) which was applied to determine the exact values of optimal process parameters that simultaneously compromise all responses. Based on the experimental results, the results of multi-objective optimization obtained optimal process parameters that can be applied to the machine is HTF temperature factor of 60°C, voltage at PEF 25kV, stirring speed in the tube 30 rpm, PEF process time 120 minutes.*

Keywords: *pasteurization; pulsed electric field; randu honey; RSM.*

I. INTRODUCTION

Honey is one of the superior forest products besides wood originating from Indonesia. In 2021, Indonesia will be able to produce 189,780 liters of honey with the largest production area being on the island of Java (BPS, 2021). However, honey produced by Micro, Small, and Medium Enterprises (MSMEs) is mostly low-quality due to a lack of post-harvest equipment support (KSDAE, 2017).

The main requirements for commercial honey must contain a diastase enzyme of at least 3 DN (diastase number), a maximum microbial content of 100 CFU/g, a maximum acidity level of 50 ml NaOH/kg, a maximum hydroxymethylfurfural (HMF) content of 40 mg/kg, and a maximum water content of 22% (SNI 8664:2018). If honey is processed using a thermal process at temperatures above 71ºC, it allows the nutritional content of the honey to be degraded and caramelization occurs (Skinner, 2019). Wibowo, et al. (2022) explained that high HMF values due to thermal processes result in low DN values which affect the honey content. Therefore, it is important to develop non-thermal pasteurization technology for food preservation that can inactivate microbes and maintain honey quality (Arshad et al., 2021).

Non-thermal pasteurization aims to reduce microbial contamination of food contents (IFT, 2020). Galanakis (2018) stated that the appropriate non-thermal pasteurization technology to be applied to MSME honey farmers in Indonesia is Pulsed Electric Field (PEF). PEF technology has the advantage of being used to process honey with a capacity of under 150 liters, does not require a lot of cost, and the equipment is easy to use (Fua'ida, 2015; Hariono, 2010). Non-thermal pasteurization uses an electric shock to reduce the amount of mold and yeast in honey without changing the taste or smell.

According to Chasanah (2001) and Evehalda (2015), there are types of yeast that can grow in conditions of low pH and high sugar concentration, causing the honey fermentation process to occur. The fermentation that occurs in honey is caused by the high water content of honey. The water content in honey can trigger the

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growth of mold and yeast which can damage the quality of the honey.

One type of honey that has a high water content is randu honey. According to Harno (2016), randu honey has a thinner viscosity than honey from other flowers because randu honey has a high water content. The high water content in randu honey is because the randu tree has a nocturnal flowering period so honey bees collect nectar at night which has low temperatures and high humidity.

Honey fermentation is caused by a type of osmophilic yeast from the genus Zygosaccharomyces, resulting in a decrease in the quality of honey, a decrease in nutritional value, and a decrease in the shelf life of honey (Illyya, Haryanti, & Suedy, 2017). Yeast activity can be inhibited by minimizing the water content of honey through the heating process (Howse, 2018). However, heating honey to high temperatures during the pasteurization process can cause an increase in HMF concentration, which is an indication of honey damage. High concentrations of HMF trigger cell and DNA damage, are mutagenic, carcinogenic, and cytotoxic so they are not consumed in large quantities (Meli et al., 2018).

Several pasteurization parameters applied include parameters voltage at PEF, temperature, stirring speed, and process time. Each applied parameter has several levels. This process uses temperature levels consisting of 24°C and 60°C. The temperature level of 24°C is room temperature or the minimum temperature which aims to maintain the content of HMF and diastase enzymes. The temperature level of 60°C in the HTF process is the maximum level limit. If honey is heated to more than 60°C it becomes unstable and thermolabile which has an impact on reducing the diastase number (DN) and its nutritional content (Razali, 2018).

The voltage levels on the applied PEF include 25, 30, and 35kV. The recommended voltage level setting on the PEF is 30kV with a pulse frequency of 20 KHz (Vadlamani et al., 2020). Set the PEF voltage at 25kV and 35kV to determine the effect of a voltage difference of 5kV on the content of randu honey. Apart from that, the PEF voltage setting of 35kV is the maximum voltage limit on the pasteurization machine. Stirring speed level settings include 20, 25, and 30rpm.

The selection of the stirring speed setting of 25rpm is based on calculating the angular speed, fluid speed, and the rotation period of the stirrer propeller on the elasticity of the honey stirrer to avoid spills and the risk of caramelization. Stirring speed settings of 20rpm and 30rpm were used to determine the effect of a difference in stirring speed of 5rpm on the content of randu honey.

In this research, all process parameters are a combination of several pasteurization processes for randu honey. Optimization of parameter settings by considering four responses simultaneously is used to sterilize honey, increase shelf life, and maintain the smell and taste that are characteristic of honey. Response Surface Methodology (RSM) was chosen to estimate the level of pasteurization process parameter settings as a new proposal that is easy to implement on factors with few levels. Combination of factors with continuous values using RSM in optimizing parameter settings in the pasteurization process of randu honey including temperature at HTF, voltage at PEF, stirring speed in the tube, and PEF process time to reduce mold and yeast levels, water content, HMF, and increase enzyme activity diastase.

II. RESEARCH METHOD

This research was conducted at the Production Planning and Design Laboratory, Industrial Engineering Department, Faculty of Engineering, Sebelas Maret University. First, we defined the responses, factors, and levels of this research. The response in this research was the quality of pasteurized randu honey which included mold and yeast content, water content, HMF, and diastase enzyme activity value. This research considered four factors, including heating temperature in the HTF, voltage in the PEF, stirring speed in the tube, and PEF processing time. Table 1 shows the factors and their respective levels.

Table 1. Setting Factor Levels for Randu Honey Pasteurization Experiment

Setting Factor and Level				
Factor	Lvl 1	Lvl 2	Lvl 3	Lvl 4
HTF (°C)	24	60		
PEF (kV)	0	25	30	35
Stirring speed (rpm)	0	20	25	30
Processing time (minutes)	0	120	180	240

Table 2. Data Sample Randu Honey

Sample	Temp (°C)	PEF (kV)	Stirring Speed (rpm)	Processing Time (minutes)
MM	24	0	0	0
MP2	24	30	0	120
MP3	60	30	0	120
MP4	60	25	20	120
MP5	60	25	20	180
MP6	60	25	20	240
MP7	60	25	25	120
MP8	60	25	25	180
MP9	60	25	25	240
MP10	60	25	30	120
MP11	60	25	30	180
MP12	60	25	30	240
MP13	60	30	20	120
MP14	60	30	20	180
MP15	60	30	20	240
MP16	60	30	25	120
MP17	60	30	25	180
MP18	60	30	25	240
MP19	60	30	30	120
MP20	60	30	30	180
MP21	60	30	30	240
MP22	60	35	20	120
MP23	60	35	20	180
MP24	60	35	20	240
MP25	60	35	25	120
MP26	60	35	25	180
MP27	60	35	25	240
MP28	60	35	30	120
MP29	60	35	30	180
MP30	60	35	30	240

These four parameters were combined to produce 30 types of treatment. Table 2 is sample data from the honey pasteurization testing process using a PEF machine.

In this research, honey was tested using non-thermal PEF pasteurization technology using a pasteurization machine designed according to honey testing needs. The pasteurization machine frame is made of aluminum extrusion with a tube that can hold 80 liters, has a 600W electric heater

Figure 1. Pulsed Electric Field Honey Pasteurization Machine

and the PEF intensity is regulated via an analog system for PEF modulation. The PEF pasteurization machine used for processing honey in this research is shown in Figure 1.

Response Surface Methodology

Response surface methodology (RSM) is a method developed for response models (y) with more than one factor variable. RSM aims to optimize responses by modeling and analyzing responses that are influenced by several variables (Montgomery, 2001). The equation model for each response in determining the optimal level setting in this research is a regression model using Minitab 2018 software.

An experiment that uses k factors X1, X2, ..., Xk with Y the response variable. The variables used can be measured and it is known that Y is partially or completely a response to X1, This is a response surface where for k=1 we get Y = f(X) which is called the response line. In determining the response surface equation, several models have been formulated so that the experiments carried out can be as close as possible to the equation. In terms of two dimensions, the first order and second order response equations are shown in equations (1) and (2).

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_{12} + \cdots + \beta_k x_k + \epsilon \quad (1)$$

$$y = \beta_0 + \sum_{i=1}^{k} \beta_1 x_1 + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum\sum_{i<j} \beta_{ij} x_i x_j + \epsilon \quad (2)$$

The interaction relationship between controlled factors and noise factors is one of the keys to a strong design. So we need a logical model of a response that includes these two factors with equations is

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + y_1 z_1 + \delta_{21}\beta_1 x_1 + \delta_{21} x_2 z_1 + \epsilon \quad \textbf{(3)}$$

The model above has the main influence of these two factors which is usually called the model response. Myer and Montgomery (2001) describe a multi-response method called desirability. Desirability is an objective function from the limit 0 to 1. For several responses and all level factors it is intended to be combined into a desirability function, D(x) with the equation

$$D = (d_1 \times d_2 \times d_3 \times \ldots \times d_n)^{\frac{1}{n}} \quad \textbf{(4)}$$

where n is the number of responses analyzed. For optimization, each response must have a high and low value with a maximum - minimum goal value, target or within a certain range.

III. Result and Discussion

The pasteurization process of randu honey is carried out using a pasteurization machine with heat transfer fluid (HTF) and pulsed electric field (PEF) technology. In this research, there were four test responses that influenced the quality of randu honey, including testing the number of molds and yeasts, water content, HMF, and diastase enzyme activity.

Based on the pasteurization test of randu honey which was carried out using a pasteurization machine with PEF technology, a sample of honey pasteurization results was obtained as shown in figure 2.

From the results of the tests that have been carried out, it shows that samples of randu honey that have not been processed using HTF and PEF contain mold and yeast numbers above 1 x 102 colonies/g (do not meet SNI for honey). This proves that the combination of HTF and PEF provided is able to inactivate the maximum number of molds and yeasts.

Randu honey processed at a heating temperature of 24°C has a higher water content than that processed at a temperature of 60°C.

Table 3. Response Results of Randu Honey

Sample	Mold and Yeast Numbers (coloni/g)	Water Containt (%)	HMF (mg/kg)	Diastase Enzyme (DN)
MM	1x102	18,00	20,36	15
MP2	<1x102	18,00	25,00	12
MP3	<1x102	17,40	26,65	10
MP4	<1x102	17,20	29,78	10
MP5	<1x102	17,00	31,28	10
MP6	<1x102	16,40	31,91	8,57
MP7	<1x102	17,20	28,42	12
MP8	<1x102	17,00	29,95	12
MP9	<1x102	16,80	31,14	10
MP10	<1x102	17,20	26,65	12
MP11	<1x102	17,00	29,04	12
MP12	<1x102	16,80	30,09	10
MP13	<1x102	17,20	30,84	10
MP14	<1x102	17,00	31,29	8,57
MP15	<1x102	16,40	32,63	7,5
MP16	<1x102	17,20	29,94	12
MP17	<1x102	17,00	31,13	10
MP18	<1x102	16,80	31,30	8,57
MP19	<1x102	17,20	28,14	12
MP20	<1x102	17,00	29,93	12
MP21	<1x102	16,80	30,84	10
MP22	<1x102	17,20	31,32	8,57
MP23	<1x102	17,00	32,18	8,57
MP24	<1x102	16,40	33,52	7,5
MP25	<1x102	17,20	30,88	10
MP26	<1x102	17,00	31,73	10
MP27	<1x102	16,80	33,41	8,57
MP28	<1x102	17,20	29,79	12
MP29	<1x102	17,00	31,46	10
MP30	<1x102	16,80	32,48	8,57

Figure 2. Samples of Raw Randu Honey (MM) and Pasteurized Honey (MP)

This proves that heating temperature can reduce the water content due to the evaporation process. Apart from that, randu honey processed with a stirring speed of 20 rpm has a lower water content than 30 rpm. This happens because the stirring process can evenly distribute the heating temperature throughout all parts of the honey without causing the release of excess heat due to the speed of the stirring rod.

Table 4. ANOVA of Mold and Yeast Number Responses

ANOVA for Response Mold and Yeast Number		
Source	Adj SS	P-Value
Model	7830.0	0,00
Linear	6324.75	0,00
Temp	23.28	0,00
PEF	64.96	0,00
Stir speed	0.00	0,00
Time process	0.00	0,00
Square	0.00	0,00
PEF*PEF	0.00	0,00
Stir speed*stir speed	0.00	0,00
Time proces*Time proces	0.00	0,00
2-Way Interaction	35.10	0,00
Temp*PEF	23.74	0,00
PEF*Stir speed	0.00	0,00
PEF*Time process	0.00	0,00
Stir speed*Time process	0.00	0,00
Error	0.00	
Total	7830.00	

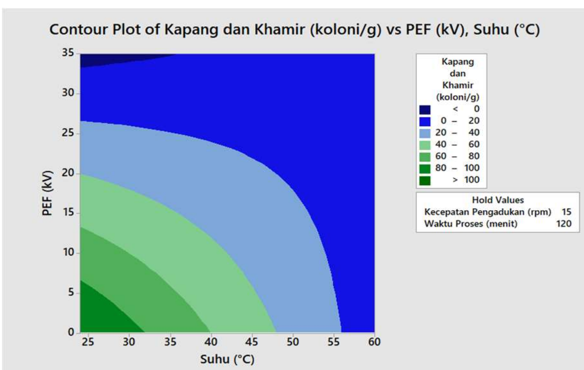

Figure 3. Contour Plot Response Numbers of Mold and Yeast

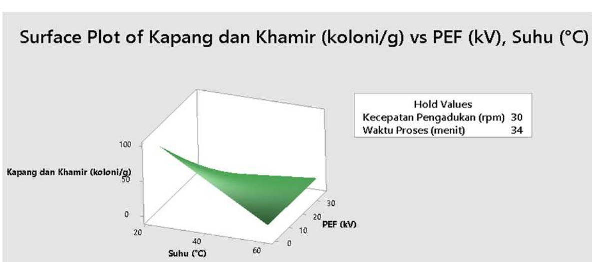

Figure 4. Response Surface Plot Numbers of Mold and Yeast

Randu honey processed at a heating temperature of 24°C tends to have a lower HMF content than that processed at a temperature of 60°C. Apart from that, honey processed with a PEF voltage of 35kV within 240 minutes also produces higher HMF content. This is because the higher heating temperature, PEF voltage, and longer processing time given in processing honey can trigger an increase in glucose degradation, thereby increasing the HMF content in honey.

Randu honey processed without pasteurization has a higher diastase enzyme value compared to that processed using HTF and PEF. The higher the heating temperature and the PEF voltage applied can damage the enzyme cell walls which results in the value of the diastase enzyme in honey decreasing. Apart from that, slower stirring speeds and longer processing times also cause a decrease in the diastase value because the heat and electric voltage received by all parts of the honey are evenly distributed.

A summary of the fit and ANOVA output of the response to mold and yeast numbers was carried out by testing the model on two equations, linear and quadratic for each response. Table 4 contains the results of processing response data mold and yeast numbers with RSM method using Minitab 2018.

The data is visualized using a contour plot as in Figure 3 and a response surface plot of mold and yeast numbers as in Figure 4 which shows the response of each parameter.

Table 5 contains the results of processing water content response data with RSM method using Minitab 2018 software.

Table 5. ANOVA of Water Content Responses

ANOVA for Response Water Content		
Source	Adj SS	P-Value
Model	3,67214	0,000
Linear	1,33518	0,000
Temp	0,00063	0,763
PEF	0,01003	0,235
Stir speed	0,00255	0,544
Time process	0,07790	0,003
Square	0,02684	0,292
PEF*PEF	0,00002	0,958
Stir speed*stir speed	0,00038	0,813
Time proces*Time proces	0,02596	0,064
2-Way Interaction	0,13939	0,006
Temp*PEF	0,00300	0,511
PEF*Stir speed	0,00000	1,000
PEF*Time process	0,00000	1,000
Stir speed*Time process	0,13936	0,000
Error	0,11986	
Total	3,79200	

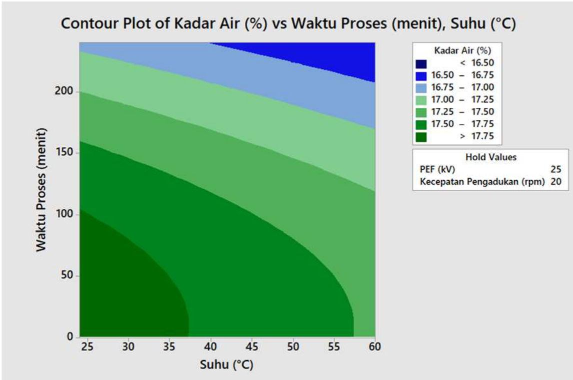

Figure 5. Contour Plot Response Water Content

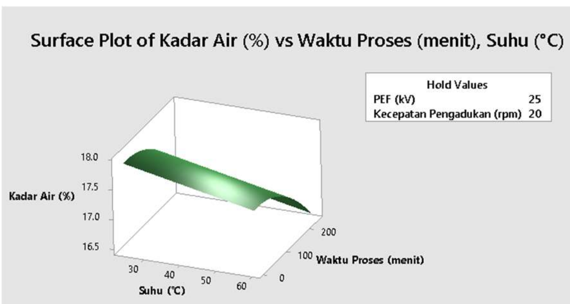

Figure 6. Response Surface Plot Water Content

Table 6. ANOVA of HMF Responses

ANOVA for Response HMF		
Source	Adj SS	P-Value
Model	207,160	0,000
Linear	117,949	0,000
Temp	0,680	0,025
PEF	0,352	0,096
Stir speed	0,562	0,039
Time process	1,071	0,007
Square	16,440	0,000
PEF*PEF	0,171	0,236
Stir speed*stir speed	15,209	0,000
Time proces*Time proces	0,159	0,253
2-Way Interaction	1,850	0,016
Temp*PEF	0,375	0,086
PEF*Stir speed	1,260	0,004
PEF*Time process	0,064	0,462
Stir speed*Time process	0,523	0,046
Error	2,048	
Total	209,208	

To make it easier to see the results of the discussion based on the resulting equations, the data is visualized using a contour plot as in Figure 5 and a response surface plot of water content as in Figure 6 which shows the response of each parameter.

Table 6 contains the results of processing HMF response data with RSM method using Minitab 2018 software.

To make it easier to see the results of the discussion based on the resulting equations, the data is visualized using a contour plot as in Figure 7 and a response surface plot of HMF as in Figure

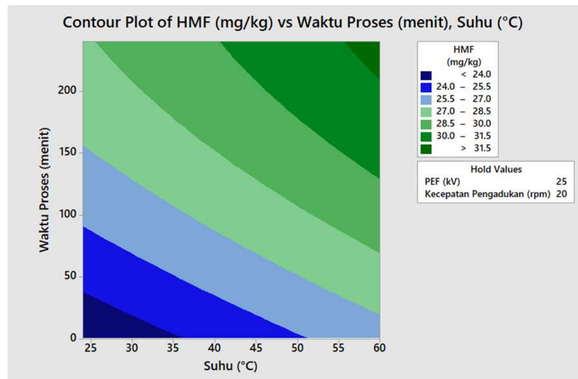

Figure 7. Contour Plot Response HMF

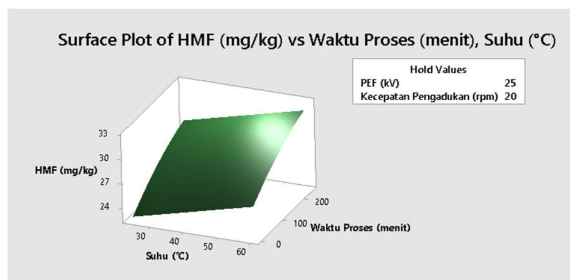

Figure 8. Response Surface Plot HMF

Table 7. ANOVA of Diastase Enzyme Activity Responses

ANOVA for Response Enzyme Diastase		
Source	Adj SS	P-Value
Model	77,7983	0,000
Linear	26,5773	0,000
Temp	0,2225	0,485
PEF	0,5729	0,268
Stir speed	0,1578	0,556
Time process	0,0286	0,801
Square	5,9902	0,015
PEF*PEF	0,0292	0,799
Stir speed*stir speed	4,8156	0,004
Time proces*Time proces	1,2660	0,106
2-Way Interaction	0,2968	0,951
Temp*PEF	0,0381	0,771
PEF*Stir speed	0,0208	0,830
PEF*Time process	0,0208	0,830
Stir speed*Time process	0,2227	0,485
Error	7,8880	
Total	85,6863	

8 which shows the response of each parameter.

Table 7 contains the results of processing diastase enzyme activity response data with RSM method using Minitab 2018 software.

Based on the resulting equations, the data is visualized using a contour plot as in Figure 9 and a response surface plot of diastase enzyme activity as in Figure 10 which shows the response of each parameter.

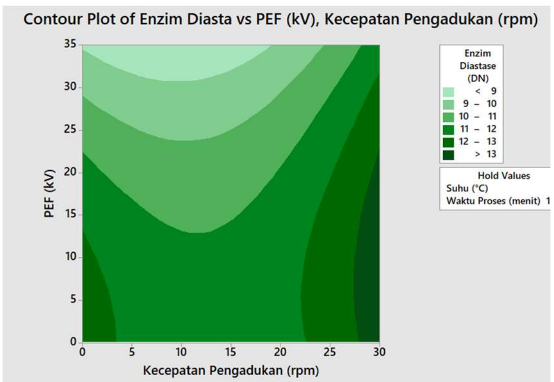

Figure 9. Contour Plot Response Diastase Enzyme Activity

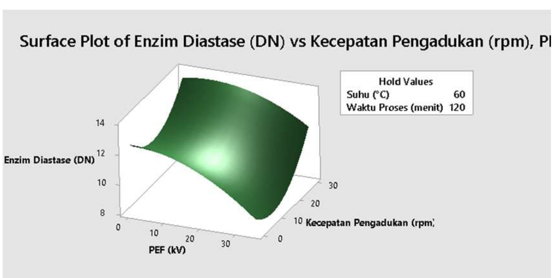

Figure 10. Response Surface Plot Diastase Enzyme Activity

Table 8. Model Summary

Model Summary			
	Standard Deviation	R2	Adjusted R2
Mold and Yeast Numbers	0	100%	100%
Water content	0,0816005	96,84%	94,91%
HMF	0,337319	99,02%	98,42%
Enzyme Diastase	0,661984	90,79%	85,17%

The summary fit results output clearly shows that the second order polynomial provides relatively good results for each response from the Minitab 2018 software. By eliminating factors that are not significant in each response, the model for each response can be expressed as follows:

$$Y_1 = 160 - 2.5\ X_1 - 5\ X_2 + 0.08333\ X_1 * X_2$$

$$Y_2 = 17{,}74 + 0{,}0110X_1 + 0{,}0605X_2 - 0{,}0482X_3 - 0.00677X_4 - 0.00007X_2^2 - 0.000068X_3^2 - 0{,}000018X_4^2 - 0{,}00094X_1*X_2 + 0{,}000356X_3*X_4$$

$$Y_3 = 11{,}69 + 0{,}361X_1 + 0{,}149X_2 - 0{,}016X_3 + 0{,}0264X_4 + 0{,}00675X_2^2 - 0{,}01352X_3^2 - 0{,}000045X_4^2 - 0{,}01047X_1*X_2 + 0{,}01296X_2*X_3 - 0{,}000244X_2*X_4 + 0{,}000689X_3*X_4$$

$$Y_4 = 18{,}85 - 0{,}160\ X_1 - 0{,}192\ X_2 - 0{,}144\ X_3 + 0{,}0433\ X_4 - 0{,}0028\ X_2^2 + 0{,}00761\ X_3^2 - 0{,}000128\ X_4^2 + 0{,}0033\ X_1*X_2 + 0{,}00167\ X_2*X_3 - 0{,}000139\ X_2*X_4 - 0{,}000449\ X_3*X_4$$

Y_1 = Response of Mold and Yeast Numbers
Y_2 = Response of Water Content
Y_3 = Response of HMF
Y_4 = Response of Diastase Enzyme Activity
X_1 = Temperature
X_2 = Voltage at PEF
X_3 = Stirring speed
X_4 = Processing time

The resulting response surface equations for the four responses were then optimized for factor level settings. The desirability function value was calculated using Minitab 2018 software, to obtain a solution for setting factor levels with a combination of four continuous responses. The optimal desirability value at the HTF temperature factor is 60°C, the voltage at the PEF is 13kV, the stirring speed in the tube is 30 rpm, and the PEF process time is 34 minutes at 0.6274 as shown in Figure 11. Meanwhile, the optimal desirability value for the HTF temperature factor is 60°C, the voltage at the PEF is 25kV, the stirring speed in the tube is 30 rpm, and the PEF process time for 120 minutes is 0.4138 as shown in Figure 12.

After processing the data using RSM, the most optimal desirability value was obtained at 0.6274 with a HTF temperature factor of 60°C, a voltage at the PEF of 13kV, a stirring speed in the tube of 30 rpm, and a PEF processing time of 34 minutes. However, a PEF voltage of 13kV cannot be applied to a pasteurization machine due to

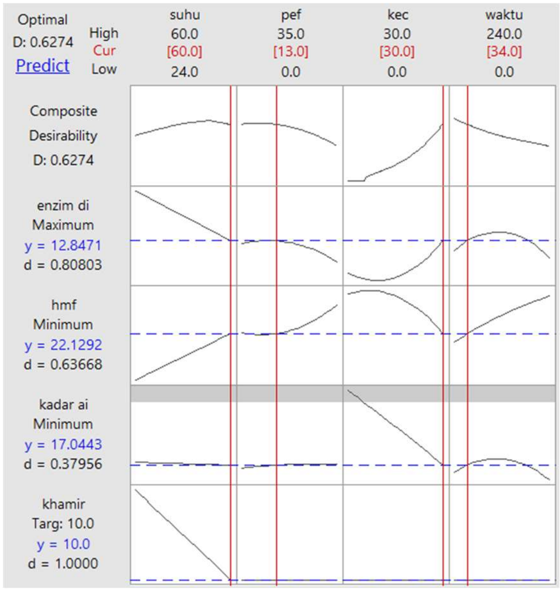

Figure 11. Desirability I

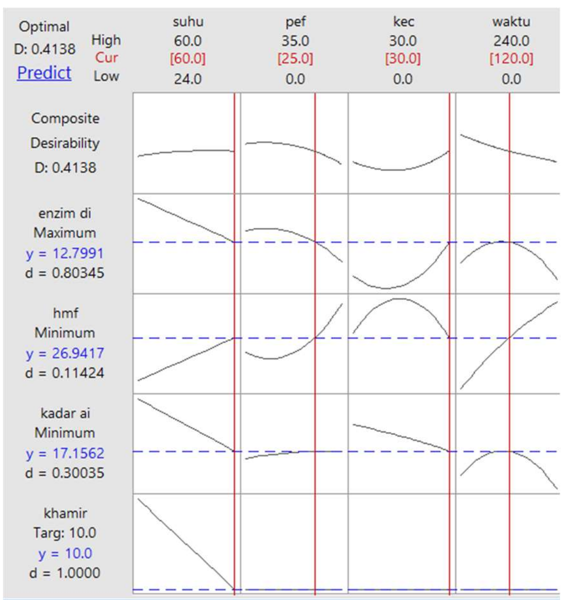

Figure 12. Desirability II

limited machine capabilities. Therefore, desirability is re-determined as an optimal value that has been adjusted to the capabilities of the pasteurization machine. The optimal desirability value that has been adjusted is 0.4138 at a HTF temperature factor of 60°C, voltage at PEF 25kV, stirring speed in the tube of 30 rpm, and PEF process time of 120 minutes.

Based on the comparison of the two response optimizations using RSM, different desirability values were obtained. This difference in desire is due to the greater the PEF voltage and the longer the PEF processing time, which has an impact on increasing the HMF value. This happens because there is a combination of the HTF and PEF processes which can increase glucose degradation thereby increasing the HMF value. A high HMF value in honey will reduce the quality of honey because the HMF content is related to several other chemical properties of honey such as water content, pH, free acid content, reduced sugar content, and enzymatic activity in honey (Kowalski et. al., 2013).

IV. Conclusion

The optimal level setting desirability value using the RSM method in the first response test is 0.6274 with the optimal level setting at a HTF temperature factor of 60°C, a voltage on the PEF of 13kV, a stirring speed on the tube of 30 rpm, and a PEF process time of 34 minutes.

The optimal level setting desirability value using the RSM method in the second response test is 0.4138 with the optimal level setting at a HTF temperature factor of 60°C, a voltage on the PEF of 25kV, a stirring speed on the tube of 30 rpm, and a PEF process time of 120 minutes.

The level setting in the first response test cannot be applied in the pasteurization machine due to limited machine capabilities. Therefore, the machine level setting that is possible to apply is in the second response test with an optimal desirability value of 0.4138

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