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Optimization of process conditions for the application of edible coating emulsion on guava (Psidium guajava) using response surface methodology

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***Abstract*—Response surface methodology (RSM) was employed to search for the best conditions of applying edible coating emulsion on guava surface via a dipping technique. The two parameters which affect the coating process conditions the most were identified as temperature of coating emulsion and dipping time. Based on central composite rotatable designs (CCRD) of RSM and coating pickup as a response, 9 coating conditions were established involving 4 factorial points, 4 axial points and 1 center point. From the RSM-generated model, optimum coating conditions for minimizing coating pickup was identified as temperature of coating emulsion (63°C) and dipping period (15s). Under this optimum condition, the predicted coating pickup of coated guava and the experimental results gave close values of about 0.15%. The RSM-predicted and experimental coating pickup values were not significantly different from each other.**

Keywords-RSM, guava, edible coating, coating pickup.

I. INTRODUCTION

Guava (*Psidium guajava*) is a tropical, climacteric fruit that ripens rapidly and is highly perishable. The shelf-life ranges from 3 to 10 days at room temperature [1, 2]. If the guavas are kept without any treatment, they may be spoiled mainly due to loss of water from fruit surface, faster respiration rate, attack of microorganisms and the development of physiological disorders. The application of edible coating represents a method that can extend the shelf life of picked guava by minimizing the weight loss due to natural migration process of moisture and gases.

Various types of coating condition have been reported widely involving different types of coating materials as well as temperature and dipping time such as the coating of sweet cherry [3], strawberry [4], avocado [5] and kiwi fruit [6]. The applied temperature varied from ambient to elevated temperature and dipping time within seconds to minutes.

In our previous study, we have already developed a suitable coating emulsion based on palm oil products and successfully prolonged the shelf life of coated guava [7]. However, the effectiveness of coating and fruit acceptability are greatly affected by the temperature of coating emulsion and the dipping time. Dipping at low temperature may increase the coating thickness and consequently may lead to the formation of oily surface and undesirable effects such as anaerobic respiration and fermentation to the coated guava. Dipping at higher temperature may cause physical injury or scalding effects to the guava surface. Moreover, high temperatures up to 70°C may also cause evaporation effects to the coating emulsion. Meanwhile, dipping time affects the productivity of coating process. Short period of dipping may increase the productivity whereas long period may decrease the productivity of coating process. In order to search for optimum level of temperature and dipping time, response surface methodology (RSM) may be a very useful approach to obtain as minimum as possible the coating pickup.

RSM enables the evaluation of the effects of many factors and their interactions on response variables. The advantages of using RSM are reported to be the reduction in the number of experimental runs needed to evaluate multiple variables, and the ability of the statistical tool to identify interactions [8, 9]. Therefore, it is less laborious and time-consuming compared to one-variable at a-time. RSM has been widely applied for optimizing conditions and processes in various food studies [10, 11].

To the best of the author's knowledge, there is no paper so far discussing about the optimization of process conditions for applying edible coating emulsion formulated from palm oil products on guava surface. In this study, all lipid-based coating ingredients were emulsified with water to form coating emulsion and subsequently applied on fruit surface by dipping technique. The central composite rotatable design (CCRD) of RSM was used as a statistical method to optimize the process conditions of applying edible coating emulsion in minimizing the coating pickup of guava.

II. MATERIALS AND METHOD

A. *Guava*

Samples of fresh guavas were obtained from a commercial farm, Sui Yuan Fruit Trading, in Bidor, Perak, Malaysia. The guavas were carefully selected (maturity index 2) to obtain uniformity based on weight (400-405 g), size, shape, colour and absence of injuries. The fruits were packed in small boxes and transported to the laboratory in Shah Alam, Selangor, Malaysia. Pre-treatment of guavas were made according to the method applied by [12]. Selected guavas were unwrapped, washed and sanitized in potassium sorbate 0.5% for 2-3 minutes and left to dry at ambient conditions (25-27°C, RH 80-90%). Guavas were labeled before being dipped in the coating emulsion at desired temperature and dipping time.

B. Coating Ingredients

Refined, bleached and deodorised palm kernel olein and palm stearin with a slip melting point of 49.6°C and iodine value 40.3 were obtained from Cargill Palm Products Pte. Ltd., Port Klang, Malaysia. Beeswax (Sigma-Aldrich, GmbH, Sternheim, Germany) was purchased from a local supplier. All other chemical and ingredients used were either of analytical or food grade.

C. Preparation and Application of Coating Emulsion

The coating emulsion was prepared conceptually similar to the method applied by several researchers [13, 14]. Beeswax (1% w/v) was heated up to 90°C in distilled water while being stirred on hot plate (Fisher Scientific, USA) until the solution became clear. Palm stearin (4.5% w/v), palm olein (1% w/v) and emulsifier (Tween 40, 0.5% v/v, Sigma-Aldrich, GmbH, Sternheim, Germany) were added immediately followed by high speed mixing using an Ultra Turax T10 (IKA®, Germany). The coating emulsions were cooled while stirred to the desired temperature before applying to the fruit.

D. Coating Pickup

Coating pick-up was determined by the difference in weight of guava before and after the coating process. The coated fruit was consistently dried under forced-air of a table fan for 5 minutes before weight measurement. The procedure for measurement of coating pick-up has been described by previous study [15, 16].

E. Experimental Design and Statistical Analysis

The process condition of coating application was optimized using the CCRD of RSM software (Design-Expert Version 6.0.4 Stat-Ease Inc., Minneapolis, USA). Each variable was examined at five different levels (relatively low, low, basal, high, relatively high) coded (--, -, 0, +, ++) as shown in Table 1. The design required 29 runs derived from 9 combinations of the independent variables performed in random order, including replicates of the centre region and factorial points. The responses obtained were subjected to an analysis of variance (ANOVA), R-square and were evaluated for lack of fit (LOF). Accordingly, an equation (1) in terms of coded of second-order polynomial could be calculated of the type:

$$Z = \beta_o + \beta_1 A + \beta_2 B + \beta_{12} AB + \beta_{11} A^2 + \beta_{22} B^2 \qquad (1)$$

where, Z was the dependent variable (coating pickup); A and B were the independent variables for temperature of coating emulsion and dipping time, respectively; β_0 was the regression coefficient at center point; β_1, and β_2 were linear coefficients; β_{12}, was second-order interaction coefficients; and β_{11}, and β_{22} were quadratic coefficients. Optimum composition was obtained using the optimization module of RSM software. The experimental and predicted values were compared in order to determine the validity of the developed model. The verification of model was performed similar to the method applied by previous study [17].

Statistical Analytical software SAS was applied at significance level 0.05. Values followed by the same letter are not significantly different.

III. RESULTS AND DISCUSSION

Details about the experimental runs, temperature of coating emulsion, dipping time as well as the percentage of coating pick-up were tabulated in Table 1.

TABLE 1. COATING PICKUP AS A FUNCTION OF TEMPERATURE AND DIPPING TIME OF EDIBLE COATING EMULSION.

Standard	Run	A:Temperature (°C)	B: Dipping time (s)	Z: Coating pickup (%)
1	19	50 (-)	10 (-)	1.09
2	14	50 (-)	10 (-)	0.96
3	9	50 (-)	10 (-)	0.99
4	29	70 (+)	10 (-)	0.26
5	26	70 (+)	10 (-)	0.30
6	22	70 (+)	10 (-)	0.28
7	23	50 (-)	30 (+)	1.13
8	27	50 (-)	30 (+)	1.11
9	5	50 (-)	30 (+)	1.19
10	21	70 (+)	30 (+)	0.30
11	4	70 (+)	30 (+)	0.41
12	25	70 (+)	30 (+)	0.43
13	17	45.86 (--)	20 (0)	1.32
14	13	45.86 (--)	20 (0)	1.39
15	18	45.86 (--)	20 (0)	1.40
16	11	74.14 (++)	20 (0)	0.35
17	24	74.14 (++)	20 (0)	0.29
18	16	74.14 (++)	20 (0)	0.40
19	1	60 (0)	5.86 (--)	0.42
20	3	60 (0)	5.86 (--)	0.37
21	20	60 (0)	5.86 (--)	0.33
22	12	60 (0)	34.14 (++)	0.52
23	10	60 (0)	34.14 (++)	0.62
24	15	60 (0)	34.14 (++)	0.69
25	2	60 (0)	20 (0)	0.25
26	6	60 (0)	20 (0)	0.23
27	28	60 (0)	20 (0)	0.29
28	7	60 (0)	20 (0)	0.24
29	8	60 (0)	20 (0)	0.21

A. Model Fitting and Analysis of Response

A good process condition should be able to minimize the percentage of coating pickup after five minutes of drying period. Table 2 presents the summary of the results for fitting a model. Fitting of the data to various models (i.e. linear, two factorial interactions (2FI), quadratic and cubic) showed that the reactions of temperature of coating emulsion and dipping time in minimizing coating pickup were most suitably described by a quadratic polynomial model.

TABLE 2. RESULTS SUMMARY OF FITTING A MODEL IN THE OPTIMIZATION OF COATING CONDITIONS.

Source	Sequential P-value	LOF P-value	R-Squared	Adj R-Squared	PRESS
Linear	<0.0001	<0.0001	0.7230	0.7017	1.51
2FI	0.9099	<0.0001	0.7231	0.6899	1.55
Quadratic	<0.0001	0.3285	0.9859	0.9829	0.10
Cubic*	0.4324	0.1897	0.9870	0.9827	0.12

* identified as aliased model.

The LOF test measured a variation of the data around the fitted model. If the model did not fit the data well, the LOF test would be significant. A model should be rejected if the result showed significance in the LOF test [18]. The analysis of variance to the models source revealed that the LOF test for the quadratic model was insignificant with a high of P-value (Probability>F) of 0.3285. This suggested that the quadratic polynomial model was statistically acceptable and could be used to predict the new response. Both linear and 2FI models showed significant results in the LOF test with a small P-value (Probability>F) of less than 0.0001, which indicated that the models were not statistically suitable and should be neglected. The cubic model was aliased (to the quadratic model) due to not enough experiments have been run to independently estimate all the terms for this aliased model. Thus, the cubical model would be simply neglected for further discussion.

The coefficient of determination, R-squared for the quadratic model was satisfactorily high at 0.9859 which also reflected the degree of fit of the model [19]. Further model reduction by removing the insignificant model terms might improve the values of R-squared and adjusted R-squared in the quadratic polynomial model. In the meantime, the predicted residual sum of square (PRESS) value measures how the model fits each point in the design. The lower the value of PRESS, the better the model fits the point. The quadratic model showed a relatively lower PRESS values than the other models. Lowest PRESS value supports the decision to select the quadratic polynomial model for further in depth study.

B. Model Reduction

Table 3 shows the results of the ANOVA for the reduced quadratic model. By analyzing the difference between the initial model and the reduced model especially on the probability value, the term AB (2FI between temperature of coating emulsion and dipping time) was statistically identified as an insignificant model term. This was evidenced by the high value of probability>F (0.6310) when the term AB was included in the model. Reference [18] suggested that non-significant term can be dropped from a model or fixed at one level. Thus, we had decided to remove the term AB in order to improve the quadratic model.

Further analysis with the LOF test revealed that the higher value of probability>F was observed in the reduced model (0.4429) than that of the initial model (0.3285). This could also indicate the reduced quadratic model fits the data better than the initial model.

TABLE 3. ANOVA CALCULATED FROM THE REDUCED CCRD MODEL IN THE OPTIMIZATION OF COATING CONDITIONS.

Source	Sum of squares	df	Mean square	F value	Prob > F
Model	4.59	4	1.15	416.45	<0.0001
A	3.25	1	3.25	1178.33	<0.0001
B	0.12	1	0.12	43.36	<0.0001
A^2	1.22	1	1.22	441.26	<0.0001
B^2	0.21	1	0.21	76.73	<0.0001
Residual	0.066	24	2.76×10^{-3}		
Lack of fit	0.011	4	2.70×10^{-3}	0.98	0.4429
Pure error	0.055	20	2.77×10^{-3}		
Cor total	4.66	28			

The statistical summary of the reduced model comprised of several important properties as listed in Table 4. Reference [20] suggested that the coefficient of variance (CV) should be less than the standardized value of 10%. The CV value (8.57%) of the reduced model was below than that of the standardized value. In the case of PRESS value, when the term AB was dropped the PRESS value decreased to 0.097, indicating that the term AB should be dropped from the model. Reference [20] suggested that for a good fit of a model, R^2 should be at least 0.80. The R-squared value implied that 98.58% of the variations could be explained by the reduced model. Adjusted R-Square was used to measure the amount of variation around the mean which was adjusted for the number of terms in the model. The adjusted R-Square decreased as the number of terms in the model increased if those additional terms did not add value to the model. For instance, when the term AB was included, the adjusted R-Square decreased from 0.9834 to 0.9829, which indicated that the term AB did not contribute significantly to the response. The adequate precision measures the signal to noise ratio and a ratio higher than 4 indicates an adequate signal [18]. The value of adequate precision (52.70) of the reduced model was higher than that minimum value (4) and therefore the model can be used to navigate the design space.

TABLE 4. STATISTICAL SUMMARY OBTAINED FROM THE REDUCED CCRD MODEL IN THE OPTIMIZATION OF COATING CONDITIONS.

Parameters	Value
Standard deviation	0.053
Mean	0.61
C.V	8.57
PRESS	0.097
R-Squared	0.9858
Adjusted R-Squared	0.9834
Predicted R-Squared	0.9792
Adequate Precision	52.705

The reduced model can also be represented by a coded equation as shown in (2). The equation is used to correlate a relationship between the two variables and the percentage of coating pickup.

$$Z = 0.24 – 0.37A + 0.07B + 0.31A^2 + 0.13B^2 \tag{2}$$

C. *Diagnostic and Optimum Coating Composition*

Fig. 1 shows the relationship between the coating pickup, temperature of coating emulsion and dipping time in the form of three-dimensional plot. The plot accommodate a spherical region with the lowest value of coating pickup could be observed at temperature range from 60-70°C and dipping time of 10-25 seconds. The highest value of coating pickup could be observed at temperature and dipping time ranging from 50-55°C and 25-30 seconds, respectively.

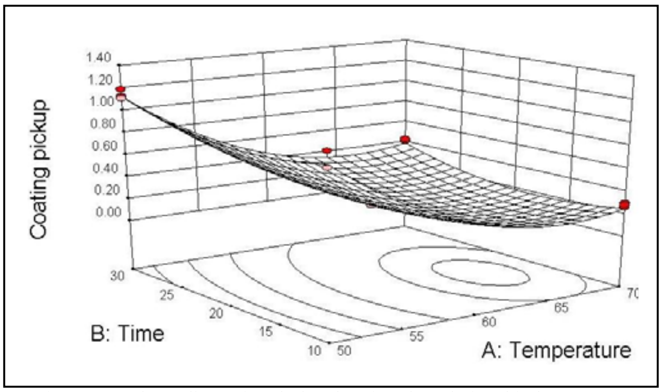

Figure 1. Three-dimensional plot showing the relationship between coating pickup as affected by the temperature of coating emulsion and dipping time.

In searching for the 'best' and optimum coating condition, proper consideration should be given to the required outcomes such as the guava surface should be covered as minimum as possible by the coating materials. In view of the above considerations, it was therefore decided to set a minimum goal for coating pickup with the application temperature of coating emulsion preferably not exceeding 65°C and the dipping time as short as possible. With these conditions set, the RSM software automatically generated many solutions for the optimized coating conditions as shown by some examples in Table 5.

TABLE 5. SOME EXAMPLES OF OPTIMIZED COATING CONDITIONS GENERATED BY THE RSM SOFTWARE BASED ON THE REDUCED CCRD MODEL.

Solution number	Temperature (°C)	Dipping time (s)	Coating pickup (%)	Desirability
1	62.75	21.75	0.18	1.00
2	63.14	15.17	0.15	1.00
3	62.39	18.72	0.17	1.00

Solution number 2 had been chosen based on the above considerations and the small variations between the solutions number. The optimum coating conditions for minimizing coating pickup was identified as temperature of coating emulsion 63°C and dipping time 15 second. Using this optimized coating condition, the predicted response of coating pickup was about 0.15%. It should also be mentioned that the desirability value of all solutions showed satisfactory good values. The value can range from zero to one and it should only be evaluated relative to the upper and lower limits that were chosen for the responses and variables. In this case, upper and lower limits of all variables were set according to the ranges of study while the coating pickup was set to be at minimum.

D. *Model Verification*

Model verification was performed with additional three sets of independent trials using the mentioned optimized coating conditions and compared to the predicted value from the CCRD model. The result of the validation experiment is presented in Table 6. The predicted value of coating pickup was about 0.15% as suggested by the CCRD model and the experimental results gave close values which meant that the results of validation parameters were satisfactory.

TABLE 6 EXPERIMENTAL VALUES ACCORDING TO OPTIMIZED COATING CONDITION.

Independent trials	Experimental value of coating pickup (%)*
Trial 1	0.156 ± 0.059^{a}
Trial 2	0.153 ± 0.051^{a}
Trial 3	0.154 ± 0.054^{a}

* Value expressed as means ± SD. ^{a} SAS® program statistic: F value and Duncan's test, significance level 5%. All trials shown in this table were not significantly (P>0.05) different.

IV. CONCLUSION

In this study, RSM was considered to have succeeded in minimizing the coating pickup as a function of temperature of edible coating emulsion and dipping time. The optimum process conditions for application of edible coating emulsion derived from palm oil based products were best at temperature of coating emulsion (63°C) and dipping time (15s). Under this optimum condition, the predicted coating pickup of coated guava and the experimental results gave close values of about 0.15%. The RSM-predicted and experimental coating pickup values were not significantly different from each other.

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