Optimization of Hydrocolloids and Maltodextrin Addition on Roselle-Based Fruit Leather Using Two-Level Full Factorial Design

Siti Nadiah Shafi'i, Noorlaila Ahmad, Mohd Zahid Abidin, Norziah Mohd Hani, and Normah Ismail

***Abstract*—Texture of fruit leather is important and its quality will be affected by different types of hydrocolloid used. In this work, based on statistical designs, a two-level full factorial design was employed to analyse the extensibility of roselle-based fruit leather with the additional of hydrocolloids (0 - 0.5%) and maltodextrin (1 – 5%). The extensibility of fruit leather was measured using tensile grip (A/TG) by TA.XT2*i*® Texture Analyzer. The influences of four potential independent variables, namely carrageenan (A), xanthan gum (B), locust bean gum (C) and maltodextrin (D) were examined. According to half normal plot analysis, the results showed that xanthan gum, maltodextrin and locust bean gum significantly contributed towards the extensibility of fruit leather with the values of 56.0%, 19.5% and 5.6%, respectively. A near optimum composition for the extensibility of roselle-based fruit leather is as follows; carrageenan, 0.1%; xanthan gum, 0.3%; locust bean gum; 0.3%; and maltodextrin, 1.0%.**

***Index Terms*—Fruit leather, hydrocolloid, roselle, two-level factorial.**

I. Introduction

Roselle (Hibiscus sabdariffa Linn) is known locally in Malaysia as "asam paya", "asam kumbang" and "asam susur". The bright red calyx contains the valuable components which determine the quality of the products: colour (anthocyanins), flavour (organic acid) and aroma. The red acid succulent calices of sabdariffa are used for producing roselle drink, jellies, sauces, ketchups, chutneys, wines, preserves, fruit leather and as natural food colourants (anthocyanins) [1]-[6]. The utilisation of roselle of such products is still considered new in Malaysia.

Fruit leather, often also called fruit sheet, fruit bar or fruit roll, is produced by dehydration of fruit puree [7]. It is an established product, particularly in the North American and European markets. Typically, edible portion of fruit (one or more types) is pureed, mixed with other ingredients to improve physicochemical and sensory characteristics, heated, formed (flattened and shaped), and then dried on a flat tray until the cohesive fruit leather is attained. Preparation of fruit leather from a variety of fruits such as apple, apricot, banana, papaya, mango, sweet potato, ciku, jackfruit and durian [8]-[14] has been investigated. Their studies focused on the drying temperatures, effects of hydrocolloids on the physicochemical and rheology properties, storage stability and sensory characteristics of these fruit leather [10]-[14]. However, there are little studies on roselle based fruit leather that has been carried out by other researchers.

Development, improvement and optimization of formulation, process and product design often involved various variables that likely to be important in the response surface methodology. A two-level, k factor (2^k) factorial design is the fundamental factorial design with utmost importance in screening experiment to determine the significant variables of initial response surface experiment, to achieve first-order response surface model and subsequently as a building block to produce other response surface design such as central composite design. Full factorial design (2^k) and fractional factorial design (2^{k-1}) are common experiment design used in the screening experiment [15].

This study will be focused on the texture improvement of roselle-pineapple fruit leather because the fruit leather produce in an earlier study lacks of extensibility and chewiness. Therefore, this study will be carried out to investigate the usage of hydrocolloids and maltodextrin as an alternatives solution to improve the textural properties of the fruit leather and also to check the contribution effect of each variable and their interactions towards the extensibility of roselle-based fruit leather. In addition, very little has been reported on its texture attributes, which are critical for consumer acceptance.

II. Methods and Materials

A. Materials

Fresh roselle fruit from Sudan cultivar (*Hibiscus sabdariffa* Linn.) of the red variety was purchased from Roselle Farms, Klang, Selangor. Pineapple of Morris var. was purchased from local market in Klang, Selangor. Xanthan gum, Locust bean gum and Carrageenan were purchased from Sigma Aldrich, Malaysia while Maltodextrin was purchased from Meilun Food Chemical Sdn. Bhd, Selangor, Malaysia.

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B. Methods

In the production of fruit leather, the roselle and pineapple were washed with water to remove dirt, leaves and foreign materials. Then, the pineapple was sliced into small pieces, blended and filtered through muslin cloth. The 2:1 ratio (w/w) of roselle to pineapple juices was used. The pineapple juice was used and blended together with roselle to produce mixed fruit puree. The mixtures were heated until boiled. 21 formulations of roselle based fruit leather were formulated based on the 2-level factorial design. Sugar and glucose syrup (dextrose equivalent = 42.0%) were added into the fruit puree mixture until 50°Brix was achieved in these mixtures. Sodium citrate was added to adjust the mixture to pH 3.2. Carrageenan gum, Locust bean gum and Xanthan gum was added in all formulations in a range of 0 – 0.5%. Maltodextrin (dextrose equivalent = 11.5%) was added in a range of 1 – 5%. These mixtures were boiled and left to cool. Each treatment batch was poured into 22.9 x 22.9 cm plastic mould to a (depth of: 3 mm) and dried in the cabinet dryer at 55°C for 12 hours. Texture assessment of roselle based fruit leather with hydrocolloids was performed by measurement of extensibility by using a TA.XT2*i*® Texture Analyzer (Stable Micro Systems Ltd., Godalming, Surrey, U.K.) programmed with the Texture Expeonent software. The samples were cut into uniform strips 2.5 × 5.0cm. A probe labeled Tensile Grips (A/TG) was used to extend each sample until it breaks. An attributes (extensibility) was extracted. Design Expert Software Version 6.0.4 (Stat-Ease Inc., USA) was used to generate the experimental designs, statistical analysis and regression model. A two-level full factorial level was employed. Four independent variables namely Carrageenan (A), Xanthan gum (B), Locust bean gum (C) and Maltodextrin (D) were chosen. Each independent variable assigned two levels of high and low. The details of the factors, units, and ranges are given in the Table I, Table II shows the standard array for four factors and 21 experiments plus the five center points. It also shows the standard order and the observed responses.

TABLE I: ACTUAL FACTORS AND THEIR LEVELS

Parameter	Unit	Nomenclature	Low (-1)	High (+1)
Carrageenan	%	A	0	0.5
Xanthan gum	%	B	0	0.5
Locust bean gum	%	C	0	0.5
Maltodextrin	%	D	1	5

TABLE II: THE CODED DESIGN MATRIX AND RESPONSE

Std	Run	Block	Factor 1 A %	Factor 2 B %	Factor 3 C %	Factor 4 D %	Response 1 Extensibility g
1	20	Block 1	-1.000	-1.000	-1.000	-1.000	365.0
2	4	Block 1	1.000	-1.000	-1.000	-1.000	293.6
3	6	Block 1	-1.000	1.000	-1.000	-1.000	429.9
4	8	Block 1	1.000	1.000	-1.000	-1.000	538.3
5	5	Block 1	-1.000	-1.000	1.000	-1.000	228.9
6	14	Block 1	1.000	-1.000	1.000	-1.000	290.4
7	15	Block 1	-1.000	1.000	1.000	-1.000	729.8
8	7	Block 1	1.000	1.000	1.000	-1.000	703.4
9	13	Block 1	-1.000	-1.000	-1.000	1.000	159.0
10	9	Block 1	1.000	-1.000	-1.000	1.000	143.7
11	19	Block 1	-1.000	1.000	-1.000	1.000	391.1
12	1	Block 1	1.000	1.000	-1.000	1.000	299.1
13	11	Block 1	-1.000	-1.000	1.000	1.000	202.1
14	18	Block 1	1.000	-1.000	1.000	1.000	213.8
15	3	Block 1	-1.000	1.000	1.000	1.000	389.7
16	10	Block 1	1.000	1.000	1.000	1.000	531.6
17	16	Block 1	0.000	0.000	0.000	0.000	443.9
18	21	Block 1	0.000	0.000	0.000	0.000	401.9
19	2	Block 1	0.000	0.000	0.000	0.000	445.5
20	12	Block 1	0.000	0.000	0.000	0.000	419.5
21	17	Block 1	0.000	0.000	0.000	0.000	432.2

III. RESULTS AND DISCUSSION

Using Design-Expert software statistical analysis of the data is performed. The results include a half-normal plot in which the ranks of the absolute value of various effects are determined. Four potential independent variables, namely Carrageenan (A), Xanthan gum (B), Locust bean gum (C) and Maltodextrin (D) were investigated by a four-factor and two-level full factorial design to identify the important factors that exerted significant effect on the extensibility of fruit leather. From the experiment, the results showed that the extensibility of roselle-based fruit leather obtained ranged from 143.7 to 729.8g.

Half normal plot analysis was performed to identify the significant variables and their effects on the response. Half normal plot analysis is particularly effective when there are at least 16 experimental runs with no replication due to the limited raw materials and sources which lead to the limited number of replication and provides a single replicate for the design [16]. Additional data from the center point in the factorial design allows the calculation of the standard error when the factorial point was not replicated. Replicated center points (5 or 6 are usually suggested) are use to estimate pure error for the lack of fit test. Lack of fit indicates how well the chosen model fits the data. With fewer than five or six replicates, the lack of fit test has a very low power because too few center points inflate the error [15]. According to half normal plot analysis (Fig. 1), the insignificant effects fall along the straight line extending from the origin if the observed effects and interactions are due to solely to the experimental error and are normally distributed. Any effects appreciably to the right side of this line were considered to be statistically significant. The studied factor which is located to the right side and far from the line shows the increasing effects of the variables on the response [16]. The significant factors and interactions identified from the half normal plot analysis were chosen for generating the first-order model for the response of texture after the effects and interactions were evaluated. In this case A, B, C, D, AC, BC, BD, ACD, BCD and ABCD were the significant model terms. According to [15], non-significant terms can be dropped from a model or fixed at one level. Thus, it was decided to fix the term A at a concentration of 0.1%. From Table III, high contributions were obtained from xanthan gum (56.03%) and maltodextrin (19.52%) respectively.

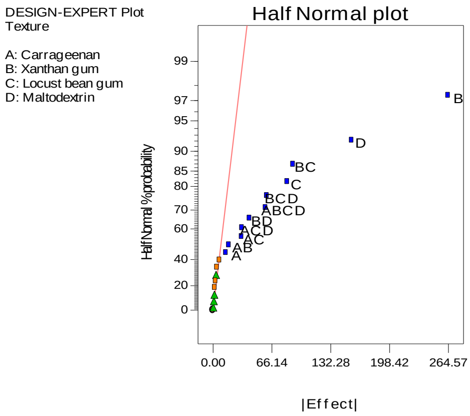

Fig. 1. Half normal plot used to identify significant effects of each variable and their interactions towards the response (extensibility).

TABLE III: Percent Contribution of Variables of Model Terms

Model Term	Contribution (%)
A	0.18
B	56.03
C	5.62
D	19.52
AB	0.26
AC	0.84
AD	0.01
BC	6.53
BD	1.37
CD	0.00
ABC	0.05
ABD	0.02
ACD	0.87
BCD	2.96
ABCD	2.82
Curvature	2.68
Pure Error	0.27

Note:A, B, C and D represent carrageenan, xanthan gum, locust bean gum and maltodextrin respectively.

Table IV showed the curvature value of selected model and the testing for its significant using ANOVA. The curvature compares the average response of the factorial points to the average response of the center points and test for non-linearity between the factorial points. The results show a significant status which reflected that the selected model was not fully linear. According to [17], to evaluate a curvature, a second-order model must be used. The two-level factorial design was used in the estimation of first-order effects, but failed when additional effects, such as second-order effects were significant. Thus, it was decided to use a second-order model such as CCRD in the nest RSM experimental sequence (phase 2).

The lack of fit (LOF) test measures the variation of the data around the fitted model and is one of the important aspects of a reduced model. If the reduced model does not fit the data well, the LOF test will be significant. In this case, the selected model showed a significant (p>0.1) result with probability>F value of 0.8699. A model should be rejected if the results showed significant in LOF test [15].

TABLE IV: Analysis of Variance (ANOVA) Calculated for the Reduced Factorial Model

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	Status
Model	484634.1	11	44057.64	205.6485	< 0.0001	Significant
A	878.9372	1	878.9372	4.102628	0.0774	
B	279979.7	1	279979.7	1306.865	< 0.0001	
C	28068.27	1	28068.27	131.0147	< 0.0001	
D	97547.14	1	97547.14	455.3222	< 0.0001	
AB	1323.513	1	1323.513	6.177783	0.0378	
AC	4197.857	1	4197.857	19.5944	0.0022	
BC	32609.18	1	32609.18	152.2103	< 0.0001	
BD	6838.401	1	6838.401	31.9197	0.0005	
ACD	4323.276	1	4323.276	20.17982	0.0020	
BCD	14787.68	1	14787.68	69.02469	< 0.0001	
ABCD	14080.05	1	14080.05	65.72164	< 0.0001	
Curvature	13387.61	1	13387.61	62.48952	< 0.0001	Significant
Residual	1713.901	8	214.2376			
Lack of Fit	387.2418	4	96.81046	0.291892	0.8699	Not significant
Pure Error	1326.659	4	331.6648			
Cor Total	499735.6	20				

Note:A, B, C and D represent carrageenan, xanthan gum, locust bean gum and maltodextrin respectively.

A statistical summary of the reduced model were listed in Table V. This includes standard deviation, mean, correlation variation (C.V.), predicted residual error sum of square (PRESS), R-Squared, adjusted R-Squared and predicted R-Squared. The mean of average of the response in factorial design and the standard deviation associated with the experimental error were satisfactorily low at 383.45 and 14.64, respectively.

TABLE V: Statistical Summary Obtained from the Reduced Factorial Model

Parameter	Value
Std. Dev.	14.64
Mean	383.45
C.V.	3.82
PRESS	8268.77
R-Squared	0.9965
Adjusted R-Squared	0.9916
Predicted R-Squared	0.9835
Adequate Precision	50.6547

Note: C.V. and PRESS represent correlation variation and predicted residual error sum of square, respectively.

According to [16], the value of C.V. should be less than 10%, and for the reduced model the value was 3.82% which was below the standardised value.

For the reduced factorial model, the coefficient of determination R-Squared (0.9965) which implied that 99% of the variations in texture could be explained by the reduced factorial model. Also, this indicated that the R-Squared value of the reduced model satisfactory surpassed the minimum standardised value (80%) of R-Squared [16]. The adjusted R-Squared is used to measure the amount of variation around the mean explained by the model, adjusted for the number of terms in the model. The adjusted R-Squared decreases as the number of terms in the model increases if those additional terms do not add value to the model (Design Expert Version 6.0.4). The predicted R-Squared is a measure of the amount of variation in the new data explained by the model. The values of predicted-Squared and the adjusted R-Squared should be within 0.20 of each other (Design Expert Version 6.0.4). Otherwise there may be a problem with either the data or the model and might be solved by carefully observing for outliers, considering transformation or a different order polynomial. Since the values of predicted R-Squared (0.9965) was in reasonable agreement with the adjusted R-Squared (0.9916), thus no further amendment of the reduced model should be made.

The adequate precision measure the signal-to-noise ratio and it compares the range of the predicted values at the design points to the average prediction error. A ratio greater than 4 indicates an adequate signal (Design Expert Version 6.0.4) and thus the model can be used to navigate the design space. The adequate precision (50.66) obtained was higher than that limit (4) and perhaps can be used to explore the design space created by the reduced model.

Diagnostic plots serve as a quick view of any abnormality of the response from the reduce model. Most of the plots displayed studentised form of residuals which showed how well the model satisfied the assumptions of analysis of variance (Design Expert Version 6.0.4). Fig. 2 shows the normal probability plot versus studentised residual. Observation by eye concentrating on the central of the data revealed that the residuals follow a normal distribution, in which the points were approximately scattered along the straight line and there was no obvious bad shape like an "S-shape" could be observed in this plot. The presence of the "S-shape" curve indicates abnormality of the data and perhaps transformation of the response may provide a better plot analysis.

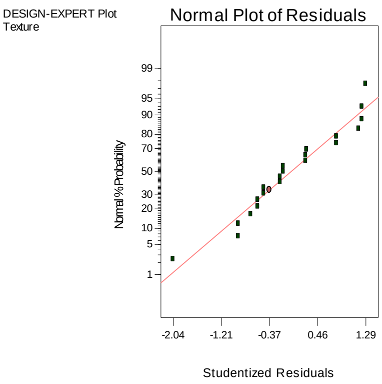

Fig. 2. Normal plot of residuals for the reduced factorial model.

Residual versus predicted plot for texture is normally distributed. The studentized residuals versus predicted plot (Fig. 3) shows the values were low (do not exceed the value between -3 and 3) and the residual appear to be randomly distributed which suggested the variance of observation is constant for the response value. If the value of the studentized residual exceeds ±3, the data should be investigated for checking errors [16].

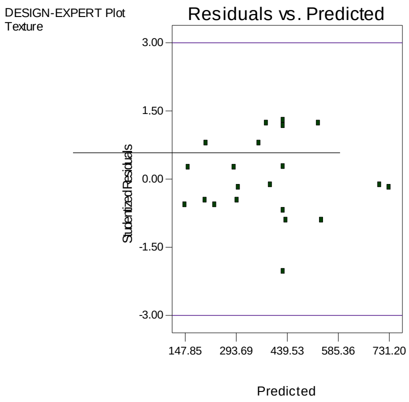

Fig. 3. Studentized residuals vs. Predicted plot for the reduced factorial model.

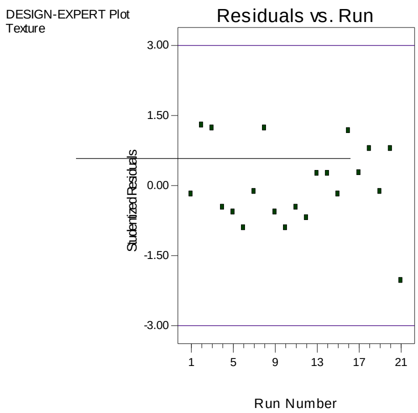

Fig. 4. Residuals against run number for the reduced factorial model.

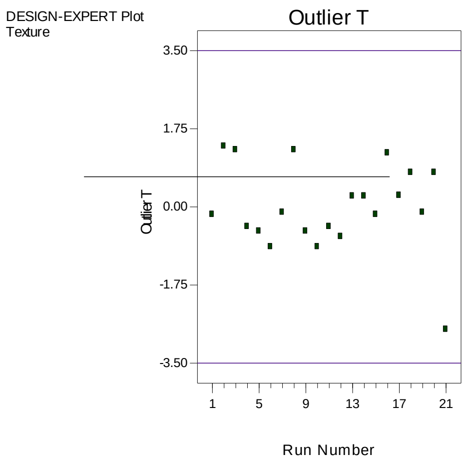

Fig. 5. Outlier against run number for the reduced factorial model.

Plot residual versus experimental run order (Fig. 4) was used to check for any lurking variables that might influence the response during the experiment. Similar pattern or shape was observed in Fig. 5, where the plot outlier against run number which indicated that all points were randomly scattered within ±3. This could be indicating that no serious outliers in all response data of texture as a result of no blocking of experiment and randomisation were completely applied. Fig. 6 tabulated the predicted response values versus the actual response values for the reduced model in factorial design. A linear relationship could be observed between predicted response and the actual response values. This plot might be useful in detecting a value, or group of values, that are not easily predicted by the model.

The design expert provides optimal designs with the desirability factors. Table VI shows an optimal design with desirability factor of 1.000, which determines the optimal level for each factor. The alternative solutions are also shown in Table VII.

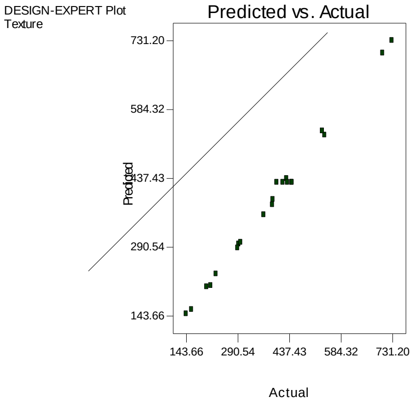

Fig. 6. Predicted against actual for the reduced factorial model.

TABLE VI: OPTIMAL SOLUTIONS

No.	A	B	C	D	Texture	Desirability
1	0.10	0.34	0.29	1.00	465.69	1.000
2	0.10	0.17	0.48	1.00	324.96	1.000
3	0.10	0.22	0.12	1.00	383.26	1.000
4	0.10	0.47	0.29	1.00	554.33	1.000
5	0.10	0.11	0.30	1.00	303.45	1.000
6	0.10	0.37	0.42	1.00	541.49	1.000
7	0.10	0.38	0.40	1.00	541.09	1.000
8	0.10	0.18	0.23	1.00	359.55	1.000
9	0.10	0.10	0.26	1.00	307.24	0.998
10	0.12	0.10	0.10	1.00	356.07	0.975

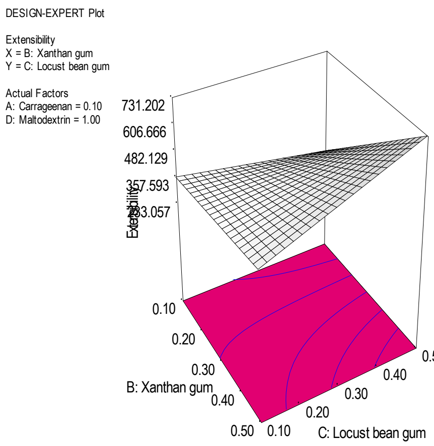

Fig. 7. A three-dimensional plot generated by the model.

IV. CONCLUSION

An investigation of the extensibility of roselle-based fruit leather has been presented. A 2^4 factorial design with five center points was used for investigating the effects of carrageenan, xanthan gum, locust bean gum and maltodextrin. Taking main and interactive effects into account, the results have shown that xanthan gum has the most pronounced effect on the texture of the fruit leather followed by maltodextrin.

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