RESPONSE SURFACE OPTIMIZATION OF DEACIDIFICATION PROCESS OF MENGKUDU EXTRACT USING ION-EXCHANGE RESIN

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ABSTRACT

The unpleasant odor and sour taste of mengkudu (*Morinda citrifolia L.*) extract were reduced by deacidification using ion exchange resin. This work was initiated to optimize the effects of deacidification process of mengkudu extract using response surface methodology (RSM). The experiments were based on 3-level factorial design (column diameter (categorical factor), weight of resin and pump flow rate). Mengkudu extract was deacidified at different column diameter (15, 20 and 25 mm), weight of resin (0, 1.89, 6.45, 11 and 12.89 g) and pump flow rate (1.14, 3, 7.5, 12 and 13.86 ml/min). These three variables were used as independent variables, whose effects on pH, titratable acidity, total polphenol content and vitamin C content of deacidified mengkudu extract were evaluated. Significant regression models describing the changes on the responses with respect to the independent variables were established with coefficient of determination, R^2, greater than 0.70. The results indicated that the weight of resin was the most important factor affecting the characteristics of deacidified mengkudu extract as it exerted a significant influence on most of the dependent variables. The recommended deacidification condition from the study was at 2.56 g weight of resin at 12min/ml pump flow rate using 20 mm diameter column with 65.9% desirability.

Keywords: *Mengkudu (Morinda citrifolia L.); deacidification; ion-exchange resin optimization*

INTRODUCTION

Mengkudu is the Malaysian name for the fruit of *Morinda Citrifolia* L. (Rubiaceae) (Blanco et al. 2006). In the tropics, it seems to have been much valued medicinally and the plant is normally cultivated for its roots, leaves and fruits (Mohd Zin et al. 2002). However, consumer acceptance level is very low because of the unpleasant odor and the sour taste of mengkudu. The unpleasant odor of mengkudu extract was reported to have been contributed by medium chain fatty acids such as capric, caproic and caprylic acids (Norma et al., 2004). Farine et al. (1996) reported that volatile components of mengkudu extract consist of carboxylic acid (83%), alcohol (5%) and esther (3%). As the main component of the volatiles is acidic in nature, manipulation of the acid content may provide an opportunity to improve the undesirable odor of the mengkudu extract.

Deodorization of mengkudu extract has been attempted previously using activated charcoal (Norma et al., 2004) and calcium carbonate (Sharmella et al., 2005; and Maskat 2006). Some studies to deacidify fruit juices with ion exchange resin have been performed (Vera et al. 2003). Nevertheless, this procedure has never been used to deacidified mengkudu extract. In this paper, ion exchange resin has been used as deacidification method. The aim of this study is the deacidification of noni extract using ion exchange resin and to evaluate the effect of column diameter, height of resin and flow rate of extraction on the pH, titrable acidity, total polyphenol content and vitamin C content of mengkudu extract.

MATERIALS AND METHODS

Samples of ripe mengkudu were washed and cut into small pieces. The mengkudu pieces were blend with distilled water at a ratio of 1:1 using a food blender (Summeet®). The blended mengkudu was then filtered and centrifuged at 5000rpm at 8^0C for 20 min. After extraction, the mengkudu extract was deacidified using packed column of ion exchange resin (Amberlite IRA67). The extract was pumped using a peristaltic pump (MasterFlex®) to the top of the column.Three factors studied for the deacidification process were column diameter, weight of resin and pump flow rate.

The experimental design used was Central Composite Rotatable (CCRD) and optimization was performed using response surface methodology (RSM) with computer software Design Expert Version 6.0.10. The experiments were based on 3-level factorial design to study the combined effect of three independent variables (pump flow rate, weight of resin and column diameter). Column diameter was chosen as categorical factor. A total of 39 combinations including five replication of center point and replication of other points were carried out in random order according to 3-level factorial configuration for the three chosen variables. The dependent variables measured were pH value using pH meter (Model PHM

210, Radiometer Analytical), titrable acidity (AOAC, 1990), total polyphenol content using Folin-Ciocalteau reagent (AOAC, 1990), and vitamin C content using 2,6 dichlorophenol-indophenol reagent (AOAC, 1990) of mengkudu extract.

RESULTS AND DISCUSSION

A. STATISTICAL ANALYSIS

The experimental value and analysis of variance for four response variable i.e. pH value, titrable acidity, total polyphenol content and vitamin C content under different treatment conditions according to the three independent factors. The R^2 values for these variables were higher than 0.75, indicating that the regression model explained the reaction well. A higher value of the correlation coefficient R^2 justifies an excellent correlation between the independent variables. Positive symbol (+) from coefficient showed increasing effect to measured parameters and negative symbol (-) showed decreased effect.

The model equation for pH value (y_1) in terms of coded factors:

$$y = 6.69 + (-0.40x_1) + (-0.47x_2) + (-0.15x_{3a}) + (-0.020x_{3b}) + (-0.41x_{11}) + (0.84x_{22}) + (0.14x_{12}) + (0.10x_{13a}) + (-0.045x_{13b}) + (0.41x_{23a}) + (0.024x_{23b}) + (0.73x_{111}) + (-0.064x_{222})$$

The model equation for titrable acidity (y_2) in terms of coded factors:

$$y = 0.41 + (0.53x_1) + (0.35x_2) + (0.023x_{3a}) + (0.013x_{3b}) + (0.38x_{11}) + (-0.13x_{22}) + (-0.053x_{12}) + (-0.076x_{13a}) + (0.097x_{13b}) + (0.048x_{23a}) + (-0.052x_{23b}) + (-0.65\, x_{111}) + (-0.21\, x_{222})$$

The model equation for total polyphenol content (y_3) in terms of coded factors:

$$y = 1001.44 + (-148.99x_1) + (-153.77x_2) + (97.98x_{3a}) + (242.28x_{3b}) + (360.21x_{11}) + (-46.77x_{22}) + (-4.28x_{12}) + (0.58x_{13a}) + (-57.06x_{13b}) + (18.51x_{23a}) + (-13.64x_{23b}) + (-238.61x_{111}) + (-225.15x_{222})$$

The model equation for vitamin C content (y_4) in terms of coded factors:

$$y = 132.20 + (-23.11x_1) + (57.21x_2) + (-7.18x_{3a}) + (49.74x_{3b}) + (69.09x_{11}) + (-25.50x_{22}) + (-8.17x_{12}) + (9.22x_{13a}) + (-9.65x_{13b}) + (-0.75x_{23a}) + (6.48x_{23b}) + (-52.56x_{111}) + (-3.21x_{222})$$

where,

y = dependent variable; x = independent variables

subscript: 1 = weight of resin; 2 = pump flow rate; 3 = column diameter

a = 15mm column; b = 20mm column

B. pH VALUE

pH value was significantly affected by weight of resin ($p<0.01$) and pump flow rate ($p<0.05$). According to Fig. 1, pH value increased as weight of resin and pump flow rate decrease. This result were similar to Vera et al. (2003), which recommend the reduction of flow rate to deacid passion fruit until pH 4. Calle et al. (2002) reported in his study, that pH decreased with the resin saturation.

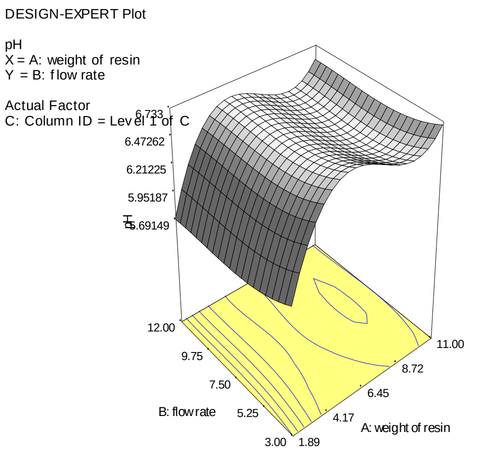

Figure 1 3D surfaces plot for pH of mengkudu extract as a function of weight of resin and flow rate

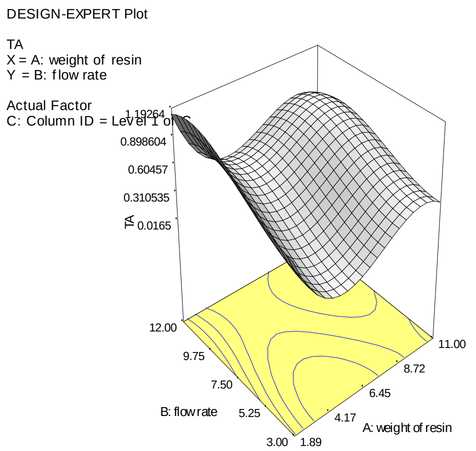

Figure 2 3D surfaces plot for titratable acidity of mengkudu extract as a function of weight of resin and flow rate

C. TITRATABLE ACIDITY

From the analysis, the only factor that affects titratable acidity significantly ($p<0.05$) is weight of resin. Fig. 2 shows titratable acidity increased as weight of resin increased and at a certain point of different level of flow rate and weight of resin, titratable acidity was decreased and then increased over again.

D. TOTAL POLYPHENOL CONTENT (TPC)

All the factors studied show significant ($p<0.05$) effect to total polyphenol content in mengkudu extract (Fig. 3). Pump flow rate and column diameter show positive effect while increasing the weight of resin reduced total polyphenol content in mengkudu extract.

E. VITAMIN C CONTENT (VIT C)

Analysis of vitamin C content shows significant ($p<0.05$) effect by all the factors positively except for weight of resin factor as in Fig. 4. Vitamin C content decreased as weight of resin incereased.

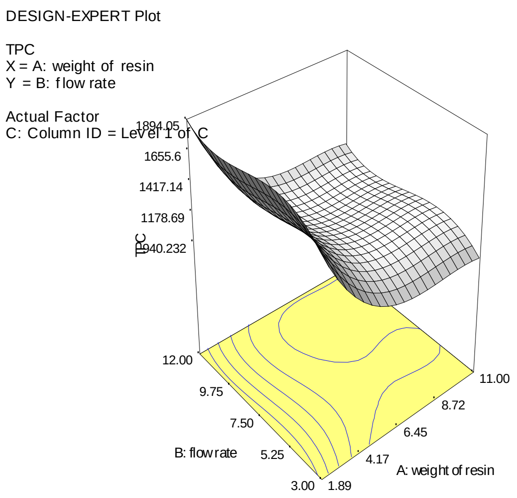

Figure 3 3D surfaces for total polyphenol content of mengkudu extract as a function of weight of resin and flow rate

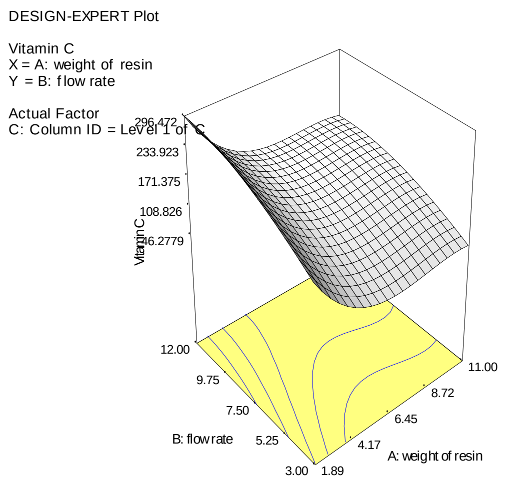

Figure 4 3D surfaces for vitamin C content of mengkudu extract as a function of weight of resin and flow rate

F. OPTIMIZATION

The optimum condition for the purpose of the deacidification of mengkudu extract using ion-exchange resin was determined by superimposing the contour plots of all four responses. The criteria applied for graphical optimization were as follows: (a) maximum pH value, (b) minimum titratable acidity, (c) maximum total polyphenol content, and (d) maximum vitamin C content. The computer generated plots for pH value, titratable acidity, totalpolyphenol content and vitamin C content (Figs. 1–4); and the criteria set above produced an optimum region in the 3D surface plot as shown in Fig. 5. These criteria were selected as they are important characteristics of the mengkudu extract as it represent the value and nutrition of the fruit. The optimum deacidification process was found to be at 2.56g weight of resin at 12ml/min flow rate using 20mm diameter column.

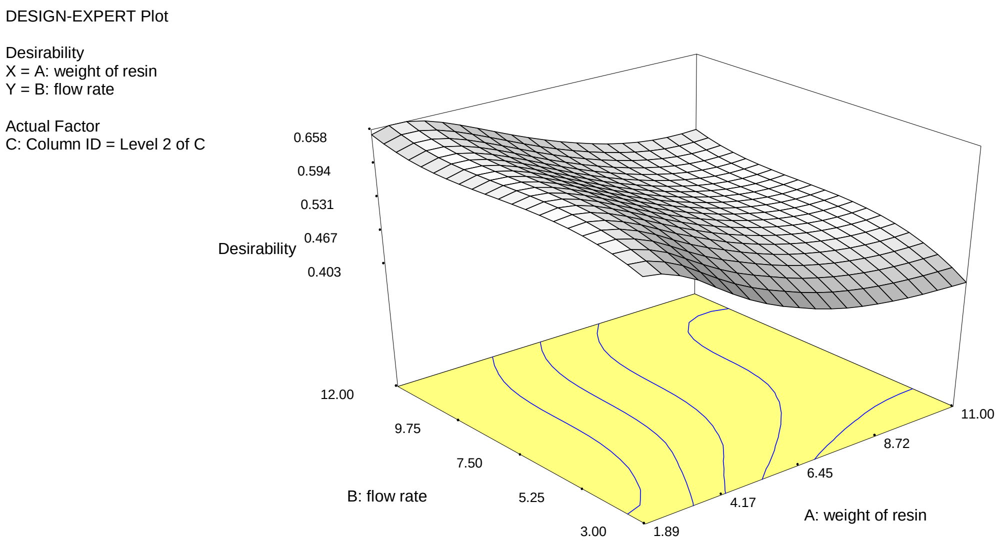

Figure 4 3D surface plot for optimum deacidification process of mengkudu extract as a function of weight of resin and flow rate

CONCLUSION

Response surface methodology was used to establish the optimum deacidification process variables (column diameter, flow rate and weight of resin) for deacidification of mengkudu extract. By using response surface and contour plots, the optimum set of operating variables can be obtained graphically, in order to achieve the desired properties affected levels for the deacidified mengkudu extract. Therefore, it was suggested that the optimum deacidification process condition being at 2.56g weight of resin and 12ml/min of flow rate using 20mm column diameter with 65.9% desirability.

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