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Optimization of concentration process on pomelo fruit juice using response surface methodology (RSM)

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Abstract: Within the globalization of the food industry, the demand for quality juice and juice type beverages has expanded. The pomelo fruit has recently gained attention because of its antioxidant capacity as studies found extracts of carotenoids, phenolics, vitamin C, lycopene, and anthocyanin. Concentration of fruit juices is important due to their low water activity, have a higher stability than single-strength juices. To reduce the storage and shipping costs, and to achieve longer storage, fruit juices are usually concentrated by multi-stage vacuum evaporation. In this work vacuum evaporation method was applied to concentrate pomelo juice. Response surface methodology (RSM) was applied to investigate the effects of the three main independent parameters; rotation, temperature and time on the pomelo juice concentration and optimizing the operating conditions of the treatment process. It is clear from the result that the concentration increased when the temperature, rotational speed and time increased. It can be inferred that any parameters, individually, had positive effect on increase of concentration. The main effects of parameters are in following order: Main effect of time> temperature> rotational speed.

Keywords: Response surface methodology (RSM), pomelo juice, concentration, vacuum evaporation, optimization

Introduction

Pomelo, pommelo, shaddock or limua bali [*Citrus grandis*(L) Osbeck] is referred to a type of giant citrus fruit native to southern Asia and Malaysia. It is thought to be the ancestor of the grape fruit. The pomelo is named Shaddock after an English sea captain who introduced the fruit to the West Indies from the Malay Archipelago. In New Zealand and North American region, the fruit is still known as Shaddock, but the name pomelo is also well known (Keshani, 2009). Pomelo is commonly consumed as fresh fruit. They are also good for salad, jams, jellies, marmalades and syrups. Pomelo is used in religious ceremony in Malaysia, especially during Chinese New Year and the moon or autumn festival. Pomelo is used as a symbol of good luck and prosperity in Chinese New Year celebrations. The skins and the leaves could be boiled to prepare a ceremonial bath to ritually cleans a person and repel evil. The word for Pomelo in Chinese is pronounced the same as the word for blessing, or protection, thus its widespread presence in many Buddhist shrines (Turk, 2002).

Fruit juices are valuable semi finished products for use in the production of fruit juice beverages and fruit juice powders. The conventional mode in which fruits are processed and preserved is the form of fruit juices/pulps (purees). However, preservation of juices is not economical, since the water content of fruit juices is very high, i.e. 75 to 90% (Ramteke *et al*., 1993).

Concentration of fruit juices not only provides microbiological stability, but also leads to economical packaging, transportation and distribution of the final products. However, the concentration of fruit juices is a susceptible process. In as much as their constituents are chemically unstable, even at moderate temperatures. Furthermore, the quality of concentrated juices is dependent on the configuration of odorous compounds in the fresh juice (Moresi and Spinosi, 1980; Belibagli and Dalgic, 2007). In concentration processes, the solids content is increased up to 65 to 75% so that the final product is still in liquid form (Ramteke *et al*., 1993).

The first fruit juice concentrate, produced by vacuum evaporation, refers to the beginning of the

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1920's. During last sixty years, several methods for concentration of liquid fluids have been developed, e.g. evaporative concentration, freeze concentration and membrane concentration (reverse osmosis) which they have received attention for commercial application (Thijssen, 1975). Several studies have been performed to find an effective and economical way for concentration liquid foods. The advantages and disadvantages of thermal evaporation, freeze drying, freeze concentration and reverse osmosis are listed in Table 1. Evaporation is probably the oldest method of concentration. Furthermore, it is considered to be the best developed, economical and widely used method for concentration of liquid foods. Heat sensitivity of the product is of particular importance in selecting the evaporator, as it affects the quality of the concentrate (Ramteke *et al*., 1993).

Design of experiments is important for multifactor experiments to save time and capable of predicting the optimum of the combined factors. Previously, the most popular approach in determining the optimum or best condition of any responses studied is through the classical one-variable-at-time technique. The classical method of the optimization involves changing one variable at a time while keeping the others at fixed levels (Erin, 2005). While such experiments are simple to plan and execute, they are inefficient and failed to detect any interaction amongst the independent variables. Furthermore, it will require more experimentation than a design of experiment by factorial and there is no assurance that it will produce the correct and re-presentable results (Montgomery *et al*., 2001). Thus, to overcome such drawbacks, the technique of response surface methodology (RSM) is being progressively employed for modeling, interaction study and optimizing any processes or experiments. RSM has been used extensively for optimizing processes in the tropical fruit juice production (Chan *et al*., 2009; Lee *et al*., 2006; Sin *et al*., 2006; Wong *et al*., 2003; Yusof *et al*., 1988).

RSM is defined as the statistical tool that used the quantitative data from various experimental designs to determine and simultaneously solve the multivariate equations. RSM explores the relationships between several explanatory variables and one or more response variables (Carley *et al*., 2004). In this work, RSM is used as method in order to optimize the concentration of pomelo juice.

Materials and Methods

Preparation of samples

Fruits were purchased from a local market and washed with water to remove any adhering substances, sliced and hand peeled. Juice was extracted from the fruits by homogenizing in waging blender at 8000 rpm for 3 min followed by filtration and centrifugation at 9000 rpm for 10 min. The concentrated pomelo juice is obtained by a small scale laboratory vacuum evaporation (HEIDOLPH, Germany).

In order to select the variables which are likely to be important in preparing the juice concentrates, response surface methodology (RSM) is used. It is usually called a screening experiment. The objective of factor screening is to reduce the list of candidate variables to a relatively few so that subsequent experiments will be more efficient and require fewer runs or tests. The purpose of this phase is the identification of the important independent variables. The related from the RSM can be used to prepare different concentration of pomelo juice under different process conditions and described.

Experimental design

Response Surface Methodology (RSM) was used in this study to determine the optimum conditions of the treatment process for the concentration of pomelo juice. The effect of three independent variables by a small scale laboratory vacuum evaporation, x_1 (rotational speed), x_2 (temperature) and x_3 (time), on one response variables (Y_1, namely concentration) was evaluated by using the RSM. A central composite design (CCD) was employed (1) to study the main effect of parameters, (2) to create models between the variables and (3) to determine the effect of these variables to optimize the concentration of pomelo juice. Therefore, 20 experiments were designed based on the second-order CCD with three independent variables at three levels of each variable. Independent variable ranges studied were: speed rotation (60-120 rpm), temperature (40-60^{o}C) and time (5-60 min). Experiments were randomized in order to minimize the effects of unexplained variability in the actual responses due to extraneous factors. Table 2 shows the arrangement of experiments based on standard order.

Statistical analyses

There are four major steps in the application of RSM (Erin, 2005) (1) Experimental set up, (2) Experimental design, (3) Statistical analysis, (4) Model Selection. For the experimental set-up stage, the experimental factor and factor level were chosen. Factors are the characteristics of a process that can be varied within a system and factor levels are the degree or quantity of the factors. The specific test samples were determined by the experimental design

Table 1. Advantages and disadvantages of the current processes of concentrating fruit juices

Treatment methods	Advantages	Disadvantages
Thermal Evaporation	Reduce the energy consumption at the drying operation. Reduce water activity that will enhance the storage stability. Reduce weight and volume of fluids. (Ramaswamy and Marcotte, 2006a).	Loss of volatiles and aromas and some food liquids are heat sensitive (Ramaswamy and Marcotte, 2006a). High energy consumption of evaporators (Ramteke *et al*., 1993).
Freeze Drying	High quality of the product. Preserve the vitamin content (Ramaswamy and Marcotte, 2006b).	Long drying time (Ramaswamy and Marcotte, 2006b). The high equipment and operational costs (Ramaswamy and Marcotte, 2006b).
Freeze concentration	Require less energy (Cassan o *et al*., 2007). Flavor loss is minimum at low temperature.(Lee and Lee, 1999)	Process is expensive (Shamsudin, 2000). Degree of concentration achievable is limited (Cassano *et al*., 2007). Use for high value juices or extracts (Ramaswamy and Marcotte, 2006a).
Reverse osmosis	Reduced loss of volatile organic Increased aroma and flavor retention (Jiao *et al*., 2004). Lower energy consumption and greater retention of product quality (Jiao *et al*., 2004). Lower equipment costs (Cassano *et al*., 2007).	Low flux; (Ramteke *et al.*, 1993). Obtaining high concentrated juice is limited due to high osmotic pressure (Cassano *et al*., 2007).

Table 2. Experimental design matrix and response value

Treatment runs	Rotational Speed (rpm)	Temperature (°C)	Time (min)
1	90.00	60.00	30
2	120.00	60.00	60
3	60.00	60.00	5
4	90.00	50.00	50
5	90.00	50.00	45
6	60.00	50.00	45
7	90.00	50.00	30
8	90.00	50.00	60
9	90.00	40.00	30
10	120.00	40.00	60
11	60.00	50.00	30
12	90.00	60.00	45
13	120.00	60.00	5
14	60.00	40.00	60
15	60.00	40.00	5
16	90.00	50.00	25
17	90.00	50.00	15
18	120.00	50.00	30
19	120.00	40.00	5
20	60.00	60.00	60

stage and tested. Data from the experiment perform were analyzed using the statistical software and then interpreted. There are three main analytical steps: analysis of variance (ANOVA), a regression analysis and plotting of the response surface. The first task in analyzing the response surface is to estimate the parameters of the model by least square regression and to obtain information about the fit in the form of ANOVA. Of particular importance are values for the Fischer variance ratio (F-ratio) and the coefficient of determination (R-squared).

The F-ratio provides information on how well the factors describe the statistical variation in the data from its mean. The R-squared evaluates the suitability of the model in representing the real relationship among the factors studied. A value of 0.75 implies the model is adequate for representing the relationship among the factors while a value of > 0.90 indicates the model describe the real situation well. A regression analysis was then performed to generate coefficient $(\beta_o, \beta_1, \ldots, \beta_n)$ for the selected empirical model. The significance of the coefficients with P-values of <0.05 is generally considered highly significant and therefore included in the mathematical model. The model is often a linear, quadratic or cubic order polynomial function and when fitted to a set of sample data, characterizes the relationship between the responses and the factors (Montgomery et al., 2001). In general, the relationship can be written as follows;

$$y = f(\xi_1, \xi_2, \ldots, \xi_k) + \varepsilon \qquad (1)$$

Where the form of the true response function f is unknown and ε is a term that represents other sources of variability not accounted for in f. Usually is ε treated as a statistical error, often assuming in to have a normal distribution with mean zero and variance, σ^2 , and therefore; includes effects such as measurement error on the response, background noise, the effect of other variables, and so on.

$$E(y) = E\,[f(\xi_1, \xi_2, \ldots, \xi_k) + E(\varepsilon) = f(\xi_1, \xi_2, \ldots, \xi_k) \qquad (2)$$

The variables $\xi_1, \xi_2, \ldots, \xi_k$ in equation above are usually called the natural variables, because they are expressed in the natural units of measurement, such as degrees Celsius, pounds per square inch, *etc*. In much RSM work it is convenient to transform the natural variables to coded variables $x_1, x_2, \ldots, x_k$ which are usually defined to be dimensionless with mean zero and the same standard deviation. In terms of the coded variables, the response function will be written as:

$$Y = f(x_1, x_2, \ldots, x_k) \qquad (3)$$

Because the form of the true response function f is unknown. In fact, successful use of RSM is critically dependent upon the experimenter's ability to develop a suitable approximation for f . Usually a low-order polynomial in some relatively small region of the independent variable space is appropriate. In many cases, either a first-order or a second-order model is used. The first-order model is likely to be appropriate when the experimenter is interested in approximating the true response surface over a relatively small region of the independent variable space in a location where there is little curvature in f. For the case of two independent variables, the first-order model in terms of the coded variables is,

$$Y = \beta_o + \beta_1 x_1 + \beta_2 x_2 \tag{4}$$

The form of the first-order model is sometimes called a main effects model, because it includes only the main effects of the two variables x_1 and x_2. If there is an interaction between these variables, it can be added to the model easily as follows:

$$Y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 \tag{5}$$

This is the first-order model with interaction. Adding the interaction term introduces curvature into the response function. Often the curvature in the true response surface is strong enough that the first-order model (even with the interaction term included) is inadequate. A second-order model will likely be required in these situations. For the case of two variables, the second-order model is:

$$Y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 \tag{6}$$

This model would likely be useful as an approximation to the true response surface in a relatively small region. The second-order model is very flexible. It can take on a wide variety of functional forms, so it will often work well as an approximation to the true response surface. It is also easy to estimate the parameters (the β's) in the second-order model. The method of least squares model can be used for this purpose. In addition, there is considerable practical experience indicating that second-order models work well in solving real response surface problems (Mirhosseini *et al*., 2008a, b).

Models that represent the data well can then be used to generate response surface. They are three-dimensional diagrams with the responses plotted on the y-axis and the x_1 and x_2 axes each representing different factors in different permutations. While response surface are commonly dome-shaped, those with cradle and saddle points are also possible (Montgomery *et al*., 2001).

Results and Discussion

Response surface methodology

In the present work, multiple regression analyses were carried out using response surface analysis (1) to fit mathematical models to the experimental data aiming at an optimal region for the response variables studied and (2) to define the relationship between three independent variables and the criteria of three response variables as presented in Table 3. The response surface analysis allowed the development of an empirical relationship where each response variable (Yi) was assessed as a function of rotational speed (x_1), temperature (x_2) and time (x_3) and predicted as the sum of constant (β_o), three first-order effects (linear terms in x_1, x_2 and x_3, three interaction effects (interactive terms in x_1x_2, x_1x_3, and x_2x_3) and three second-order effects (quadratic terms in $x_1^2 x_2^2$ and x_3^2). The obtained results were analyzed by ANOVA to assess the "goodness of fit". Only terms found statistically significant ($p<0.05$) were included in the reduced model. As shown in Equation (7), the obtained model for predicting the response variables explained the main quadratic and interaction effects of factors affecting the response variables. The estimated regression coefficients of the polynomial response surface models along with the corresponding R^2 values and lack of fit tests are given in Table 4. The significance of each term was determined using the F-ratio and p-value as presented in Table 5.

It was found that the values of "Prob > F" less than 0.05 indicate model terms are significant. In this case x_1, x_2, x_3 , x_3^2, x_2x_3 are significant model terms. Values greater than 0.1 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

Analysis of variance also confirmed that the models were highly significant ($p<0.05$) for all response variables (Table 5). The probability (p) values of all regression models were less than 0.05, which had no indication of lack of fit. The R^2 values for these response variables were higher than 0.80 (0.9808), thus ensuring a satisfactory fitness of the regression models to the experimental data. The following response surface models Equation (7) were fitted to the response variable (Y_1) three independent variables (x_1, x_2 and x_3):

$$\begin{aligned} Y_1 = & -17.63133 + 0.14364\, x_1 + 0.88792\, x_2 - 0.066773\, x_3 \\ & -6.39354E\text{-}004\, x_1^2 - 8.49033E\text{-}003\, x\, x_2^2 - 4.84807E\text{-}003\, x_3^2 \\ & -2.91667E\text{-}004\, x_1 x_2 + 9.39366E\text{-}004\, x_1 x_3 + 7.64963E\text{-}003\, x_2 x_3 \end{aligned} \tag{7}$$

Optimization of concentration process

The predicted versus actual plots for concentration (Y_1) is shown in Figure 1. The observed points on these plots reveal that the actual values are distributed relatively near to the straight line in this case ($R^2 = 0.98$). The 3D response surfaces was plotted to better visualize the significant ($p < 0.05$) interaction effects of independent variables on the concentration of pomelo juice. The plots are drawn as a function of

Table 3. Central composite design: independent (Xi) and response variables (Yj)

runs	Rotation Speed (rpm) (x_1)	Temperature (℃) (x_2)	Time (min) (x_3)	Concentration (˚Brix) (Y_1)
1	90	60	30	26.7
2	120	60	60	30.4
3	60	60	5	13.4
4	120	50	5	14.1
5	90	60	60	29.8
6	60	50	45	22.7
7	90	50	30	23.1
8	90	50	60	27.2
9	60	50	5	13.3
10	120	40	60	21.3
11	60	50	30	21.3
12	120	50	60	30.4
13	120	60	5	14.2
14	60	40	60	18.6
15	60	40	5	12.6
16	90	60	5	13.9
17	90	50	5	13.8
18	120	50	30	24.8
19	120	40	5	13.5
20	60	60	60	28.3

Table 4. Regression coefficients, R^2, adjusted R^2 probability values and lack of fit for five variables

Regression coefficient	**Concentration (^{o} Brix) (Y_1)**
β_o	-17.63133
β_1	0.14364
β_2	0.88792
β_3	0.066773
β_{11}	-6.39354E-004
β_{22}	-8.49033E-003
β_{33}	-4.84807E-003
β_{12}	-2.91667E-004
β_{13}	+9.39366E-004
β_{23}	+7.64963E-003
Regression (p-value)	0.98
Lack of fit	10

Table 5. ANOVA and regression Coefficients of the first-and second-order polynomial regression models

Variables Y1	Main effects			Quadratic effects			Interaction effects		
	x1	*x2*	*x3*	*x12*	*x22*	*x32*	*x1x2*	*x1x3*	*x2x3*
p-Value	0.0031	0.0001	0.00001	0.4068	0.2188	0.0008	0.8499	0.0720	0.0004
F-ratio	15.00	37.62	324.93	0.75	1.72	22.57	0.038	4.05	26.51

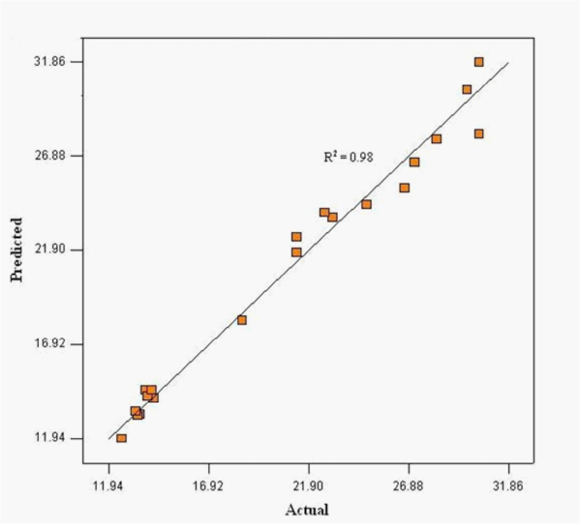
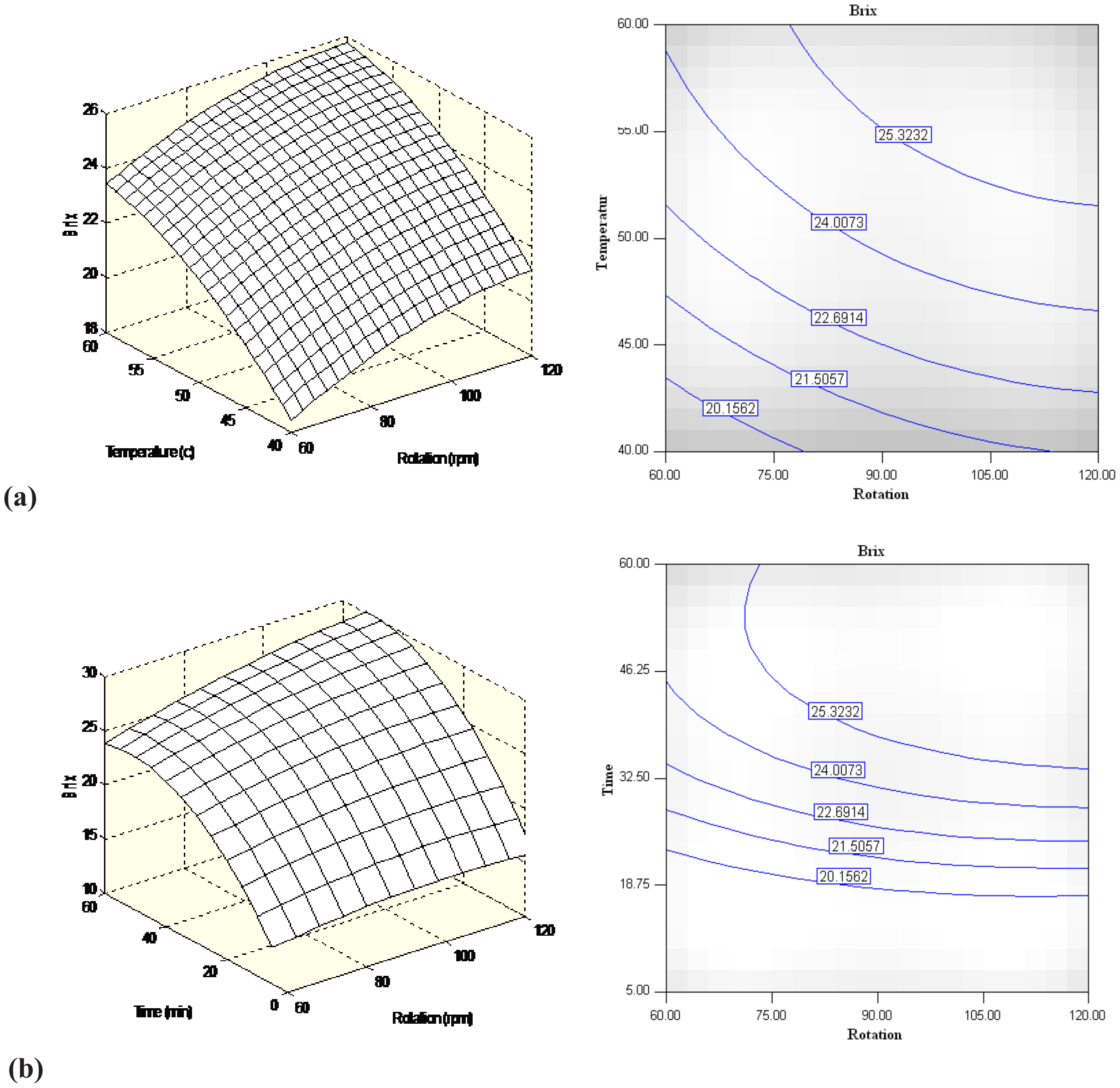
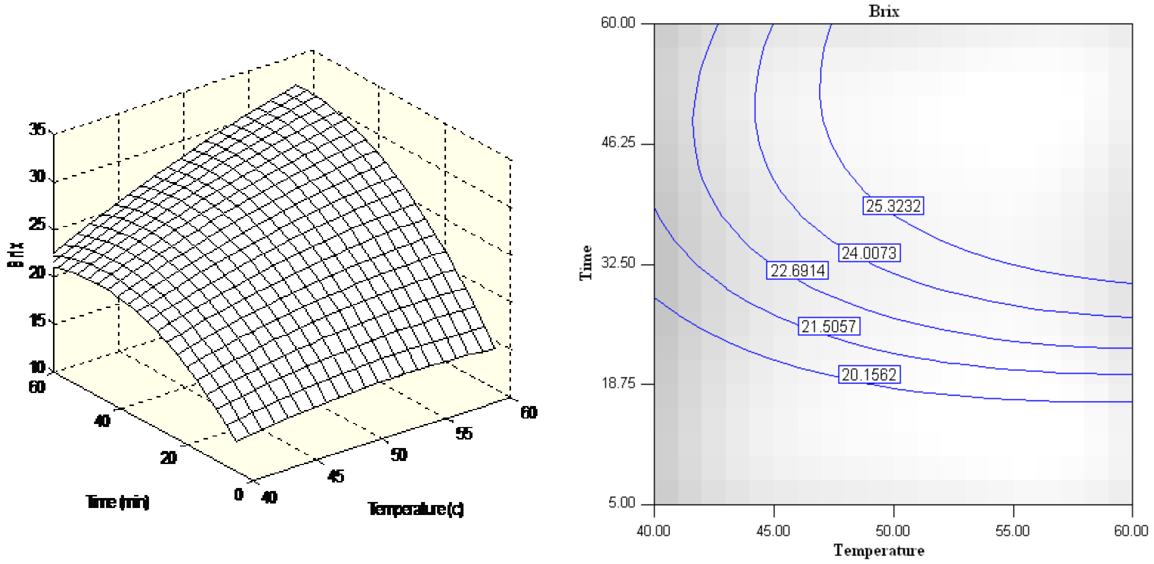


Figure 1. Predicted versus Actual data for concentration of pomelo juice





(c)

Figure 2. Response surface plots for concentration of pomelo juice as a function of (a) Rotation and Temperature, (b) Rotation and time and (c) Temperature and Time

two factors at a time, holding the third factor at fixed levels (at the mid level). Those plots are helpful in understanding both the main and the interaction effects of these two factors. As shown in Figure 2(a–c), the presence of curvatures in the concentration curves confirmed that the variation of concentration (Y_1) was explained as a nonlinear function (exponentially decaying) of pomelo juice. It is clear from the figure that the concentration increased when the temperature, rotational speed and time increased. It can be inferred that any parameters, individually, had positive effect on increase of concentration. According to Table 5, the main effects of parameters are in following order: Main effect of time> temperature> rotational speed. P-values of parameters are 0.00001, 0.0001 and 0.0031, respectively. It can be observed from Figure 2 (a) that when temperature varied 20 °C the concentration varied from 18 to 23^{o} Brix, while rotational speed varied 60 rpm the concentration varied from 18-21^{o} Brix. It can be observed from Figure 2 (b) that when the time varied 55 min the concentration varied from 13.3 to 24^{o} Brix, while the rotation varied 60 rpm the concentration varied from 13.3 to 14.1^{o} Brix. From Figure 2 (c) it can be observed that when temperature varied 20 °C the concentration varied from 13.1 to 13.9^{o} Brix, while the time varied 55 min the concentration varied from 13.1 to 23^{o} Brix.

Özilgen (1998) showed that the drying curve is divided into two distinct portions. The first is the constant rate period, in which unbound water is removed. Water evaporates as if there was no solid matrix present, and its rate of evaporation is not dependent on the solid matrix. This continues until free water molecules are no longer available. During drying period, the drying rate decreases with time.

The same trend was observed for concentrating pomelo juice. As it could be observed from Figure 4.2(b–c) in the constant rate period, the rate of drying was constant as the moisture content was reduced. During this period drying took place from a saturated surface and the vaporized water molecules diffused through a thin film of air close to the surface of the material before being transported into the bulk of the air stream. The rate of drying decreased in the falling rate period. In the falling rate period the fruit juice surface was no longer capable of supplying sufficient free moisture to saturate the air above it. This means that the rate of drying was then influenced by the mechanism of transport of moisture from within the fruit juice to the surface. Evaporation then depended upon the diffusion of vapor through the material and was therefore increasingly slow (e.g. when rotation is 90 rpm and temperature is 50°C efficiency of drying from5 to 30 min is 75.8 % while from 30 to 60min the efficiency of drying is 24.16 %) (Okos *et al.*, 2007; Smith, 2003; Potter, 1978).

Value of parameters required to obtain efficiency of concentration pomelo juice is listed in Table 6. It can be observed that in all of the conditions the best efficiency was achieved by the maximum rotational speed (120 rpm).

Table 6. Value of parameters required for obtaining the efficiency of concentration of pomelo juice

Rotation (rpm)	Temperature (°C)	Time (min)	Concentration (^{o} Brix)	Efficiency (%)
120	40	60	22.6	100
120	40	26.3	20.34	90
120	40	31.81	21.47	95
120	45	60	25.55	100
120	45	30.27	23	90
120	45	36.83	24.3	95
120	50	60	28	100
120	50	33.48	25.2	90
120	50	40.34	26.6	95
90	40	60	21.06	93
90	40	30	20.27	89
60	40	60	18.21	80
60	40	30	18.36	81
90	45	60	24.04	94
90	45	30	22.01	86
60	45	60	21.32	83
60	45	30	20.30	79
90	50	60	26.55	94
90	50	30	23.54	84
60	50	60	23.86	85
60	50	30	21.76	77

Conclusions

Response surface methodology was used to establish the optimum process variables (rotational speed, temperature and time) for concentration of pomelo juice. These can be related to the operating conditions of the treatment process by second order polynomials. By using response surface and contour plots, the optimum set of operating variables can be obtained graphically, in order to achieve the desired pretreatment levels for the pomelo juice. Therefore, it was recommended that the concentration increased when the temperature, rotational speed and time increased. It can be inferred that any parameters, individually, had positive effect on increase of concentration. Thus, they had exponentially decaying pattern. The main effects of parameters are in following order: Main effect of time> temperature> rotational speed.

References

Belibagli, K. B. and Dalgic, A.C. 2007. Rheological Properties of Sour-Cherry Juice and Concentrate. International Journal of Food Science and Technology 42: 773–776.

Carley, K.M., Kamneva, N.Y. and Reminga, J. 2004. Technical Report of Response Surfaces Methodology CASOS. USA: School of Computer Science, Carnegie Mellon University, ISRI-CASOS.CMU-ISRI-04—136.

Cassano, A., Conidi, C., Timpone, R., D'Avella, M. and Drioli, E.2007.A membrane-based process for the clarification and the concentration of the cactus pear juice. Journal of Food Engineering 80: 914–921.

Chan, S. W., Lee, C. Y., Yap, C. F., Wan Aida, W. M. and Ho, C. W. 2009. Optimisation of extraction conditions for phenolic compounds from limau purut (*Citrus hystrix*) peels. International Food Research Journal 16:

203-213.

Erin, R. G. 2005. Factors effecting the optimization of lipase-catalyzed palm-based esters synthesis. Serdang, Malaysia: Universiti Putra Malaysia, PhD Thesis.

Geankoplis, C.J. 1993. Drying of process Materials. In Geankoplis, C. J. Transport Process and Unit Operations, p.520-584. New York: Prentice Hall.

Jiao, B., Cassano, A. and Drioli, E. 2004. Recent advances on membrane processes for the concentration of fruit juices: a review. Journal of Food Engineering 63:303–324.

Keshani, S. 2009. Rheology of pomelo juice. Serdang, Malaysia: University Putra Malaysia, MSc thesis.

Lee,Y.C. and Lee, S.W. 1999. Quality changes during storage in Korean Clear Pear Juices concentrated by three methods. Journal of Food Quality 22: 565-571.

Lee, W.C, Yusof, S., Hamid, N.S.A. and Baharin, B.S. 2006. Optimizing conditions for enzymatic clarification of banana juice using response surface methodology (RSM). Journal of Food Engineering 73: 55-63.

Mirhosseini, H., Tan, C.P., Hamid, S.A.H. and Yusof, S. 2008a. Optimization of the contents of Arabic gum, xanthan gum and orange oil affecting turbidity, average particle size, polydispersity index and density in orange beverage emulsion. Food Hydrocolloids 22: 1212–1223.

Mirhosseini, H., Tan, C.P., Hamid, S.A.H. and Yusof, S. 2008b. Effect of Arabic gum, xanthan gum and orange oil contents on ξ-potential, conductivity, stability, size index and pH of orange beverage emulsion. Colloids and Surfaces A: Physico Chemical Engineering Aspects 315: 47–56.

Montgomery, D.C., Runger, G.C., and Hubbele, N.F. 2001. Engineering Statistics. p. 23-40 New York: John Wiley and Sons, Inc.

Moresi, M. and Spinosi, M.1980.Engineering factors in the production of concentrated fruit juices. Journal of Food Technology 15:265-276.

Okos, M.R., Campanella, O., Narsimhan, G., Singh, R.K. and Weitnauer,A.C. 2007. Food Dehydration. In *Handbook of Food Engineering* ed. D.H. Heldman and D.B. Lund, p.601-744. New York: CRC Press.

Özilgen, M.1998. Mathematical modeling in food engineering operations. In Food Process Modeling and Control. In Hughes R. (Eds.), p. 227-378. Amesterdam: Amsteldijk.

Potter, N.N. 1978. Food dehydration and concentration. In Food science, p.260-322. New York: The AVI Publishing Company INC.

Ramaswamy, H. and Marcotte, M. 2006a. Separation and Concentration. In Food processing Principles and Applications, p.317-379. United States of America: CRC Press.

Ramaswamy, H. and Marcotte, M. 2006b. Food dehydration. In Food processing Principles and Applications, p.233-317. United States of America: CRC Press.

Ramteke, R.S., Singh, N.I., Rekhaand, M.N. and Eipeson, W.E 1993. Methods for concentration of fruit juices: A critical evaluation. Journal of Food Science and Technology 30: 391-402.

Shamsudin, R. 2000. Design, concentration and Testing of freeze concentration, PhD Thesis, University Putra Malaysia.

Sin, H.N., Yusof, S., Hamid, N.S.A. and Rahman, R.A. 2006. Optimization of enzymatic clarification of sapodilla juice using response surface methodology. Journal of Food Engineering 73: 313-319.

Smith, P.G. 2003. Evaporation and drying. In Introduction to food process engineering,(Eds). D.R. Heldman, p. 303-334. NewYork: Kluwer Academic/ Plenum Publishers.

Turk, T.C. 2002. The effect of low temperature storage treatment on color changes in pomelo *(Citrus grandis* (L) *Osbeck*). Serdang, Malaysia: University Putra Malaysia, MSc thesis,

Thijssen, H.A.C. 1975. Current developments in the freeze concentration of liquid foods. In Goldblith, S.A., Rey L. and Rothmayr, W. W. (Ed.), Freeze Drying and Advanced Food Technology, p. 481–501. New York: Academic Press.

Wong, P.K., Yusof, S., Ghazali, H.M. and Che Man, Y. 2003. Optimization of hot water extraction of Roselle juice by using response surface methodology: a comparative study with other extraction methods. Journal of the Science of Food and Agriculture 83: 1273-1278.

Yusof, S., Talib, Z., Mohamed, S. and Bakar, A. (1988). Use of response surface methodology in the development of guava concentrate. Journal of Science and Food Agricultural 43: 173-186.