

Characterization of Mixed Apple and Carrot Retentates Using Response Surface Methodology

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Abstract

Models capable of predicting the product quality of mixed apple and carrot retentates (MACR) have been developed using response surface methodology and used to characterize the effects of processing conditions including average transmembrane pressure (ATP), temperature, and blend ratio. Color, soluble solids, total sugar, vitamin C, acidity, turbidity, and viscosity were used to assess the product quality following the ultrafiltration (UF) process. L^* -value decreased with increased ATP, but the value was not affected by changes in temperature. Blend ratio also greatly influenced the L^* -value. Redness (a^* -value), on the other hand, was less affected by temperature and ATP. As the ATP and temperature increased, yellowness increased gradually. Soluble solids contents appeared to decrease gradually as the ATP increased for all blend samples, but the effect of temperature seemed to be less. Total sugar content was more affected by temperature than ATP. In general, samples containing 75% carrot had higher amounts of vitamin C regardless of processing conditions. Changes in acidity were also complex and appeared to respond to interactions among ATP, temperature, and blend ratio. Turbidity increased for all samples as both ATP and temperature increased. The higher the amount of carrot in the blend samples, the higher values for turbidity. Although the changes were small, viscosity appeared to increase as the ATP and temperature increased during UF.

Key words: ultrafiltration, blend juice, retentate, quality, RSM

INTRODUCTION

Fruit and vegetable juices are favored by many consumers because of their high nutritional value since they are rich in minerals, vitamins and other beneficial nutrients for human health. In general, there are presently two types of fruit juice products on the market: fresh juices, simple squeezing and then submitted to a mild pasteurization; and juices reconstituted from concentrate (1). Clarified juice prepared by an ultrafiltration (UF) process can also be subjected to a concentration process in order to obtain a product suitable for the preparation of juices and beverages.

UF can be used as a unique operation for the clarification and pasteurization of fruit juice (2) and offers an efficient and reliable means to clarify noncloudy-type fruit and vegetable juices (3). The separating capability of UF can be described as the rejection coefficient of a membrane against a specific molecular weight (4). Large species such as microorganisms, lipids, and proteins are retained by UF membranes, while small solutes including vitamins, salts, and sugars flow through the

membrane together with water (1).

Several studies have been evaluated UF clarification of juices (5-13). Unfortunately, most of the research has focused on the permeate, but investigation on the retentate (residual fibrous phase) is scarce. The retentate coming from the UF process could be submitted to a stabilizing treatment and reused for the preparation of beverages enriched in fibers.

Therefore, the present work is designed to study the effects of UF processing conditions (average transmembrane pressure, temperature) and blend ratio on the quality (color, soluble solids, total sugar, vitamin C, acidity, turbidity, and viscosity) of mixed apple and carrot retentate (MACR) using response surface methodology.

MATERIALS AND METHODS

Sample preparation

Fresh apples ("Busa") and carrots were obtained from a local market in 20 kg lots and stored at 4°C until further processing for less than 2 weeks. Each sample was washed with tap water and sorted. Decayed fruit was discarded.

Carrot samples were blanched for 30 sec in 80°C water and cooled in cold water. Juice was then extracted using a commercial juicer (model DO-9001, Donga-osca, Co., Korea) to extract juice. Ascorbic acid (2 g per 1 L sample) was added to each extracted juice to prevent color degradation. Each extracted juice was then centrifuged (4°C, 10,000 rpm, 15 min) and the supernatant was filtered to remove remaining solid particles using an AP25 filter (Millipore Corp., Bedford, USA). Pre-filtered apple and carrot juices were blended at the ratios of 1:3, 1:1, and 3:1 (v/v, %) prior to the UF.

Ultrafiltration

A plate-type UF system (Minitan™ II, Millipore Corp., Bedford, USA) was used to remove suspended solids in the juice blends. Four low binding regenerated cellulose UF membranes (effective area = 2.4 m², Biomax polysulfone membrane) with a nominal molecular weight cutoff (MWCO) point of 10,000 Daltons were used. A peristaltic pump (MasterFlex L/STM, model No. 7523-20, Barnant Co., Barrington, USA) was used to sustain the pressure in the system. The system was operated at average transmembrane pressures (ATP) of 100, 150, or 200 kPa and sample temperatures of 5, 25 or 45°C. Juice samples were centrifuged at 480 rpm for 30 min prior to the sample characterization.

Color

Color parameters were measured using a Minolta Chroma Meter 210 (Minolta Camera Co., Ltd., Osaka, Japan) using the CIE 1976 Chromameter L*a*b* color scale equipped with a standard C illuminant using a 0° illumination angle and a 0° viewing angle. Samples were presented in 2 mm thick glass cuvettes, and calibration was done with distilled water. The reported color parameters are the mean values of three observations.

Soluble solids, total sugar and vitamin C

Soluble solids were determined using a hand refractometer (Type N1, Atago Co., Japan). Total sugar was determined by the phenol-H₂SO₄ method; 1 mL of sample was mixed with 1 mL 5% phenol, and the mixture was then allowed to react with 5 mL H₂SO₄ for 20 min at room temperature. The absorbance was measured at 480 nm. Ascorbic acid was determined by 2,4-dinitrophenylhydrazine titration method. All measurements were done in triplicate.

Acidity, turbidity and viscosity

Acidity was determined by titration and expressed as the amount of 0.1 N NaOH solution used to neutralize 20 mL of juice. Turbidity was measured using a UV spectrophotometer (model UV-1201, Shimadzu Co., Kyoto, Japan) at A₆₆₀ nm. Viscosity of the sample was

Table 1. Independent variables and their coded and actual values used for analysis

Independent variable	Unit	Symbol	Coded levels		
			-1	0	1
Temperature	°C	X ₁	5	25	45
Pressure	kPa	X ₂	100	150	200
Blend ratio	Apple (v/v, %)	X ₃	25	50	75

measured using a Brookfield viscometer (model LDVD-II+, Brookfield Engineering Labs, USA) at 25°C. An UL adaptor with ULA-DIN-Y spindle was used and the shear rate was at 122.3 sec⁻¹. All measurements were done in triplicate.

Experimental design and analysis

An attempt was made to fit a multiple regression equation describing quality composition attributes and the design depended on the symmetrical selection of variation increments about the center composition. The increments of variation for each variable spaced around the center point are presented in Table 1.

A central composite experimental design (Table 2), with three variables, was used to functionally relate the quality characteristics to process parameters. In this design, experiments were randomized in order to minimize the effects of unexplained variability in the observed response due to extraneous factors. The function was assumed to be proximate to a second-order polynomial equation:

$$Y_k = b_0 + \sum_{i=1}^3 b_i X_i + \sum_{i=1}^3 b_{ii} X_i^2 + \sum_{i \neq j=1}^3 b_{ij} X_i X_j$$

where b_0 was the value of fitted response at the center point of design, and b_i , b_{ii} and b_{ij} were the linear, quadratic and cross-product regression coefficients, respectively.

RESULTS AND DISCUSSION

Statistical analysis

The experimental values for color (L*, a*, and b*-value), soluble solids, total sugar, and vitamin C contents, acidity, turbidity, and viscosity under different treatment conditions are presented in Table 2. The regression coefficients for the second order polynomial equations and results for the linear, quadratic and interaction terms are presented in Table 3. Regression analyses for all the models indicated that the fitted quadratic models accounted for more than 76% of the variations in the experimental data except for L*- and a*-values. The proposed models for b*-value, flux, and soluble solids were found to be highly significant and

Table 2. The central composite experimental design with the observed responses

Treatment No.	Variable levels			Color			Soluble solids (°Brix)	Total sugar (mg/mL)	Vitamin C (mg%)	Acidity (mL)	Turbidity (A ₆₆₀)	Viscosity (cP)
	X ₁	X ₂	X ₃	L*	a*	b*						
1	1	1	1	78.8	0.55	11.7	11.4	92.81	201.46	11.76	1.281	1.56
2	1	-1	1	82.9	0.47	11.0	11.4	91.09	203.71	16.22	1.054	1.28
3	0	0	1	72.3	1.01	14.8	11.4	136.44	242.11	9.45	1.957	1.43
4	-1	1	1	86.5	0.02	12.9	11.2	34.17	328.06	6.72	0.509	1.46
5	-1	-1	1	85.2	0.13	12.9	11.2	41.46	286.77	6.74	0.521	0.94
6	1	0	0	67.5	1.04	14.3	10.6	63.80	129.55	8.28	1.995	1.97
7	0	1	0	62.2	0.92	16.2	9.8	63.11	183.12	20.53	2.270	2.11
8	0	0	0	68.6	1.02	15.9	9.8	95.41	331.43	7.00	1.796	3.56
9	0	0	0	77.3	0.39	16.0	9.6	84.78	256.43	9.70	1.702	3.49
10	0	-1	0	78.8	0.21	14.8	9.6	72.93	219.35	7.23	1.593	2.69
11	-1	0	0	66.0	0.86	15.2	9.6	44.59	319.63	6.59	0.604	1.95
12	1	1	-1	73.9	0.59	17.7	9.0	56.26	197.44	6.29	1.790	1.46
13	1	-1	-1	72.0	0.66	19.7	9.0	58.67	349.97	6.53	1.913	1.36
14	0	0	-1	73.1	1.19	17.1	10.0	49.11	333.12	5.55	1.635	2.84
15	-1	1	-1	67.8	0.76	17.5	9.0	47.37	263.17	12.07	2.094	3.53
16	-1	-1	-1	84.5	0.06	15.2	10.0	45.90	344.92	5.90	0.693	1.40

Table 3. The equations derived using RSM for the prediction of the dependent variables (physicochemical properties)

Response	The second order polynomial	R ²
L*	$Y = 145.344875 - 0.273716X_1 - 0.617859X_2 - 0.964944X_3 - 0.000055172X_1^2 + 0.001647X_1X_2 + 0.001493X_2^2 - 0.000905X_1X_3 + 0.001202X_2X_3 + 0.009453X_3^2$	0.6670
a*	$Y = -4.183231 + 0.023398X_1 + 0.064256X_2 - 0.007357X_3 - 0.000215X_1^2 - 0.000071875X_1X_2 - 0.000188X_2^2 + 0.000111X_1X_3 - 0.000066500X_2X_3 + 0.000102X_3^2$	0.7082
b*	$Y = 13.189500 + 0.294195X_1 + 0.040857X_2 - 0.084320X_3 - 0.002502X_1^2 - 0.000454X_1X_2 - 0.000086345X_2^2 - 0.001953X_1X_3 + 0.000021000X_2X_3 + 0.000343X_3^2$	0.8952
Soluble solids	$Y = 9.507500 - 0.041578X_1 + 0.031241X_2 - 0.090729X_3 + 0.000147X_1^2 + 0.000125X_1X_2 - 0.000137X_2^2 + 0.000350X_1X_3 + 0.000100X_2X_3 + 0.001054X_3^2$	0.9093
Total sugar	$Y = -33.359609 + 2.869890X_1 + 1.448595X_2 - 1.913343X_3 - 0.065264X_1^2 + 0.000642X_1X_2 - 0.004914X_2^2 + 0.021654X_1X_3 - 0.000462X_2X_3 + 0.019959X_3^2$	0.7644
Vitamin C	$Y = 444.752871 + 2.800881X_1 + 2.383722X_2 - 12.753566X_3 - 0.021940X_1^2 - 0.014291X_1X_2 - 0.012853X_2^2 - 0.037247X_1X_3 + 0.027331X_2X_3 + 0.086790X_3^2$	0.7689
Acidity	$Y = 19.105312 + 0.255578X_1 - 0.371387X_2 + 0.410588X_3 - 0.004704X_1^2 - 0.002106X_1X_2 + 0.001825X_2^2 + 0.006417X_1X_3 - 0.001641X_2X_3 - 0.002907X_3^2$	0.7917
Turbidity	$Y = -0.653394 + 0.108899X_1 + 0.006574X_2 + 0.014368X_3 - 0.001432X_1^2 - 0.000161X_1X_2 + 0.000023655X_2^2 + 0.000097250X_1X_3 - 0.000106X_2X_3 - 0.000121X_3^2$	0.7749
Viscosity	$Y = -3.161938 + 0.085369X_1 + 0.045958X_2 + 0.067984X_3 - 0.001659X_1^2 - 0.000284X_1X_2 - 0.000089379X_2^2 + 0.000637X_1X_3 - 0.000143X_2X_3 - 0.000782X_3^2$	0.7638

the R² values for each response were 0.8952, 0.9338 and 0.9093, respectively. R² measures the amount of variation in new data explained by the model.

Color

The response surfaces for color parameters (L*, a*, and b*-value) due to UF of MACR juice as a function of average transmembrane pressure (ATP), temperature, and blend ratio are shown in Fig. 1. It is evident that at a fixed blend ratio, the L*-value decreased with increases in ATP but the value was not affected by changes in temperature. Blend ratio also greatly influenced the L*-value. Samples with the blend ratio of 3:1 (apple:carrot) had the highest L*-value. It is interesting

to note that the amount of apple in the juice blends did not directly affect the brightness of the sample and similar results were found for permeate samples (data not shown).

Redness (a*-value), on the other hand, was less affected by temperature and ATP. An increase in ATP from 100 to 150 kPa gradually increased the a*-value, which then decreased thereafter. Similar results were reported for permeate juice (data not shown). a*-value of MACR juice was higher than that of permeate juice (data not shown) which was due to the green color pigments (water soluble) passing through the membrane during UF processing, which resulted in relatively high amount of

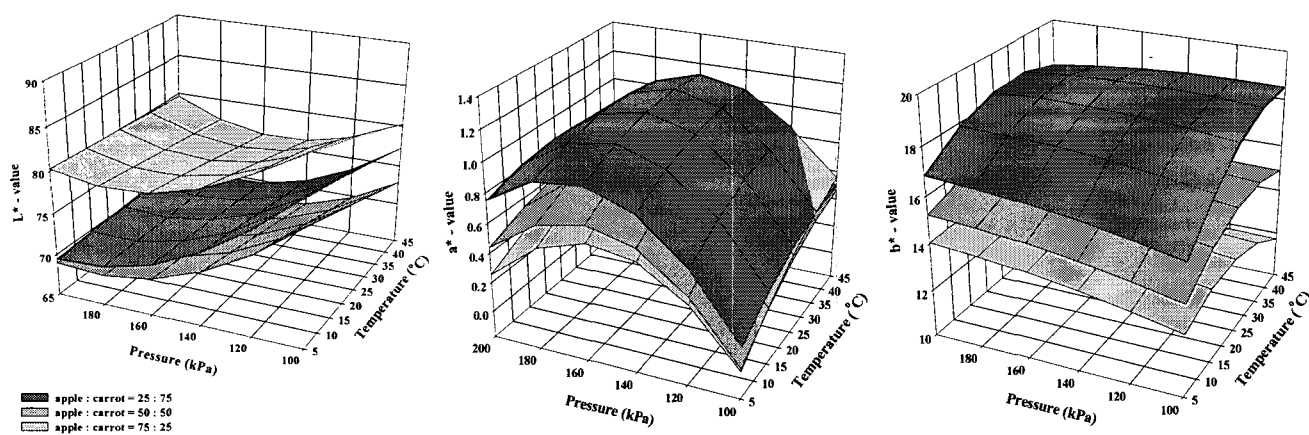


Fig. 1. Response surfaces for color characteristics of mixed apple and carrot retentate as a function of ATP, temperature and blend ratio.

red color pigment such as the carotenoids remained in the retentate (14). Thus, samples containing high amounts of carrot showed higher a^* -value.

Yellowness (b^* -value) was substantially affected by blend ratio than ATP or temperature. Higher amount of carrot in the blend sample resulted in higher b^* -values. As the ATP and temperature increased, yellowness increased gradually. These similar trends were also found for a^* -value and can be explained by reasons mentioned earlier.

Soluble solids, total sugar and vitamin C

Response surfaces for soluble solids, total sugar, and vitamin C contents of MACR as a function of ATP, temperature and blend ratio are presented in Fig. 2. Soluble solid contents appeared to decrease gradually as the ATP increased for all blend samples, but the effects of temperature seemed to be less. Blend samples with 75% apple showed distinctly higher soluble solids contents, which was perhaps due to the fact that apple

juice contained more soluble solids than carrot juice initially. Total sugar contents were more affected by temperature than ATP. Total sugar contents increased considerably as the temperature increased. Similar to soluble solids, blend samples with 75% apple had more total sugar than other samples. This may suggest that higher than room temperature conditions during UF may be required to retain high concentrations of soluble solids and total sugar in MACR.

Vitamin C contents in MACR ranged from 129.55 to 344.93 mg% (Table 2). Responses to ATP and temperature appeared to be rather complex depending upon the blend ratio. In general, samples containing 75% carrot had higher amount of vitamin C under most processing conditions.

Acidity, turbidity and viscosity

Response surfaces for acidity, turbidity, and viscosity of MACR as a function of ATP, temperature and blend ratio are shown in Fig. 3. Changes in acidity were also

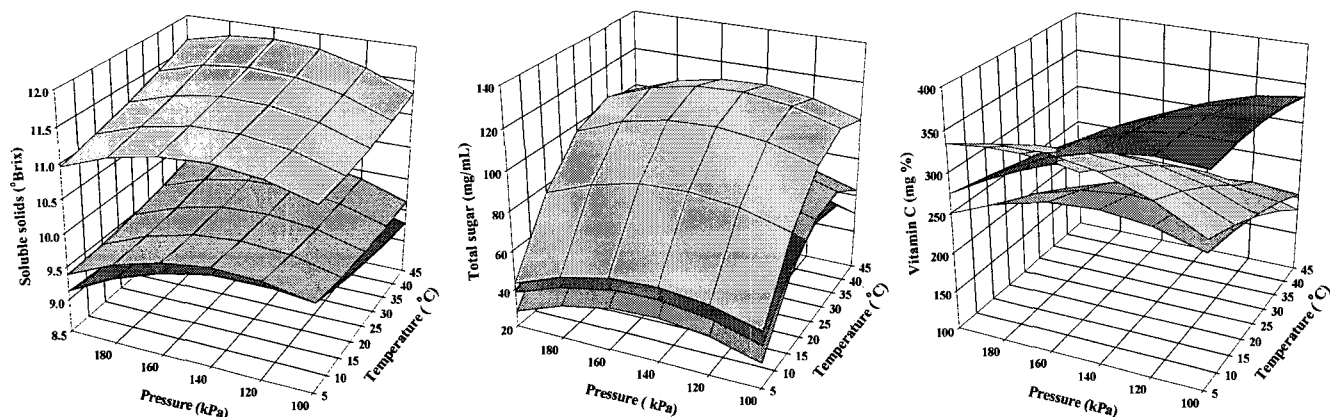


Fig. 2. Response surfaces for soluble solids, total sugar, and vitamin C contents of mixed apple and carrot retentate as a function of ATP, temperature and blend ratio.

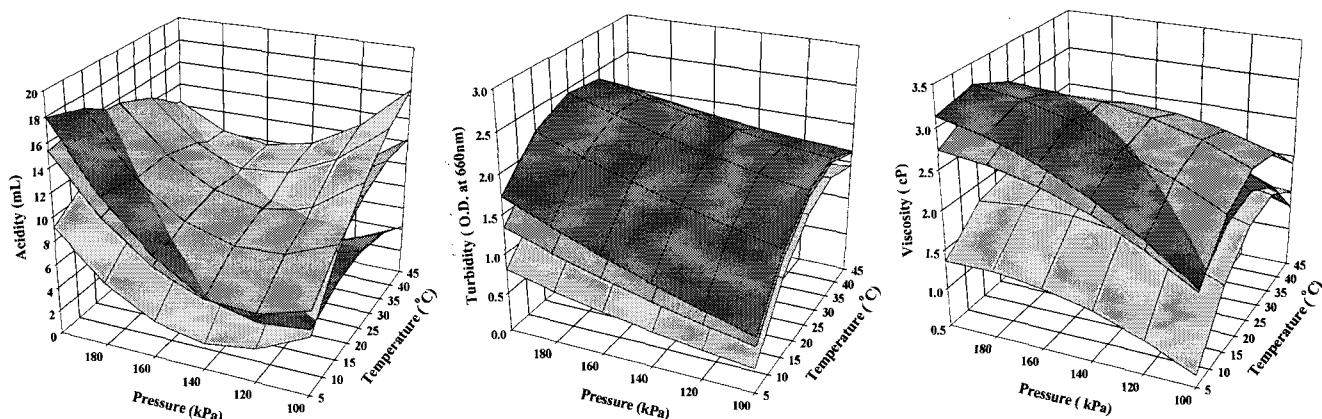


Fig. 3. Response surfaces for acidity, turbidity, and viscosity of mixed apple and carrot retentate as a function of ATP, temperature and blend ratio.

complex and appeared to have interactions among ATP, temperature, and blend ratio. At higher temperatures and lower ATP conditions, blend samples with 75% apple showed higher acidity; however, the values were lower at lower temperature and higher ATP conditions.

Turbidity in fruit juices can be a positive or a negative attribute depending on consumers' expectation (15). For clarified fruit juices, unstable cloud or "muddy" turbidity is unacceptable to be marketed as clear juices (16). Turbidity increased for all samples as both ATP and temperature increased. The higher the amount of carrot in the blend samples, the higher values for turbidity. This can be explained by the fact that retentate carrot juice showed significantly higher values for turbidity than did retentate apple juice (17).

Viscosities ranged from 0.94 to 3.56 cP (Table 2). Although the changes were small, viscosity appeared to increase as the ATP and temperature increased during UF. In addition, samples containing 75% apple showed relatively lower viscosity than others.

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