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Influence of thermal treatment, homogenization and xanthan gum on physicochemical properties of watermelon juice: A response surface approach

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ABSTRACT

Appearance of the watermelon juice, reflecting the safety and quality, is affected by the chemical reactions which take place during juice processing. In this study, the response surface methodology (RSM) was used to investigate the effect of xanthan gum (0.1-0.2 g/100 g), homogenization (7000 -10 000 rpm), heating temperature (80-90 °C) and time (4-10 min) on pectin methylesterase (PME) activity, cloud stability, lightness, fractal dimension (FD), pulp sedimentation and viscosity of the watermelon juice. The homogenization affected the structure of the pectin and reduced its accessibility to the PME. The viscosity of the juice increased as result of releasing of the pectin into the juice, adding gum and the PME thermal inactivation. These phenomena resulted in improvement of the homogeneity (FD reduction), cloud stability, sedimentation prevention and reduction in lightness of the juice. Based on the lowest PME activity, lightness, FD, highest cloud stability and viscosity, the optimum condition was 0.2 g/100 g gum, 10 000 rpm and heating at 89.95 °C for 6.58 min. At this condition, the accuracy of the models was verified as experimental values were very close to the predicted ones.

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1. Introduction

Watermelon (*Citrullus lanatus*) is grown in tropical or subtropical countries like Iran. The previous studies on processing of watermelon state that watermelon yields 41.5–60 g/100 g juice, 31–49.55 g/100 g rind and 8.9–23.59 g/100 g pomace (Oberoi & Sogi, 2015). Watermelon contains different vitamins such as thiamine, niacin, riboflavin, ascorbic acid and also minerals like calcium, phosphorous, iron and potassium (Sogi, Oberoi, & Malik, 2010). Due to amazing color, pleasant odor and sweet taste, watermelon is often served freshly and used in juice production.

Juice color is affected by chemical reactions occurring during process. Turbidity or cloudy appearance in some juices such as watermelon juice is a desirable quality attribute which influences the color and organoleptic properties (Tiwari, Muthukumarappan, O'donnell, & Cullen, 2009). Pectin has a significant effect on turbidity, viscosity and stability of the juice. There are lots of esterified carboxyl groups as methoxy groups in structure of the pectin. These groups prevent from precipitation occurs via link

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formation between galacturonic acids and cations such as calcium (Aadil et al., 2015). But in presence of enough amounts of deesterified carboxyl groups due to PME activity, the insoluble pectate is produced, resulting in cloud loss (Tiwari et al., 2009). These events increase the molecular weight of aggregates which resulted in precipitating and settling of the cloud particles at the bottom of the containers and the juice clarification. Thermal processing is a common and useful method in PME inactivation and preservation of the juice appearance (Aghajanzadeh, Kashaninejad, & Ziaiifar, 2016; Ibrahim et al., 2011). In addition, the lower PME activity resulted in the lower lightness of the juice color (Zhang et al., 2011).

Another effective method for maintaining the cloudy stability is application of hydrocolloids in the formulation of the juice. Hydrocolloids such as xanthan gum have many hydroxyl groups that participate in binding to water molecules and formation of gel and viscose solution (Akkarachaneeyakorn & Tinrat, 2015). Xanthan gum, an extracellular heteropolysaccharide, is used in formulation of different food products as a stabilizer, emulsifier, thickening agent and generally for mouth-feel modification in different beverages (Sallaram, Pasupuleti, Durgalla, & Kulkarni, 2014). Due to stability of the xanthan gum towards the temperature, ionic strength and pH, it is widely used in food industry (Liang et al.,





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2006). The rheological properties of the juice is important in the design of unit operations, process optimization and quality of the product (Augusto, Ibarz, & Cristianini, 2012). Usually, xanthan gum (up to 0.5 g/100 g) is used in juice formulation to bring the desirable viscosity and good mouth feel (Akkarachaneeyakorn & Tinrat, 2015).

Size distribution, surface charge and density of the cloud particles affect the sedimentation and cloud stability of the juice (Sallaram et al., 2014). Homogenization is considered as a nonthermal process that is frequently used in food processing. In this process, changes in size and structure of the suspended cloud particles causes modification of the rheological properties of the juice during a short treatment time (Liu et al., 2009). As, it was reported that the viscosity of watermelon juice increased by increasing in the size of suspended particles (Sogi et al., 2010). Studying fractal dimension (FD) is a useful method to quantify the color and structural changes of the food during different treatments. Fractal indicates complex geometry of the objects that can't be simply described by Euclidean dimension (Lopes & Betrouni, 2009). FD directly shows the compactness of aggregates where the more heterogeneous or irregular structure has the higher FD but the more homogeneous or smooth one has the lower FD (Nieuwland, Bouwman, Pouvreau, Martin, & de Jongh, 2016). Fractal was described as an effective indicator in the banana ripening (Quevedo, Mendoza, Aguilera, Chanona, & Gutiérrez-López, 2008). This parameter was also studied to investigate the enzymatic browning kinetics of different fruits (Quevedo, Díaz, Caqueo, Ronceros, & Aguilera, 2009; Quevedo, Díaz, et al., 2009; Ouevedo, Jaramillo, Díaz, Pedreschi, & Aguilera, 2009: Ouevedo et al., 2008). Also, FD was used for quantification of the oxidation of meat surface (Quevedo et al., 2013).

The PME activity and size of suspended particles consequently influence the color, viscosity and finally the quality of the juice; hence in this study, the effect xanthan gum, homogenization and thermal treatment on physic-chemical properties of watermelon juice was studied based on response surface method (RSM). In addition, the optimum conditions were determined according to PME activity, fractal dimension, pulp sedimentation, cloud stability, lightness and viscosity of the watermelon juice.

2. Materials and methods

2.1. Samples preparation

Fresh watermelon (*Citrullus lanatus*) was purchased from a local market (Gorgan, Iran) and kept at 4 °C until the experiments were carried out. The washed, clean watermelon was peeled and grounded. Then, the watermelon juice was filtered to remove seeds and pomace from the juice.

2.2. Experimental design

In this study, a central composite rotatable design (CCRD) was used to analyze the effect of four independent variables to examine the response pattern and also to determine their optimum synergy. These independent variables and their optimized ranges consisted of gum (0.1–0.2 g/100 g), speed of homogenization (7000–10 000 rpm), temperature (80–90 °C) and time (4–10 min) of thermal treatment. The ranges of these factors were chosen based on the results of preliminary experiments. The CCRD in the experimental design consisted of 16 factorial points and eight axial points (Table 1). Five replicates at the center of the design were used to estimate the error sum of squares.

According to Table 1, different percentages of the xanthan gum (Merck Co., Darmstadt, Germany) were added to 50 ml of

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Experimental range levels of four independent variables, A: gum (g/100 g), B: speed of homogenization (rpm). C: Temperature ($^{\circ}$ C), D: Time (min).

Run no	Codeo	Coded values			Actual	Actual values			
	A	В	С	D	A	В	С	D	
1	0	0	-2	0	0.15	8500	75	7	
2	$^{-1}$	$^{-1}$	$^{-1}$	+1	0.1	7000	80	10	
3	0	0	0	+2	0.15	8500	85	13	
4	+2	0	0	0	0.25	8500	85	7	
5	0	+2	0	0	0.15	11 500	85	7	
6	$^{-1}$	$^{-1}$	$^{-1}$	$^{-1}$	0.1	7000	80	4	
7	0	0	+2	0	0.15	8500	95	7	
8	+1	+1	$^{-1}$	$^{-1}$	0.2	10 000	80	4	
9	+1	+1	$^{-1}$	+1	0.2	10 000	80	10	
10	$^{-1}$	+1	+1	+1	0.1	10 000	90	10	
11	+1	+1	+1	+1	0.2	10 000	90	10	
12	-1	+1	+1	-1	0.1	10 000	90	4	
13	+1	$^{-1}$	+1	$^{-1}$	0.2	7000	90	4	
14	0	-2	0	0	0.15	5500	85	7	
15	0	0	0	0	0.15	8500	85	7	
16	+1	$^{-1}$	$^{-1}$	+1	0.2	7000	80	10	
17	0	0	0	-2	0.15	8500	85	1	
18	+1	$^{-1}$	+1	+1	0.2	7000	90	10	
19	0	0	0	0	0.15	8500	85	7	
20	-1	+1	$^{-1}$	$^{-1}$	0.1	10 000	80	4	
21	-1	$^{-1}$	+1	$^{-1}$	0.1	7000	90	4	
22	-1	$^{-1}$	+1	+1	0.1	7000	90	10	
23	0	0	0	0	0.15	8500	85	7	
24	0	0	0	0	0.15	8500	85	7	
25	0	0	0	0	0.15	8500	85	7	
26	+1	+1	+1	$^{-1}$	0.2	10 000	90	4	
27	+1	$^{-1}$	$^{-1}$	$^{-1}$	0.2	7000	80	4	
28	$^{-1}$	+1	$^{-1}$	+1	0.1	10 000	80	10	
29	-2	0	0	0	0.05	8500	85	7	

watermelon juice. To disperse the gum homogeneously, the juice was mixed using magnet stirrer for 1 h at 25 °C. The sample was then homogenized at different speed using silent crusher (Heidolph, Germany) for 5 min at room temperature (25 °C). The sample was transferred to a 100 ml covered beaker and heated at different times and temperatures according to CCRD in water bath (WNB-22, Memmert, Germany, 1800 W). Initial time was set as the juice reached to the desired temperature. The data were recorded using 1 mm diameter copper—constant thermocouple (T-type) and data logger (TC-08, Pichotechnology Co, UK). Finally, the sample was cooled in an ice-water bath.

Design-Expert Software (Version 9.0.6.2, 2015, Stat-Ease, Inc., Minneapolis, MN) was used to find the mathematical model for any response and to present the relationship between the dependent and independent variables as three dimensions curves. A second-order polynomial model was used to study the variations caused by linear and quadratic order effects in addition to their interaction effects (Mudahar, Toledo, & Jen, 1990) according to Eq. (1):

$$y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \sum \beta_{ii} x_i x_j$$
(1)

where, y represents the response or dependent variable (PME, FD, lightness, cloud stability). β_0 is a model constant. Regression coefficients including β_i and β_{ij} are linear (main effect) and quadratic coefficients, respectively. x_i and x_j are levels of the independent variables. The adequacy and fitness of the obtained models were evaluated using analysis of variance (ANOVA). Lack-of-fit test, R² (coefficient of determination), adjusted R² and predicted R² were determined to estimate the adequacy of the regression models (Nath & Chattopadhyay, 2007). Lack-of-fit determines a statistical model fits well. It describes the functional relationship between the experimental factors and the predicted response variable in the models.

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