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Optimization of Functional Properties of Three Stabilizers and *κ*-carrageenan in Ice Cream and Study of their Synergism

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ABSTRACT

To obtain an optimum formulation for stabilizers–emulsifier in ice cream, different combinations of three stabilizers, i.e. basil seed gum, guar gum, and carboxymethyl cellulose, at two concentrations of 0.15 or 0.35% with or without E471 emulsifier were studied using a simplex-centroid mixture design. The regression models for physical properties and texture smoothness of samples were also established. Generally, increasing ratios of basil seed gum in gums mixture increased the apparent viscosity of ice cream mixes and smoothness of texture, but decreased the melting rate. Increasing proportions of guar gum enhanced overrun of samples. Results suggested that the combination of 96.94% basil seed gum and 3.06% guar gum at the concentration of 0.35% with 0.15% emulsifier produced the optimum ice cream. Subsequently, the interaction of the obtained optimum formulation with κ-carrageenan as a secondary stabilizer was studied at two levels of 0.01 and 0.02%. The pH, draw temperature, overrun, hardness, and melting rate were measured through physical methods. Rheological and sensory analyses were also performed. Inclusion of κ-carrageenan in formulations decreased the values of instrumental hardness and improved the smoothness of the samples; while it had no significant effects on other properties such as draw temperature, overrun, sandiness, and foaminess.

Keywords: Frozen dessert, Hydrocolloid, Optimization, Secondary stabilizer, Quality.

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INTRODUCTION

Ice cream as a complex food consists of small air cells dispersed in a partially frozen continuous aqueous phase. There are many formulation and processing factors that influence the texture and acceptability of ice cream. Stabilizers are one such ingredient, which, in spite of their low consumption level in the formulation, impart specific and important functions to the finished product. These ingredients retard ice crystal growth and improve body, texture, and melting properties (BahramParvar and Mazaheri Tehrani, 2011). Despite the availability of many researches, there is no agreement on mechanisms through which stabilizers affect freezing properties or limit ice recrystallization (Marshall *et al.*, 2003).

A wide variety of gums such as xanthan, guar, locust bean gum, carboxymethyl cellulose, and alginate as primary stabilizers and *κ*-carrageenan as a secondary stabilizer are used in ice cream industry (Marshall *et al.*, 2003). Blends of these ingredients find many applications, because of specific characteristics of each stabilizer as well as synergistic effects between gums. The synergistic interactions of these polymers are useful in cost reduction (Trgo, 2003; Bahram Parvar and Mazaheri Tehrani, 2011). *κ*-carrageenan is used in many stabilizer blends at levels of 0.01–0.02% to prevent phase separation (wheying off) through its interaction with milk protei (Snoeren *et al.*, 1975; Dalgleish and Morris,

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1988; Bourriot *et al*., 1999; Schorsch *et al*., 2000; Spagnuolo *et al*., 2005). Emulsifiers which are also included in most stabilizer blends, have important functions such as reduced whipping time, controlled fat destabilization, increased smoothness, enhanced melting resistance and improved dryness (Baer *el al*., 1999).

Ocimum basilicum L. with vernacular name of basil (or Reyhan) is one of the endemic plants in Iran and is used as a pharmaceutical plant (Naghibi *et al*., 2005). Its seed, when soaked in water, swells into a gelatinous mass which has reasonable amounts of gum (Razavi *et al*., 2009). It has been reported that the polysaccharide extracted from basil seed is comprised of two major fractions of glucomannan (43%) and (1→4)-linked xylan (24.29%) and a minor fraction of glucan (2.31%) (Hosseini-Parvar *et al*., 2010). The presence of highly branched arabinogalactan in addition to glucomannan and (1→4)-linked xylan has also been shown (Hosseini-Parvar *et al*., 2010). Suitable functionality of this gum as a novel source of hydrocolloids in model systems has been recently proved (BahramParvar and Razavi, 2012; Hossini-Parvar *et al*., 2010, 2011).

The mixture design is now widely used in the formulation experiments of food, chemicals, fertilizers, pesticides, and other products. It can estimate the relationship between formulation and performance through regression analysis in fewer experiment times (Jian-Zhong *et al*., 2007). There are some valuable studies concerning the use of mixture design in optimization processes applied in food industry (Prinyawiwatkul *et al*., 1997; Abdullah and Cheng, 2001; Yang and Vickers, 2004; Laneuville *et al*., 2005; Zorba and Kurt, 2006; Jian-Zhong *et al*., 2007; Garcia *et al*., 2009; Ghorbel *et al*., 2009; Abdullah and Chin, 2010; Ryland *et al*., 2010).

We have recently studied the effects of different ratios of basil seed gum (BSG), guar gum (GG), and carboxymethyl cellulose (CMC) in ice cream formulations both with and without emulsifier, separately (BahramParvar *et al*., unpublished data). As an extension of our previous work, we aimed to optimize these variables, i.e. different ratios and concentrations of selected hydrocolloids as well as the presence or absence of the emulsifier in the formulation, simultaneously. In addition, more factors including both physical and sensory ones were assessed as responses. In the second step of our research, we investigated the interaction between the optimum primary stabilizers formulation obtained in the first step and κ-carrageenan as a secondary stabilizer.

MATERIALS AND METHODS

Ice Cream Preparation

In the first step of the research, ice cream formulations consisted of 10% fat (obtained from both cream and fluid milk), 11% milk solid non fat (MSNF, Multi Milk Powder Industry Co, Mashhad, Iran), 15% sucrose, 0.1% vanilla (vanillin 100%, Polar Bear Brand, China), 0 or 0.15% emulsifier (mono and diglyceride of fatty acids (E471), Multec Mono 9402 sfp, Puratus, Belgium), and 0.15 or 0.35% stabilizer. The BSG powder was prepared according to the work of Razavi *et al.* (2009). The CMC and GG were supplied by Sunrose (Mashhad, Iran) and Rhodia (Germany) companies, respectively. Fluid milk (homogenized-UHT with 3% fat, Pegah Dairy Industry Co, Mashhad, Iran) and cream (homogenized-pasteurized with 30% fat, Pegah Dairy Industry Co., Mashhad, Iran) were mixed and warmed up to 50°C. Then, pre-blended dry ingredients were added and mixed by a mixer (Model R10, Moulinex, France) for approximately 3 minutes prior to pasteurization (80°C for 25 seconds) and homogenization at the required speed (17000-22000 rpm) for 1 minute (Ultra Turax T-25, IKA Instruments, Germany). The produced mixes were cooled and aged overnight at 4°C before freezing in a batch freezer (Feller ice cream maker, Model IC 100, Feller Technologic GmbH, Germany) for 30 minutes. Ice creams were collected in 50 and 100 mL lidded plastic containers and hardened at -18°C within 24 hours after freezing. Two batches of each ice cream formulation were made and all experiments, except determination of draw temperature, were done twice per each batch. All analyses were carried out on the hardened samples.

In the next step of the research, all procedures of ice cream preparation, except the inclusion of two levels of κ-carrageenan (0.01 or 0.02%) (Fluka analytical 22048, Sigma-Aldrich Cheme GmbH, Denmark) were similar to the first step.

Physicochemical Analysis

pH

It was tested with a pH meter at 5°C (Metrohm pH meter, Model 691, Switzerland). Each sample was mixed thoroughly and pH was recorded (Abdullah *et al*. 2003).

Draw Temperature

Draw temperature (ºC) of ice creams was recorded using a digital thermometer (French Cooking, Biotemp, Alla France, France).

Overrun

It was calculated by comparing the weight of a known volume of ice cream (M_2) to the weight of the same volume of unfrozen ice cream mix (M_1) as follows (Marshall *et al.* 2003):

$$\text{Overrun}\ \% = \frac{M_1 - M_2}{M_2}\times 100 \qquad (1)$$

Hardness

Hardness of hardened ice creams, which were tempered at room temperature (21±0.5°C) for 5 minutes before the experiment, was measured using a texture analyzer (CNS Farnell Com, UK). Immediately prior to starting the test, temperatures at the geometric center of samples were -16±1ºC. For each sample, three measurements were carried out using a 6-mm stainless steel cylindrical probe. The penetration depth was 15 mm and the penetration speed was set at 2.0 mm s^{-1}. Hardness (g) of samples was determined as the peak compression force during penetration (Aime *et al.*, 2001; Soukoulis *et al.*, 2008).

Melting Rate

A 30-g sample cut carefully as a cube was suspended on a wire mesh and allowed to melt at room temperature (22±1°C). The weight of drained material through the wire mesh was recorded every 15 minutes. The weight of passing sample was plotted as a function of time. Melting rates were calculated from the slope of each melting chart (Soukoulis *et al.*, 2008; Karaca *et al.*, 2009).

Fat destabilization index (Turbidity)

The fat destabilization index in the melted ice cream samples was determined by spectrophotometry, according to the Bolliger *et al.* (2000) method. Mix and ice cream samples (3.00 g) were diluted 1:500 in two steps with distilled water and absorbance was measured by a spectrophotometer (WPA UV/Visible spectrophotometer, model diode – array S2000, Cambridge, England) at 540 nm against the same distilled water, which had been used for dilution, as a blank. Turbidity as the indicator of fat destabilization in the sample was calculated by the following equation:

Fat destabilization index= [(A_{540}(Diluted mix)-A_{540}(Diluted melt))/(A_{540}(Diluted mix)] ×100. (2)

Rheological Analysis

The steady shear rheological behavior of the aged ice cream mixes at 5±0.5°C was

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determined using a rotational viscometer (Bohlin Model Visco 88, Bohlin Instruments, UK) equipped with a heating circulator (Julabo, Model F12-MC, Julabo Labortechnik, Germany). The ice cream mixes after aging were subjected to shear rates ranging from 14.2 to 512 s^{-1} and apparent viscosity at the shear rate of 117 s^{-1} was chosen for comparison.

Sensory Evaluation

In the first part of our research, the forty ice cream formulations were evaluated for degree of smoothness by ten trained panelists, six females and four males, all between the ages of 22 and 31. High degree of smoothness was defined as a smooth and uniform spread of sample onto the palate without any detectable coarse or rough texture (Aime *et al.*, 2001). Twelve panel sessions were established and three or four samples were assessed in each one. The hardened samples were presented in a random order.

After optimization, a scale of 1 to 9 was used for hedonic sensory evaluation of the optimum formulation, with 1 being "dislike extremely" to 9 being "like extremely. 57 panels, 25 men and 32 female aged 20-50, were selected from students and staffs of the Department of Food Science and Technology, Ferdowsi University of Mashhad, Iran.

In the second step of the research, descriptive sensory analysis of ice creams containing the optimum stabilizer-emulsified blend and different percentages of *κ*-carrageenan was conducted by seven trained panelists (three females, four males, aged 22 to 32 years). In order to screen panelists, in the first training session, ranking test of a series of solutions for intensity of sweet taste and triangle tests were done (Keane, 1992). Panelists who could recognize the sweetness order as well as a different sample in a triangle test were chosen for the sensory evaluation of ice creams. Panelists were selected from the student population. Three 30-minute training sessions were held and definition of attributes and assessment techniques were introduced and sample evaluation was done practically using a commercial regular fat vanilla ice cream. The ice creams were evaluated for sensory characteristics including viscosity, firmness, sandiness, foaminess, degree of smoothness, liquefying rate, and mouth coating according to their definitions in Table 1 (Prindiville *et al.*, 1999; Aime *et al.*, 2001; Soukoulis *et al.*, 2008). All samples were served in 50 mL lidded plastic containers and evaluation was carried out in standard sensory booths under white lights. Ice creams were tempered at room temperature (24±1°C) for

Table 1. Terms used in descriptive analysis of ice cream containing 0, 0.01, or 0.02% *κ*-carrageenan.

Property	Definition
Viscosity	High viscosity means the sample does not move easily within the mouth during the melting process and immediately after the sample has liquefied and may feel sticky on the palate offering resistance to movement.
Firmness	Firmness is defined as the amount of force required by your tongue to flatten the ice cream.
Sandiness	The ice cream is characterized by grittiness (sand grains texture) which remains even after the sample has melted or expectorated.
Foaminess	It looks for bubbly foam in ice cream.
Degree of smoothness	A high degree of smoothness means the sample has a smooth and uniform spread onto the palate and no coarse or rough texture is detectable.
Liquefying rate	It could be determined by pressing the sample between tongue and the upper palate and determine the liquefying rate.
Mouth coating	It shows the amount of film remaining in the mouth after swallowing ice cream.

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5-7 minutes before the evaluation, in order to rise the temperature to -14±1°C. The ideal temperature for sensory evaluation of ice cream has been reported to be between -13 and -16°C (Aime *et al.*, 2001). During panel sessions, panelists were instructed to rinse their mouths with water before each test and also to rest between tests to avoid fatigue.

Experimental Design and Statistical Analysis

The simplex-centroid mixture design method was used to determine the optimum proportions and levels of selected stabilizers as well as level of emulsifier in ice cream. The selected hydrocolloids (BSG, GG, and CMC) were included as the independent variables. Stabilizer concentrations, 0.15 or 0.35%, and presence or absence of emulsifier were considered as process variables. Minitab statistical software (version 13.20, Minitab Inc., State College, PA) was used to provide experimental design (Table 2), calculate equations and make statistical evaluations. The feasible space for a mixture experiment with three components is a triangle called a simplex. All the mixtures in the simplex must have the same final weight (BSG+GG+CMC= 100) (Laneuville *et al.* 2005). The studied responses were apparent viscosity, draw temperature, overrun, turbidity, melting rate, and degree of smoothness. Different polynomial models were fitted to the experimental data. The models with the largest values of R^2 and adjusted-R^2 were preferred. The non-significant terms with *P*-values greater than 0.05 were deleted in final regression equations. Typically, positive values in model indicate synergistic effects while negative values represent antagonism (Mastromatteo *et al.*, 2009).

In the second step of the research, a randomized complete design was chosen for the experiment. Analysis of variance (ANOVA) was carried out using the general linear model procedure within Minitab statistical software. Means were compared using Least Significant Differences (LSD) test and results were considered significant for $P< 0.05$.

RESULTS AND DISCUSSION

Formulations Optimization and Verification

To obtain more detailed information, predictive equations for apparent viscosity, draw temperature, overrun, turbidity, melting rate and degree of smoothness were developed using mixture design (Table 3). Quadratic models were chosen for the analysis off apparent viscosity and melting rate data, while other responses were analyzed with full quartic models. The determination coefficients (R^2) of the models (> 82.3) exhibited a good correlation between the variables, suggesting that the fitted models were able to explain more than 80% of the total variation. Adjusted-R^2, which ranged between 71.18 and 88.96%, confirmed the adequacy of models as well.

Using the Minitab software, optimization calculations were performed to find optimum mixture proportions and concentrations. Desirability criteria for each factor are presented in Table 4. The optimum ratio was 96.94% BSG and 3.06% GG at the level of 0.35% with 0.15% emulsifier. This promising result confirmed the capability of BSG as ice cream stabilizer, since the highest portion of the optimized formulation consisted of this gum and it was able to meet almost all expectations of the responses. The predicted responses for apparent viscosity, draw temperature, overrun, turbidity, melting rate, and degree of smoothness were 1.030 Pa s, -4.45°C, 56.35 %, 17.00%, 0.11 g min^{-1}, and 7.63, respectively.

In order to validate the predicted optimum formula, an experiment with the optimized stabilizer formulation was conducted in two replications. Mean observed values for apparent viscosity, draw temperature, overrun, turbidity, melting rate, and degree

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Table 2. Mixture design with 3 components (Basil Seed Gum: BSG; Carboxymethyl Cellulose: CMC, Guar Gum: GG) and 2 process variables (gums and emulsifier concentrations).

Formulation No.	Stabilizers ratio			Stabilizer (%)	Emulsifier (%)
	BSG	CMC	GG		
1	100	0	0	0.15	0
2	0	100	0	0.15	0
3	0	0	100	0.15	0
4	50	50	0	0.15	0
5	50	0	50	0.15	0
6	0	50	50	0.15	0
7	33.33	33.33	33.33	0.15	0
8	66.67	16.67	16.67	0.15	0
9	16.67	66.67	16.67	0.15	0
10	16.67	16.67	66.67	0.15	0
11	100	0	0	0.35	0
12	0	100	0	0.35	0
13	0	0	100	0.35	0
14	50	50	0	0.35	0
15	50	0	50	0.35	0
16	0	50	50	0.35	0
17	33.33	33.33	33.33	0.35	0
18	66.67	16.67	16.67	0.35	0
19	16.67	66.67	16.67	0.35	0
20	16.67	16.67	66.67	0.35	0
21	100	0	0	0.15	0.15
22	0	100	0	0.15	0.15
23	0	0	100	0.15	0.15
24	50	50	0	0.15	0.15
25	50	0	50	0.15	0.15
26	0	50	50	0.15	0.15
27	33.33	33.33	33.33	0.15	0.15
28	66.67	16.67	16.67	0.15	0.15
29	16.67	66.667	16.67	0.15	0.15
30	16.67	16.67	66.67	0.15	0.15
31	100	0	0	0.35	0.15
32	0	100	0	0.35	0.15
33	0	0	100	0.35	0.15
34	50	50	0	0.35	0.15
35	50	0	50	0.35	0.15
36	0	50	50	0.35	0.15
37	33.33	33.33	33.33	0.35	0.15
38	66.67	16.67	16.67	0.35	0.15
39	16.67	66.67	16.67	0.35	0.15
40	16.67	16.67	66.67	0.35	0.15

of smoothness were 1.220 Pa s, -4.00°C, 55.53 %, 15.28 %, 0.07 g min^{-1}, and 7.77 respectively. It was found that observed responses were close enough to the predicted one, indicating the adequacy of optimization process.

Hedonic sensory evaluation of ice cream containing optimum formulation of multiple stabilizers-emulsifier combinations showed scores of 6.82, 6.30, 7.70, 6.91, and 7.39 for appearance, color, flavor, body and texture, and total acceptance, respectively. As these results demonstrate, all characteristics scored good or excellent. This means that the optimum formulation not only produced desirable physical properties in ice cream,

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Table 3. Predictive regression models describing the relationship between variables and responses [a].

Attribute	Predictive model	R^2	R^2-(adj)
Apparent viscosity	$Y = 0.54BSG + 0.26CMC + 0.29GG - 1.70(BSG\times GG) + 0.39(BSG\times X_1) + 0.17(CMC\times X_1) + 0.11(GG \times X_1) + 0.08(BSG\times X_1X_2)$	91.51%	88.96%
Draw temperature	$Y = -5.41BSG - 4.68CMC - 4.34GG + 2.45(BSG\times CMC) + 1.25(BSG\times GG) - 26.66(BSG\times GG) + 4.16(CMC\times GG\times X_1) - 24.96(CMC\times GG\times X_1) + 0.46(BSG\times X_2) - 2.65(BSG\times CMC\times X_2) - 1.15 (BSG\times GG\times X_2) + 4.69(CMC\times GG\times X_2) + 79.28(BSG\times CMC^2\times GG\times X_2) + 22.06(CMC\times GG\times X_2)$	88.68%	80.81%
Overrun	$Y = 47.5BSG + 54.2CMC + 52.2GG - 77.8(BSG\times CMC) + 764.9(BSG\times CMC^2\times GG) + 6.4(BSG\times X_1) - 5.9(GG\times X_1) + 39.4(CMC\times GG\times X_1) - 4.5(GG\times X_2) - 19.9(CMC\times GG\times X_1X_2)$	82.26%	71.18%
Turbidity	$Y = 11.4BSG + 13.9CMC + 9.7GG + 18.7(BSG\times CMC) - 238.2(BSG\times CMC\times GG) - 19.3(CMC\times GG\times X_1) - 48.9(BSG\times GG\times X_1 + 326.7(BSG\times CMC^2\times GG\times X_1) + 4.0(BSG\times X_2) + 4.7(CMC\times X_2) - 22.4(BSG\times CMC\times X_2) + 13.3(BSG\times GG\times X_2) + 13.1(CMC\times GG\times X_2) + 330.4(CMC\times GG\times X_2) - 4.1(CMC\times X_1X_2) - 2.8(GG\times X_1X_2) + 17.1(BSG\times GG\times X_1X_2)$	92.72%	84.22%
Melting rate	$Y = 0.36BSG + 0.69CMC + 0.66GG - 0.89(BSG\times CMC) + 1.46(BSG\times GG) - 0.20(BSG\times X_1) - 0.30(BSG \times GG\times X_2) - 0.42(BSG\times GG\times X_1X_2)$	88.95%	84.61%
Degree of smoothness	$Y = 6.80BSG + 6.76CMC + 6.78GG - 1.79(BSG\times CMC) - 4.43(BSG\times CMC) - 30.06(BSG\times CMC) + 0.27 BSG\times X_1) + 0.29(CMC\times X_1) + 0.36(GG\times X_1) - 1.25(BSG\times CMC\times X_1) - 1.07(CMC\times GG\times X_1) - 3.61 (BSG\times GG\times X_1) + 32.58(BSG^2\times CMC\times GG\times X_1) + 20.60(BSG\times GG\times X_1) + 0.38(GG\times X_2) + 1.63(BSG \times CMC\times X_2) - 36.66(BSG\times CMC\times GG^2\times X_2) - 29.46(BSG\times GG\times X_2) - 17.6(CMC\times GG\times X_2) + 0.31(BSG \times X_1X_2 + 0.52(GG\times X_1X_2)$	94.72%	87.89%

[a] BSG, CMC, GG, X_1, and X_2 represent basil seed gum, carboxymethyl cellulose, guar gum, gum concentration, and emulsifier concentration, respectively.

Table 4. Desirable range for each response in optimization process.

Parameters	Goal	Lower	Target	Upper	Weight	Importance
Apparent viscosity	Maximum	0.103	1.107	1.107	1	1
Draw temperature	Minimum	-6.20	-6.20	-3.15	1	1
Overrun	Maximum	25.45	65.22	65.22	1	1
Turbidity	Maximum	2.57	25.70	25.70	1	1
Melting rate	Minimum	0.053	0.053	0.802	1	1
Degree of smoothness	Maximum	5.60	9.00	9.00	1	1

but also helped to create pleasing sensory attributes in the product.

Interaction of the Optimum Formulation with *κ*-carrageenan

Physicochemical Characteristics

There was a significant difference among pH values of ice cream mixes (P< 0.05) that ranged from 6.38 to 6.49 (Table 5). The normal pH of ice cream mix containing 11% milk solid non fat is about 6.31 (Marshall *et al.* 2003). BahramParvar *et al.* (2009) reported the pH values of 6.46 to 6.60 for ice cream mixes containing 0.3-0.5% of selected hydrocolloids which is consistent with this research.

Ice cream samples had a draw temperature ranging between -4.00 and -4.7 ºC (Table 5). *κ*-carrageenan did not change the draw temperature of ice creams significantly (P> 0.05). The temperature of the mix at the completion of the freezing process should relate to the amount of freezing point depression in the mix because all samples were frozen for the same length of time. It has been stated that high draw temperature or low freezing point of a frozen dessert causes the formation of relatively large ice crystals giving a coarse texture after heat shock (Alvarez *et al.*, 2005).

The effect of the addition of *κ*-carrageenan on ice cream overrun was not significant (P> 0.05). Bahram Parvar *et al.* (2009) also reported that addition of 0.3-0.5% Balangu seed gum, palmate-tuber salep, or CMC did not significantly change air entrapment into ice cream produced in a batch freezer.

Several factors such as melting point, total solids, overrun and amount and type of stabilizer affect hardness (Marshall *et al.*, 2003). Hardness of the studied ice creams significantly decreased due to the κ-carrageenan (P< 0.05, Table 5). This decrease could be related to cryo-protective role of κ-carrageenan. This hydrocolloid is a gelling agent and its ability to control recrystallization is improved in the presence of milk proteins (Soukoulis *et al.*, 2008).

Melting rates of ice creams that ranged from 0.05 to 0.11 g min^{-1} were significantly different (P< 0.05, Table 5). The addition of

Table 5. Physicochemical characteristics of ice creams containing different levels of *κ*-carrageenan (0%: Formulation 1, 0.01%: Formulation 2, and 0.02%: Formulation 3) [a].

	Formulation 1	*Formulation 2*	*Formulation 3*
pH	6.45±0.01^{b}	6.49±0.01^{a}	6.38±0.00^{c}
Draw temperature (ºC)	-4.00±0.00	-4.60±0.85	-4.70±0.42
Overrun (%)	55.53±1.03	57.63±1.15	60.86±2.43
Hardness (g)	3659.50±221.32^{a}	2857.50±187.38^{b}	1437.00±272.94^{c}
Melting rate (g min^{-1})	0.07±0.00^{b}	0.05±0.00^{c}	0.11±0.00^{a}

[a] Superscript letters indicate significant difference (P< 0.05) among ice cream samples. Differences among other results are not significant (P> 0.05).

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0.01% κ-carrageenan to ice cream formulations decreased the melting rate of ice cream, while 0.02% κ-carrageenan increased the melting rate. Soukoulis *et al.* 2008 also showed that the addition of κ-carrageenan to ice creams did not affect the melting rate in a regular trend.

Rheological Characteristics

The power law model was chosen to describe the flow curves of ice cream mixes, since its two parameters –consistency coefficient and flow behavior index– showed excellent representation of the data for all ranges of shear rates applied in this research:

$$\tau = K\dot{\gamma}^n \quad (3)$$

Where, τ is the shear stress in Pa, K is the consistency coefficient in Pa s^n, $\dot{\gamma}$ is the shear rate expressed in s^{-1} and n is the flow behavior index (dimensionless). The parameters obtained for the power law model are summarized in Table 6.

Flow behavior indices lower than unity showed that all ice cream mixes had a non-Newtonian shear thinning behavior. The decrease in apparent viscosity of all mixes with increasing the shear rate has been related to the increased alignment of the constituent molecules of the system (Bahram Parvar and Mazaheri Tehrani, 2011). It has also been suggested that the aggregation of fat globules in ice cream is partly responsible for its shear thinning behavior (Aime *et al.*, 2001). Many researchers reported that ice cream mixes exhibit shear thinning behavior (Hagiwara and Hartel, 1996; Prindiville *et al.*, 1999; Aime *et al.*, 2001; Kaya and Tekin, 2001; Alvarez *et al.*, 2005; Soukoulis *et al.*, 2008; Bahram Parvar *et al.*, 2010) which is in agreement with the results of this study. κ-carrageenan addition in ice cream mixes led to the reinforcement of shear thinning possibly caused by gelation phenomena that is consistent with results of Soukoulis *et al.* (2008).

The apparent viscosities and consistency coefficients of samples ranged from 1.220 to 1.262 Pa s and from 19.51 to 20.41 Pa s^n, respectively. The consistency coefficients which are considered to be a measure of the viscous nature of food (Sopade and Kassum, 1992) were not changed significantly (P> 0.05) with the inclusion of κ-carrageenan. The apparent viscosity of typical ice cream mixes containing 0.3–0.5% Balangu seed gum, palmate-tuber salep and CMC varied from 0.037 to 0.745 Pa s at the shear rate of 113 s^{-1} (Bahram Parvar *et al.*, 2009). According to Soukoulis *et al.* (2009) results, the apparent viscosity of an ice cream mix containing 6% fat, 11% milk solid not fat, 16% sugar, 0.2% stabilizer (blend of guar gum and microcrystalline cellulose at 1:1 ratio) and 0.2% emulsifier (mono-diglycerides of fatty acids) was 3.52±1.62 Pa s at the shear rate of 50 s^{-1}.

Sensory Properties

Table 7 presents the results of some textural sensory attributes. Oral viscosity plays an important role in texture perception of fluids and semi-solid foods such as ice cream (Akhtar *et al.*, 2005). Similar to the results of instrumental analysis, κ-carrageenan enhanced the values of sensory viscosity but not significantly (P> 0.05).

Table 6. Rheological characteristics of ice creams containing different levels of κ-carrageenan (0%: Formulation 1, 0.01%: Formulation 2, and 0.02%: Formulation 3).

	Formulation 1	Formulation 2	Formulation 3
Apparent viscosity (Pa s)[a]	1.220±0.006	1.253±0.034	1.262±0.030
n	0.42±0.02	0.41±0.03	0.40±0.03
K (Pa s^n)	19.51±1.63	19.94±1.66	20.41±1.97
r	0.998	0.998	0.997

[a] At the shear rate of 117 s^{-1}.

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Table 7. Sensory characteristics of ice creams containing different levels of κ-carrageenan (0%: Formulation 1, 0.01%: Formulation 2, and 0.02%: Formulation 3)[a].

	Formulation 1	Formulation 2	Formulation 3
Viscosity	6.09±0.85	6.89 ±0.88	7.03±0.95
Firmness	6.37±0.48[a]	5.47±0.74[b]	4.86±1.40[c]
Sandiness	2.60±1.99	2.43±1.81	2.40±2.11
Foaminess	1.43±0.79	1.57±0.79	1.89±1.08
Degree of smoothness	7.16±1.58	7.46±1.52	8.14±1.07
Liquefying rate	3.01±1.99	2.96±1.60	3.51±1.15
Mouth coating	3.93±1.54	4.86±1.07	4.86±2.60

[a] Superscript letters indicate significant difference (P< 0.05) among ice cream samples. Differences among other results are not significant (P> 0.05).

Bahram Parvar and Mazaheri Tehrani (2011) reported that many of sensory attributes of ice cream are related in some way to rheological properties. Some studies have attempted to correlate viscosity and sensory properties (Mela *et al.*, 1994; Aime *et al.*, 2001; Minhas *et al.*, 2002; Akhtar *et al.*, 2005, 2006; Soukoulis *et al.*, 2008; BahramParvar *et al.*, 2010).

Firmness of samples was in the range of 4.86 and 6.37. The presence of κ-carrageenan significantly decreased the resistance of the sample to deformation by the tongue thereby contributing to the higher firmness of ice creams (P< 0.05) that were in agreement with the results of instrumental hardness.

Sandiness that is defined as the crystallization of milk sugar or lactose in the finished ice cream (Marshall *et al.*, 2003) did not change significantly (P> 0.05) with addition of κ-carrageenan. All samples gained low scores of sandiness (2.40–2.60) that shows the selected hydrocolloids was able to act suitably to reduce lactose crystallization. Stabilizers help to hold lactose in supersaturated state and amorphous (non-crystalline) state through viscosity enhancement (Goff and Hartel, 2004).

Values of foaminess that ranged from 1.43 to 1.89 were not significantly different (P> 0.05). There was an increasing trend in foaminess with the addition of k-carageenan that was observed in the case of overrun as well.

All samples had excellent degrees of smoothness (7.16 to 8.14) that was increased with κ-carrageenan addition in formulations. This trend was similar to the change in viscosity with the addition of κ-carrageenan; since as the viscosity increases, the smoothness of texture improves (Bahram Parvar and Mazaheri Tehrani, 2011). The degree of smoothness depends on the sizes of the substances suspended in the disperse phase of the product. These suspended substances are ice crystals, fat globules, casein micelles, particles of other proteins, and air cells (Marshall *et al.*, 2003).

κ-carrageenan did not cause a significant difference in melting rates of samples (P> 0.05). The melting rate of ice cream is influenced by many factors, including the amount of air incorporated, the nature of the ice crystals, and the network of fat globules formed during freezing (Muse and Hartel, 2004). BahramParvar *et al.* (2010) reported the sensory values of 4.30 to 6.30 for liquefying rate of ice creams containing 0.3-0.5% palmate-tuber salep, Balangu seed gum and CMC as stabilizers.

No significant differences in mouth coating were detected between samples (P> 0.05, Table 7). All samples had intermediate scores of mouth coating (3.93–4.86) that were acceptable.

CONCLUSIONS

This study successfully demonstrated the feasibility of mixture design application for

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the optimization of formulation for stabilizers-emulsifier blends. The combination of 96.94% basil seed gum and 3.06% guar gum at the concentration of 0.35% with 0.15% emulsifier was proposed as the optimum formulation and was verified in practice. The interactions between the optimum stabilizers-emulsifier and *κ*-carrageenan (as a secondary stabilizer of ice cream) were also investigated. Draw temperature and hardness of samples decreased in the presence of *κ*-carrageenan, while overrun and apparent viscosity increased. The addition of κ-carrageenan also enhanced the shear thinning of ice cream mixes. *κ*-carrageenan affected some sensory attributes of ice creams as well. The results of the present study are useful in the design of processing operation, frozen dessert development, evaluation and modeling, and assessment of product quality. Due to the functionality of κ-carrageenan on phase separation and cryo-protection, further research is necessary to take advantage of interactions between this gum and the novel blend during storage period.

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بهینه‌یابی ویژگی‌های عملکردی سه نوع صمغ و کاپاکاراجینان در بستنی و مطالعه اثر سینرژیستی آنها

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چکیده

برای به‌دست آوردن یک فرمول بهینه پایدارکننده – نامیزه کننده در بستنی، ترکیبات مختلفی از سه پایدارکننده، یعنی صمغ‌های دانه ریحان، گوار و کربوکسی‌متیل‌سلولز، در دو غلظت ۰/۱۵ و ۰/۳۵ درصد، با استفاده از طرح مخلوط مرکز هندسی سادک مورد مطالعه قرار گرفتند. وجود یا عدم وجود امولسیفایر E471 هم به عنوان یک تیمار در نظر گرفته شد. مدل‌های رگرسیونی برای ویژگی‌های فیزیکی مورد ارزیابی و نرمی بافت نمونه‌ها به‌دست آمدند. به طور کلی، افزایش سهم صمغ دانه ریحان در مخلوط باعث افزایش ویسکوزیته مخلوط و نرمی بافت و کاهش سرعت ذوب شد. در مقابل، نسبت‌های بالای گوار موجب افزایش حجم بیشتر بستنی شدند. بر اساس نتایج این پژوهش، ترکیبی از ۹۶/۹۴ درصد صمغ دانه ریحان و ۳/۰۶ درصد صمغ گوار در غلظت ۰/۳۵ درصد به همراه ۰/۱۵ نامیزه کننده بهترین نتیجه را در پایداری بستنی ایجاد کرد. سپس، برهم‌کنش این فرمول بهینه با پایدارکننده ثانویه کاپاکاراجینان در دو سطح ۰/۰۱ و ۰/۰۲ درصد مورد مطالعه قرار گرفت. در این مرحله، خصوصیات رئولوژیکی، حسی، تعیین دمای خروج از بستنی‌ساز، pH، سفتی و سرعت ذوب تعیین گردیدند. افزایش کاپاکاراجینان به فرمولاسیون، سفتی نمونه‌ها را افزایش داده و نرمی بافت آنها را بهبود بخشید؛ در مقابل، اثر معنی‌داری بر روی ویژگی‌هایی مثل دمای خروج از بستنی‌ساز افزایش حجم شنی بودن، و حالت کفی نداشت.

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