

## Modeling of rheological properties of mellorine mix including different oil and gum types by combined design, ANN, and ANFIS models

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**Abstract:** In the present study, the effects of 2 different oil types (soybean and olive oil) and 3 different gums (xanthan gum, sodium alginate, locust bean gum, and their blends) on the rheological and physicochemical properties (pH, titratable acidity, moisture, and color), overrun, melting rate, melting time, and sensory properties of frozen mellorine samples were determined. Apparent viscosity of all mix samples decreased with shear rate, meaning that mellorine mix samples showed shear thinning behavior. Mellorine mix samples showed Ostwald–de Waele flow behavior ( $R^2 \geq 0.9915$ ). Viscous synergy indexes were calculated to determine if the gums had synergic interaction. The viscous synergy index value of the xanthan and locust bean gum combination was found to be approximately 1.80, indicating synergic interaction between them. The effects of different gums on the apparent viscosity values at  $50 \text{ s}^{-1}$  ( $\eta_{50}$ ) were satisfactorily modeled by a modified power-law model. The adaptive neuro-fuzzy inference system (ANFIS) model was also found to be sufficient to predict apparent viscosity values based on the oil type, gum concentrations, and shear rate ( $R^2 = 0.9121$ ). According to the combined design, the optimum gum concentration was determined to be 56.3% xanthan gum and 43.7% locust bean gum.

**Key words:** ANFIS, ANN, combined design, gum, mellorine, rheology

### 1. Introduction

Cost is one of the main factors affecting consumer food product preferences. For that reason, low-cost production of alternative products has been of interest to food researchers. Mellorine, as an alternative to ice cream, is a good example of such a product. Mellorine is an ice cream or frozen dessert product in which all or some proportion of the milk fat is substituted with plant-based oil (Clarke, 2004; Keeney, 2012). Usage of vegetable oil instead of milk fat promotes human health, since milk fat contains saturated fatty acid in concentrations of approximately 60%–70% (Nadeem et al., 2009) and 0.25%–0.38% cholesterol (Mathur et al., 1999). The use of vegetable oil in ice cream formulation results in a balanced saturated and unsaturated fatty acid composition in the product and lower cholesterol content (Nadeem et al., 2009). Mellorine is widely consumed throughout the world due to its lower cost, cholesterol content, and saturated fatty acid composition compared to ice cream. However, the low cost of the product is not the only factor affecting consumer preference. In addition to cost, customers also take into consideration the textural and sensorial properties of the

product. Therefore, it is important both for the cost and the product's acceptability to provide the desired textural and sensory properties of mellorine by changing ingredients like oil or hydrocolloid type.

The fat or oil type in ice cream has a significant effect on the quality of the end products (Dogan and Akgul, 2005; Rossa et al., 2012), since it affects qualities like the creaminess (Koxholt et al., 2001), texture, and mouthfeel (Adapa et al., 2000; Dogan and Kayacier, 2007). In addition, fat contributes to the stabilization of the air phase as well as fat aggregation levels through surrounding air bubbles, which improves melting resistance (Granger et al., 2005) and ice recrystallization (Goff, 2002). Therefore, selecting the optimum oil type is an important factor in improving the quality of mellorine. Substitution of palm olein with milk fat at a level of 3% did not negatively affect the compositional properties, overrun value, flavor, or sensory properties of ice cream (Nadeem et al., 2009). Adhikari and Arora (1994) reported that, regarding textural characteristics, sensory scores of ice cream containing vegetable oils were lower than those of the control sample; this might be avoided by using different ingredients in the

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formula, such as emulsifiers and hydrocolloids. Similar to fat, hydrocolloids also play an important role in the quality of ice cream and similar products.

Hydrocolloids affect recrystallization prevention, enhance the viscosity of the ice cream mixes, improve texture and mouth feel, and affect the shape retention (Marhal et al., 2003). In the food industry, 2 or more hydrocolloids in combination are widely used due to the synergistic effect of combined use (Kayacier and Dogan, 2006). Moreover, hydrocolloids are the most important ingredients affecting the rheological properties of ice cream mix. The rheological properties of mellorine mix are important for energy calculations, process design, and equipment selection, particularly heat exchangers and pumps (Hsu and Lo, 2003). In addition, quality criteria of ice cream, like smooth texture and cooling sensation, might be improved by optimization of the rheological properties of the mix (Chang and Hartel, 2002; Dogan, 2007). The modeling of the rheological parameters is important for optimization purposes.

There are several models widely used for modeling purposes. The adaptive neuro-fuzzy inference system (ANFIS) and artificial neural network (ANN) models are nonlinear models that have been commonly used in the food industry to model input–output relationships (Jang and Sun, 1995), since foods are very complex systems. In the literature, there have been many studies about establishment of ANN or ANFIS models to predict rheological parameters (Ghoush and Samhour, 2008; Mohebbi et al., 2008; Karaman and Kayacier, 2011; Toker et al., 2012a, 2012b; Yalcin et al., 2012b; Yilmaz, 2012; Öztürk

et al., 2013; Toker and Dogan, 2013, Toker et al., 2013a). Although ANN and ANFIS models are sufficient for the prediction of dependent variables, they do not provide information about the relationship between the dependent and independent variables. For this purpose, a combined design might be used. Combined design methodology is the combined form of mixture design and process design, which can be utilized for the investigation of function of food ingredients, and it explains the importance of interaction among food ingredients. The most important advantage of this methodology is using the parameters of mixture design and process design at the same time. The simultaneous optimization of mixture formulation and process is very important for the food industry. To the best of our knowledge, there is no study about the use of combined design in the food industry area.

The aims of the present study are: 1) to determine the rheological, physicochemical (ash content, dry matter, color properties, pH), and sensory properties of mellorine produced with different vegetable oils (soybean and olive oil) and gums (xanthan gum, locust bean gum, and alginate), and 2) to establish several models (ANN, ANFIS, power-law, and combined design) for the estimation of apparent viscosity at a shear rate of  $50 \text{ s}^{-1}$ .

## 2. Materials and methods

### 2.1. Preparation of the mellorine samples

Skimmed milk powder, sucrose, locust bean gum, xanthan gum, sodium alginate, olive oil, soybean oil, and sunflower oil were obtained from Bayrak Food Co. (Kayseri, Turkey). Table 1 shows the oil and gum types used in the formulation

**Table 1.** Physicochemical properties of the mellorine samples including different oil and gum combinations.

Sample	Formulation		Moisture (%)	pH	Acidity (%)	Color properties		
	Gum type	Oil type				$L^*$	$a^*$	$b^*$
K	XG	Sunflower oil	$66.24 \pm 1.00^a$	$6.52 \pm 0.06^{de}$	$0.20 \pm 0.01^{cde}$	$61.13 \pm 1.00^a$	$-4.42 \pm 0.10^e$	$-1.49 \pm 0.10^g$
S1	XG	Soybean oil	$65.92 \pm 1.02^a$	$6.54 \pm 0.06^{cd}$	$0.21 \pm 0.01^{cde}$	$61.12 \pm 0.05^a$	$-4.94 \pm 0.01^g$	$-1.80 \pm 0.02^i$
S2	LBG	Soybean oil	$66.34 \pm 1.10^a$	$6.63 \pm 0.01^a$	$0.20 \pm 0.01^{def}$	$56.61 \pm 0.12^f$	$-5.20 \pm 0.04^h$	$-2.88 \pm 0.06^j$
S3	A	Soybean oil	$66.20 \pm 0.75^a$	$6.59 \pm 0.02^b$	$0.20 \pm 0.01^{defg}$	$59.73 \pm 0.27^{cd}$	$-5.40 \pm 0.04^i$	$-2.36 \pm 0.08^k$
S4	LBG + XG*	Soybean oil	$66.14 \pm 0.70^a$	$6.51 \pm 0.01^{ef}$	$0.20 \pm 0.01^{defg}$	$59.15 \pm 0.11^d$	$-4.95 \pm 0.01^g$	$-2.33 \pm 0.06^k$
S5	LBG + A	Soybean oil	$66.25 \pm 1.13^a$	$6.49 \pm 0.02^f$	$0.19 \pm 0.01^{fg}$	$58.04 \pm 0.29^e$	$-4.64 \pm 0.02^f$	$-1.60 \pm 0.04^h$
S6	XG + A	Soybean oil	$65.91 \pm 0.80^a$	$6.55 \pm 0.02^{cd}$	$0.19 \pm 0.01^g$	$60.15 \pm 0.33^{bc}$	$-4.94 \pm 0.03^g$	$-1.95 \pm 0.10^j$
Z1	XG	Olive oil	$65.64 \pm 0.23^a$	$6.41 \pm 0.02^g$	$0.22 \pm 0.01^c$	$59.39 \pm 0.41^d$	$-3.78 \pm 0.04^b$	$0.30 \pm 0.02^d$
Z2	LBG	Olive oil	$66.30 \pm 1.31^a$	$6.30 \pm 0.02^h$	$0.26 \pm 0.01^a$	$56.78 \pm 0.09^f$	$-4.70 \pm 0.03^f$	$-1.36 \pm 0.04^f$
Z3	A	Olive oil	$66.26 \pm 0.84^a$	$6.43 \pm 0.02^g$	$0.21 \pm 0.01^{cd}$	$58.18 \pm 0.53^e$	$-3.89 \pm 0.08^c$	$0.16 \pm 0.01^e$
Z4	LBG + XG	Olive oil	$66.29 \pm 1.19^a$	$6.55 \pm 0.02^c$	$0.21 \pm 0.01^{cd}$	$60.23 \pm 0.05^{bc}$	$-3.49 \pm 0.01^a$	$0.92 \pm 0.01^b$
Z5	LBG + A	Olive oil	$65.92 \pm 0.88^a$	$6.41 \pm 0.01^g$	$0.23 \pm 0.01^b$	$60.75 \pm 0.11^{ab}$	$-4.47 \pm 0.02^e$	$1.50 \pm 0.05^a$
Z6	XG + A	Olive oil	$65.81 \pm 1.11^a$	$6.31 \pm 0.01^h$	$0.25 \pm 0.01^{ab}$	$59.25 \pm 0.11^d$	$-4.28 \pm 0.02^d$	$0.58 \pm 0.01^c$

XG: Xanthan gum, LBG: locust bean gum, A: sodium alginate. \*: The gums were mixed in a ratio of 1:1 (w/w). Different superscript lowercase letters show differences between the samples at  $P < 0.05$ .

of the mellorine. The mellorine mix contained 14% sucrose, 11% skimmed milk powder, 7% vegetable oil, 0.3% emulsifier, and 0.2% gum. The vegetable oil was added at 30 °C, skimmed milk powder at 40 °C, sugar (half of the total sugar) at 50 °C, and dry mixture including the remaining sugar, emulsifier, and gums at 70 °C. The prepared mixture was heated to 85 °C and pasteurized at this temperature for 30 s. The mix was then cooled rapidly to 4 °C and aged at 4 °C for 22 h. All of the samples were prepared in triplicate.

**2.2. Physicochemical properties of the mellorine samples**

In order to determine titratable acidity, 5 g of sample was homogenized in 100 mL of distilled water. After the addition of phenolphthalein, it was titrated with 0.1 N NaOH until a pink color was observed and % acidity was calculated using the following equation:

$$\text{Acidity \%} = ((0.009w) / \text{weight of sample}) \times 100, \quad (1)$$

where *w* is the weight of the NaOH used up during titration (Kurt, 1990). The pH value of the samples was determined using a pH-meter (Inolab Terminal Level 3, Germany). Moisture content of the samples was determined according to the AOAC method (1990). Color values (*L*, *a*, *b*) of the samples were measured using a colorimeter (Lovibond RT Series Reflectance Tintometer, UK). The physicochemical measurements were replicated 3 times for each sample.

**2.3. Rheological properties of the mix samples**

Steady shear rheological measurements of the samples were carried out at 20 °C using a controlled stress rheometer (RheoStress 1, HAAKE, Germany) monitored by the RheoWin Data Manager (RheoWin Pro V. 4.0, HAAKE). Cone-plate configuration (cone diameter, 35 mm; angle, 4°; and gap size, 0.140 mm) was used for shearing. Measurements were carried out in the shear rate range of 1–100 s<sup>-1</sup>. Apparent viscosity versus shear rate data were obtained and fitted to the Ostwald–de Waele model. Consistency coefficient (*K*) and flow behavior index (*n*) values were calculated by using the following equation:

$$\sigma = K(\dot{\gamma})^n, \quad (2)$$

where  $\sigma$  is shear stress (Pa), *K* is the consistency coefficient (Pa s<sup>*n*</sup>), and  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>) and *n* is the flow behavior index (dimensionless).

As is known,  $\eta_{50}$  is the apparent viscosity at a shear rate of 50 s<sup>-1</sup>, which is accepted as the shear rate in the mouth (Bourne, 2002). The variation of the  $\eta_{50}$  value as a function of concentration was described by several models (Rao et al., 1984; Ibarz et al., 1987), which are power-law and exponential-type models, as follows:

$$\eta_{50} = \eta_1(C_1^{a_1}), \quad (3)$$

$$\eta_{50} = \eta_2 \exp(a_2 C_1). \quad (4)$$

As can be seen, Eqs. (3) and (4) are used for determination of the dependency of  $\eta_{50}$  on 1 component. In order to determine effects of more than 1 component, they were modified as follows:

$$\eta_{50} = \eta_1(C_1^{a_1}) + \eta_2(C_2^{a_2}) + \eta_3(C_3^{a_3}), \quad (5)$$

$$\eta_{50} = \eta_{1\text{exp}}(b_1 C_1) + \eta_{2\text{exp}}(b_2 C_2) + \eta_{3\text{exp}}(b_3 C_3), \quad (6)$$

where *C*<sub>1</sub>, *C*<sub>2</sub>, and *C*<sub>3</sub> are the concentration fractions of xanthan gum, locust bean gum, and sodium alginate in the formulation, respectively;  $\eta_1$ ,  $\eta_2$ , and  $\eta_3$  are the constants for concentration effect (Pa s); *a*<sub>1</sub>, *a*<sub>2</sub>, and *a*<sub>3</sub> are the constants of the power-law model; and *b*<sub>1</sub>, *b*<sub>2</sub>, and *b*<sub>3</sub> are the constants of the exponential type model.

**2.4. Estimation of ANFIS and ANN for prediction of  $\eta_{50}$  values based on shear rate, oil type, and gum concentration**

The ANFIS model was optimized with a backpropagation algorithm. While the inputs were selected as shear rate, oil type (oil type was coded by numbers), and gum concentrations (xanthan gum, locust bean gum, and sodium alginate) found in the formulation of the mellorine, the output was apparent viscosity. The ANFIS model was trained with 199 data and tested with 97 data. The type and number of membership functions were determined based on the root mean square error (RMSE), mean absolute error (MAE), and determination coefficient (*R*<sup>2</sup>) values, which are used for evaluation of the model's accuracy. The RMSE and MAE values were calculated by using the following equations (Cobaner et al., 2009).

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N [x - y]^2} \quad (7)$$

$$MAE = \frac{1}{N} \sum_{i=1}^N |x - y| \quad (8)$$

Here, *x* is the measured data, *y* is the predicted data, and *N* is the number of data. The model with the lowest RMSE and MAE and highest *R*<sup>2</sup> values was the best model for predicting output based on the determined inputs.

Another nonlinear model applied for the data obtained from the steady shear measurements was the ANN model, composed of 1 or more hidden layers. The data were divided into 2 parts, like in ANFIS. The inputs and output for the ANN were the same as those of the ANFIS model. The number of hidden layers and nodes was determined by the trial-and-error approach, which gave the best prediction results. The Levenberg–Marquardt technique

was used for training the ANN, since it is a more powerful and faster technique than the conventional gradient descent technique (Hagan and Menjah, 1994; Kisi, 2007). Both the ANFIS and ANN models were implemented with MATLAB (7.0.1.24704 (R14)) software.

### 2.5. Establishment of the combined design

In the present study, a combined design was used to evaluate the effect of different gum types (xanthan gum, locust bean gum, and sodium alginate) and oil types (soybean and olive oil) on the rheological properties of the mellorine samples (Design Expert 8.0.7.1 program). A, B, C, D, E, and Y represented the xanthan gum, sodium alginate, locust bean gum concentration, shear rate, oil type, and viscosity values, respectively. While the first 3 factors (A, B, and C), as the mixture design factors, had values from 0 to 1, values for D ranged from 1 to 100 s<sup>-1</sup> as the process design factor, and E was the categorical value (soybean or olive oil). A total of 84 different experimental points, not mentioned in the text, were used for modeling purposes. The highest R<sup>2</sup> value was obtained from a reduced quadratic × cubic model when α was 95%.

### 2.6. Overrun (%) values of mellorine samples

The overrun value of each mellorine sample was determined by comparing the weight of a known volume of mellorine mix (200 mL) with the weight of the same volume of frozen mellorine. Overrun (%) was calculated using the following equation:

$$\text{Overrun}(\%) = \frac{\text{weight of mellorine mix} - \text{weight of mellorine}}{\text{weight of mellorine}} \times 100 \quad (9)$$

### 2.7. Sensory evaluation of the samples

General acceptability, consistency, color and appearance, taste, and odor properties of ice cream samples were determined according to the method described in the ice cream standard described by the Turkish Standards Institute (1992). The panel consisted of 15 panelists who

were either academic staff or graduate students of the Department of Food Engineering at Erciyes University. The panelists were initially subjected to a preliminary training to familiarize them with the samples. The ice cream samples were evaluated using a hedonic scale, in which 2 reflected a very low preference while 5 reflected very high preference.

### 2.8. Statistical analysis

Statistical analyses were performed with SPSS 17.0.1 and the Design Expert 8.0.7.1 program. Duncan's test was used for the multiple comparisons of mean values.

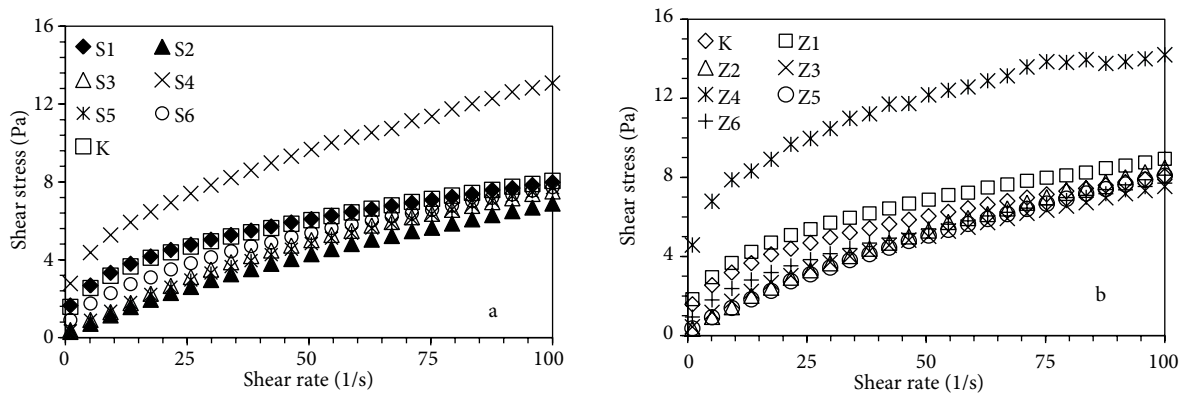
## 3. Results

### 3.1. Physicochemical properties of the mellorine mix samples

Table 1 shows the physicochemical properties of the ice cream mixes containing different oil and gum types. The moisture content of the samples ranged from 65.64% to 66.34%. pH values of the mellorine mix samples varied between 6.30 and 6.63 (Table 1). The titratable acidity (%) of the samples ranged between 0.19% and 0.26%. Contrary to pH values, the titratable acidity of the samples with olive oil was higher than that of the samples with soybean oil. The color values (*L* (lightness), *a* (redness), and *b* (yellowness)) of the samples are also shown in Table 1. As can be seen from Table 1, *L*, *a*, and *b* values of the mix samples changed from 56.61 to 61.13, -5.40 to -3.49, and -2.88 to 1.50, respectively.

### 3.2. Rheological properties of the samples

Figure 1 shows the effect of gums on the shear rate versus shear stress data of the mellorine samples with soybean and olive oil. Experimental rheological data of the mellorine mix samples were fitted using the Ostwald–de Waele model (Eq. (2)). The *K*, *n*, and R<sup>2</sup> values of the mix samples, including different oil and gum combinations, are tabulated in Table 2. As can be seen, R<sup>2</sup> values varied



**Figure 1.** Shear rate versus shear stress data of the mellorine mix samples. Figure 1a represents flow behavior of mellorine mixes including different gums for soybean oil, and Figure 1b for olive oil.

**Table 2.** Ostwald–de Waele model parameters and apparent viscosity values at a shear rate of 50 s<sup>-1</sup> of the mellorine mixes.

Sample	Ostwald–de Waele parameters			$\eta_{50}$ (Pa s)	$\eta_i$
	$K$ (Pa s <sup>n</sup> )	$n$	$R^2$		
K	1.29 ± 0.04 <sup>d</sup>	0.399 ± 0.005 <sup>d</sup>	0.9985	0.118 ± 0.019 <sup>d</sup>	-
S1	1.31 ± 0.06 <sup>d</sup>	0.380 ± 0.008 <sup>de</sup>	0.9994	0.119 ± 0.002 <sup>d</sup>	-
S2	0.28 ± 0.02 <sup>g</sup>	0.709 ± 0.010 <sup>a</sup>	0.9997	0.096 ± 0.002 <sup>e</sup>	-
S3	0.50 ± 0.03 <sup>f</sup>	0.617 ± 0.015 <sup>b</sup>	0.9990	0.102 ± 0.003 <sup>de</sup>	-
S4	2.10 ± 0.15 <sup>b</sup>	0.368 ± 0.043 <sup>e</sup>	0.9971	0.193 ± 0.005 <sup>b</sup>	1.80
S5	0.26 ± 0.02 <sup>g</sup>	0.726 ± 0.016 <sup>a</sup>	0.9998	0.096 ± 0.003 <sup>e</sup>	0.97
S6	0.82 ± 0.01 <sup>e</sup>	0.460 ± 0.012 <sup>c</sup>	0.9998	0.111 ± 0.003 <sup>de</sup>	1.00
Z1	1.69 ± 0.07 <sup>c</sup>	0.365 ± 0.011 <sup>e</sup>	0.9994	0.136 ± 0.002 <sup>c</sup>	-
Z2	0.35 ± 0.01 <sup>g</sup>	0.700 ± 0.008 <sup>a</sup>	0.9997	0.117 ± 0.002 <sup>d</sup>	-
Z3	0.45 ± 0.02 <sup>f</sup>	0.614 ± 0.017 <sup>b</sup>	0.9995	0.099 ± 0.002 <sup>e</sup>	-
Z4	2.66 ± 0.13 <sup>a</sup>	0.324 ± 0.017 <sup>f</sup>	0.9915	0.226 ± 0.017 <sup>a</sup>	1.79
Z5	0.29 ± 0.01 <sup>g</sup>	0.713 ± 0.009 <sup>a</sup>	0.9998	0.098 ± 0.004 <sup>e</sup>	0.93
Z6	0.88 ± 0.03 <sup>e</sup>	0.479 ± 0.013 <sup>c</sup>	0.9996	0.114 ± 0.003 <sup>de</sup>	1.00

Different lowercase letters show differences between the samples at P < 0.05.

K: Consistency coefficient,  $n$ : flow behavior index,  $\eta_{50}$ : apparent viscosity at a shear rate of 50 s<sup>-1</sup>,  $\eta_i$ : viscous synergy index.

between 0.9915 and 0.9998. The  $K$  and  $n$  values were in the range of 0.26–2.66 Pa s<sup>n</sup> and 0.324–0.726, respectively. For both of the oil types, the mellorine samples with xanthan and locust bean gums in their formulations had the highest  $K$  values and the samples with alginate had the lowest.

Apparent viscosity at the shear rate of 50 s<sup>-1</sup> ( $\eta_{50}$ ) is another important parameter obtained from steady shear rheological measurements and is considered to be the shear rate in the mouth (Bourne, 2002). Table 2 also shows the  $\eta_{50}$  value of the mellorine samples. The  $\eta_{50}$  value was found in the range of 0.096–0.226 Pa s. Similar to the  $K$  values, the mellorine samples including xanthan gum and locust bean gum had the highest  $\eta_{50}$  values. Viscous synergy is important for the food industry; it means that the interaction between the gums causes the total viscosity of the system to be greater than the sum of the viscosities of each component considered separately. The viscosity index value of the samples was calculated using the equation shown below.

$$\eta_{calc} = \sum_{i=1}^n x_i \eta_i \quad (10)$$

Here,  $x_i$  and  $\eta_i$  are respectively the mole fraction and the viscosity of component  $i$ . In order to ease the comparison of the viscous synergy between gum combinations, viscous synergy index was calculated using the following equation.

$$\eta_i = \eta_{exp} / \eta_{calc} \quad (11)$$

Here,  $\eta_i$  is the viscous synergy index value,  $\eta_{calc}$  is the viscosity in the absence of the interaction, and  $\eta_{exp}$  is the experimental viscosity value of the gum combination. If  $\eta_i$  is higher than unity, then there is synergic interaction between the components.  $\eta_i$  values of the samples are also shown in Table 2. As can be seen, the  $\eta_i$  values of the samples with xanthan and locust bean gums were higher than 1, indicating that there is a synergic interaction between these 2 gums. The  $\eta_i$  value of the samples containing alginate gum and xanthan or locust bean gum in formulation was lower than unity, indicating a lack of synergic interaction between those gums.

In order to describe the variation of  $\eta_{50}$  values with gum concentrations used in the formulation, modified power-law and exponential-type models were used. While the obtained data were fitted to Eqs. (5) and (6), it was seen that the variation of  $\eta_{50}$  values with gum concentrations was better described with a power-law model than exponential-type models. After fitting the data, the following equations were established for the mellorine samples containing soybean and olive oil.

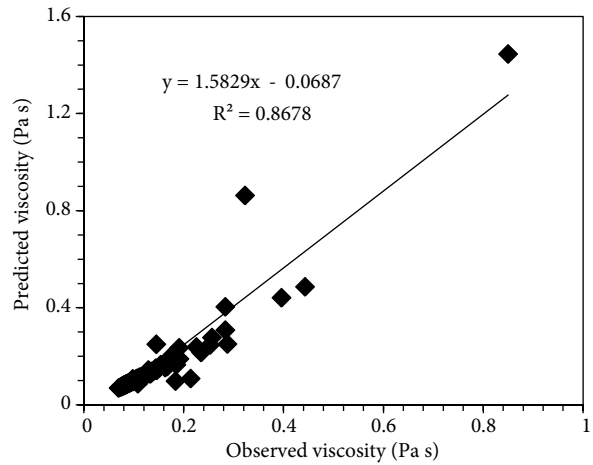
$$\eta_{50} = 0.119 \times C_1^{0.194} + 0.096 \times C_2^{0.109} + 0.102 \times C_3^{0.102} \text{ (soybean oil, } R^2 = 1.000) \quad (12)$$

$$\eta_{50} = 0.136 \times C_1^{0.197} + 0.117 \times C_2^{0.189} + 0.099 \times C_3^{36.12} \quad (\text{olive oil, } R^2 = 0.9972) \quad (13)$$

Here,  $C_1$ ,  $C_2$ , and  $C_3$  are the concentration fractions of xanthan, locust bean, and sodium alginate gums, respectively. The  $R^2$  values of the exponential model were found as 0.75 and 0.54 for the mellorine samples prepared with soybean and olive oil, respectively. The results indicate that the exponential-type model cannot adequately describe the variation of the  $\eta_{50}$  values based on the gum concentration found in the formulation, since  $R^2$  values higher than 0.75 show that established models may be used for prediction of the parameters (Henika, 1982).

**3.3. Estimation of ANFIS and ANN nonlinear models for the apparent viscosity ( $\eta_{50}$ ) based on the oil type, gum concentrations, and shear rate**

After obtaining shear rate versus apparent viscosity values of the mellorine mix samples, ANN and ANFIS nonlinear models were established to predict  $\eta$  values by using oil type, gum concentrations, and shear rate. RMSE, MAE, and  $R^2$  values were used for the comparison of the models. The  $R^2$ , RMSE, and MAE values of the testing part of the ANN model were found to be 0.584, 0.0681, and 0.0483, respectively. The ANN cannot satisfactorily predict  $\eta$  based on the determined inputs. In order to establish an optimal ANFIS model, different membership function types of input, membership function types of output, and numbers of membership function values were used. The  $R^2$ , RMSE, and MAE values of the established models are presented in Table 3. As seen from Table 3, The  $R^2$  value of the ANFIS model, including gaussmf input function type, constant output function type, and 3 membership function



**Figure 2.** Observed versus predicted data obtained from ANFIS modeling.

number, was 0.9121. This was higher than those of the other established models. The predicted and experimental  $\eta$  values are compared in Figure 2 in the form of a scatter plot. The lines between the predicted and observed values of the models are very close to the exact-fit line (45°), indicating the model’s accuracy.

**3.4. Estimation of combined design for the apparent viscosity ( $\eta_{50}$ ) based on the oil type, gum concentrations, and shear rate**

In order to determine the effect of mixture parameters (gum oil types) and process parameters (shear rates) on the apparent viscosity value of the mix samples, a combined design model was performed. The ANOVA table of the developed model is shown in Table 4. The model F-value of 283.90 implied that the model was

**Table 3.** Performance of different ANFIS models for prediction of apparent viscosity of the mellorine mix.

MFTI	MFTO	NMFs	$R^2$		RMSE		MAE	
			Training	Testing	Training	Testing	Training	Testing
Trimf	Constant	3	0.7740	0.9035	0.0157	0.0143	0.0620	0.0476
Trimf	Linear	3	0.7740	0.9035	0.0157	0.0143	0.0620	0.0476
Trimf	Constant	4	0.8434	0.8722	0.0134	0.0147	0.0481	0.0405
Trimf	Linear	4	0.9248	0.8954	0.0095	0.0107	0.0364	0.0269
Gaussmf	Constant	3	0.7485	0.9121	0.0165	0.0143	0.0596	0.0432
Gaussmf	Linear	3	0.9301	0.8732	0.0091	0.0102	0.0346	0.0293
Gaussmf	Constant	4	0.8198	0.9052	0.0142	0.0146	0.0467	0.0352
Gaussmf	Linear	4	0.9482	0.8553	0.0079	0.0101	0.0243	0.0263

MFTI: Membership function type of input, MFTO: membership function type of output, NMFs: number of membership function, RMSE: root mean square error, MAE: mean absolute error.

**Table 4.** ANOVA results of the combined reduced quadratic  $\times$  cubic model.

Source	Sum of squares	df	Mean square	F-value	P-value Prob > F	
Model	36.197518	20	1.80987592	283.89705	<0.0001	Significant
Linear mixture	4.7092603	2	2.35463014	369.34717	<0.0001	
AB	1.6164798	1	1.61647984	253.56095	<0.0001	
AD	0.4616137	1	0.4616137	72.408704	<0.0001	
AE	0.0352074	1	0.03520743	5.5226354	0.0219	
BD	0.8794282	1	0.87942815	137.94706	<0.0001	
CD	0.1678125	1	0.16781248	26.323057	<0.0001	
AD <sup>2</sup>	0.6617278	1	0.6617278	103.79859	< 0.0001	
BD <sup>2</sup>	0.0802942	1	0.08029418	12.594941	0.0007	
CD <sup>2</sup>	0.1957295	1	0.19572947	30.702115	<0.0001	
ABDE	0.517808	1	0.517808	81.223338	<0.0001	
ACDE	0.1933569	1	0.19335693	30.329958	<0.0001	
ABD <sup>2</sup>	0.5350481	1	0.53504813	83.927624	<0.0001	
ACD <sup>2</sup>	0.2398186	1	0.23981863	37.617938	<0.0001	
AD <sup>3</sup>	0.1061967	1	0.10619665	16.658002	0.0001	
CD <sup>3</sup>	0.0261785	1	0.02617848	4.1063558	0.0470	
ABD <sup>2</sup> E	0.7012549	1	0.70125491	109.99881	<0.0001	
ACD <sup>2</sup> E	0.379853	1	0.379853	59.583724	<0.0001	
ABD <sup>3</sup>	1.3638974	1	1.36389744	213.94089	<0.0001	
ACD <sup>3</sup>	0.2068669	1	0.20686689	32.44913	<0.0001	
Residual	0.4016321	63	0.00637511			
Cor. total	36.599151	83				
Std. dev.	0.079844308	R <sup>2</sup>	0.989026189			
Mean	-1.971935624	Adj R <sup>2</sup>	0.98554244			
CV %	4.049032171	Pred R <sup>2</sup>	0.834472197			
PRESS	6.058176999	Adeq Precision	101.7633342			

A: Xanthan gum, B: locust bean gum, C: alginate.

significant. The significant model terms are shown in Table 4. The Pred  $R^2$  of 0.8345 is in reasonable agreement with the Adj  $R^2$  of 0.9855. "Adeq Precision" measures the signal-to-noise ratio. A ratio of greater than 4 is desirable. Our ratio was 101.763, indicating that the signal was adequate. This model can be used to navigate the design space. A model was established for each oil type, since oil type was a categorical factor. Table 5 shows the predicted models. When the obtained model was used, optimization was carried out according to the maximum viscosity value at a shear rate of 50.5  $s^{-1}$ . The best predictions obtained from the combined design are summarized in Table 6. In addition, Figure 3 shows the highest viscosity value at a shear rate of 50.5  $s^{-1}$ . As can be seen from Table 6 and Figure 3, the highest viscosity value was obtained in a gum

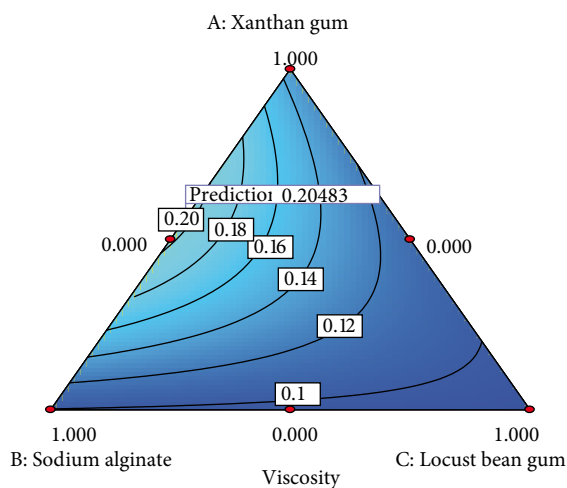
combination including 56.3% xanthan gum and 43.7% locust bean gum. A similar result was reported by Higiro et al. (2007).

Moreover, the dependence of the viscosity value based on the factors can be observed by models obtained from the combined design by means of graphs. Figure 4 is the trace (or Piepel) graph, in which the effects of the gum combination on the viscosity value at a shear rate of 50  $s^{-1}$  can be observed. Gum C (alginate) was not found in the formulation and, while it was added, viscosity exponentially decreased. Figure 5 shows the 3D plots for viscosity. Corner points of the triangle in Figure 3 show the maximum concentrations of each gum, and the base of the triangle shows the minimum concentrations. As seen from Figure 5, the highest viscosity was obtained at the

**Table 5.** Predicted models for the experimental data.

Oil type	Established equation
Soybean	$\ln(\text{viscosity}) = -0.59 \times A - 1.76 \times B - 1.56 \times C + 4.67 \times AB + 4.41 \times AC - 0.06 \times AD - 0.01 \times BD - 0.03 \times CD - 0.18 \times ABD - 0.18 \times ACD + 0.0006 \times AD^2 + 7.14 \times 10^{-5} \times BD^2 + 0.0003 \times CD^2 + 0.004 \times ABD^2 + 0.002 \times ACD^2 - 2.82 \times 10^{-6} \times AD^3 - 1.37 \times 10^{-6} \times CD^3 - 2.27 \times 10^{-5} \times ABD^3 - 8.98 \times 10^{-6} \times ACD^3$
Olive oil	$\ln(\text{viscosity}) = -0.50 \times A - 1.76 \times B - 1.56 \times C + 10.71 \times AB + 0.28 \times AC - 0.06 \times AD - 0.01 \times BD - 0.03 \times CD - 0.37 \times ABD - 0.05 \times ACD + 0.0006 \times AD^2 + 7.14 \times 10^{-5} \times BD^2 + 0.0003 \times CD^2 + 0.005 \times ABD^2 + 0.001 \times ACD^2 - 2.82 \times 10^{-6} \times AD^3 - 1.37 \times 10^{-6} \times CD^3 - 2.27 \times 10^{-5} \times ABD^3 - 8.98 \times 10^{-6} \times ACD^3$

A: Xanthan gum concentration, B: locust bean gum concentration, C: alginate concentration, D: shear rate.

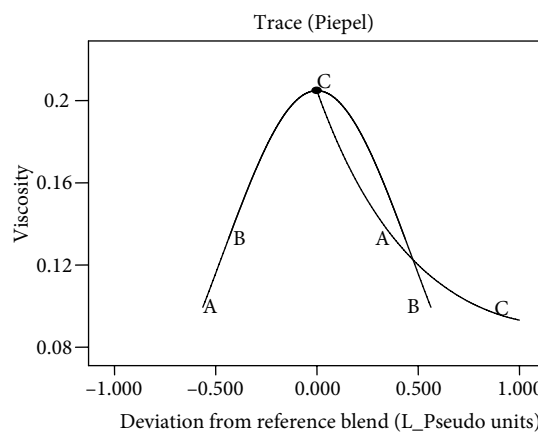


**Figure 3.** Contour diagram for viscosity.

point where the concentrations of A, B, and C gums were approximately 50%, 50%, and 0%, respectively.

**3.5. Sensory scores, overrun index, and melting properties of the mellorine samples**

The overrun values of the mellorine samples are shown in Table 7. Overrun is a measure of increase in volume, which is associated with the structure of ice cream, including melting and hardness (Sofjan and Hartel, 2004). As can be seen from Table 7, the overrun value of the samples varied between 9.66% and 45.91%. Gum type significantly affected the overrun value of the mellorine samples. The samples including xanthan gum and locust bean gum in their formulations had the highest overrun value, which might have resulted from the higher water absorption capacity of this gum combination when compared to other gum formulations. Melting rate concentrations of the mellorine samples at different times (45 and 60 min) and melting times of the samples are also shown in Table 7.



**Figure 4.** Trace (Piepel) plots of the effect of gum concentration on viscosity. A: Xanthan gum, B: locust bean gum, C: alginate gum.

General acceptability, consistency, color, appearance, taste, and odor properties of the mellorine samples were evaluated by panelists. Sensory scores of the mellorine samples are shown in Figure 6. The control sample was mostly favored in terms of general acceptability, color, and appearance properties. While the consistency of the Z4, S4, and Z1 samples was mostly preferred, the S3 sample had the highest taste and odor score as a result of sensory analyses.

**4. Discussion**

In all the samples analyzed in the present study, no significant differences were found between the moisture content of the samples ( $P > 0.05$ ), which could be expected since the moisture content of the gums used in the present study were very similar. The formulation, ingredients and their moisture contents, concentration of the ingredients in the formulation, and processing conditions (temperature

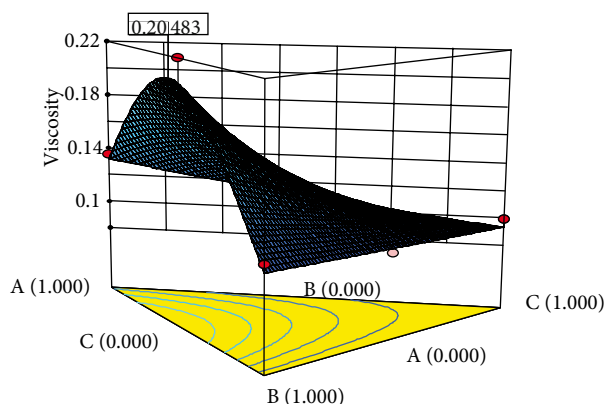


**Table 6.** Predicted gum concentrations for the mellorine samples including different oils to maximize apparent viscosity at a shear rate of 50.50 s<sup>-1</sup>.

Number	Xanthan gum	Locust bean gum	Shear rate (s <sup>-1</sup> )	Oil type	Viscosity (Pa s)
1	0.563	0.437	50.50	Olive oil	0.2048
2	0.500	0.500	50.50	Olive oil	0.20298
3	0.544	0.456	50.50	Soybean	0.19493
4	0.500	0.500	50.50	Soybean	0.19409

**Table 7.** Overrun (%), melting rate (%), and melting time (min) values of the mellorine samples.

Sample	Overrun (%)	Melting rate (%)		Melting time (min)
		45 min	60 min	
K	29.36 ± 0.52	64.00 ± 2.00	91.00 ± 2.00	65.00 ± 1.00
S1	34.30 ± 1.08	60.67 ± 0.58	84.33 ± 1.53	68.33 ± 1.53
S2	21.95 ± 0.29	71.67 ± 0.58	100.00 ± 0.12	56.33 ± 1.53
S3	15.01 ± 0.15	73.67 ± 0.58	100.00 ± 0.23	55.33 ± 0.58
S4	41.98 ± 0.36	58.33 ± 1.53	81.00 ± 1.00	72.33 ± 0.58
S5	10.16 ± 0.76	70.33 ± 0.58	100.00 ± 0.17	53.33 ± 0.58
S6	26.72 ± 0.42	67.33 ± 1.16	100.00 ± 0.26	61.00 ± 1.00
Z1	29.36 ± 1.02	72.00 ± 1.00	96.67 ± 1.53	62.33 ± 1.53
Z2	9.66 ± 0.65	68.67 ± 1.53	100.00 ± 1.19	54.00 ± 1.00
Z3	14.79 ± 0.72	64.67 ± 0.58	100.00 ± 0.94	53.33 ± 0.58
Z4	45.91 ± 1.63	56.00 ± 1.00	81.67 ± 1.56	71.33 ± 1.53
Z5	11.06 ± 0.24	76.33 ± 1.53	100.00 ± 0.85	53.67 ± 1.53
Z6	16.24 ± 1.32	71.67 ± 0.58	100.00 ± 1.25	56.00 ± 1.00



**Figure 5.** 3D surface plot for viscosity value of the apparent viscosity. A: Xanthan gum, B: locust bean gum, C: alginate.

and time duration during heating) might be the main factors affecting the moisture content of the samples.

The magnitude of pH values was slightly influenced by the gum type, which might have resulted from the chemical structure of the gums (Dogan et al., 2011). The reason why the pH value of the mellorine mixes with olive oil was lower than that of the sample with soybean oil might be the free fatty acid contents present in the oil types. There was no positive correlation between the pH and titratable acidity values of the samples, which is consistent with the study of Erkaya et al. (2012).

As can be seen from Figure 1, increase in shear stress was lower than that of the shear rate, indicating that apparent viscosity values of the mellorine samples decreased with increase in shear rate, which demonstrates the shear thinning behavior of the mellorine samples

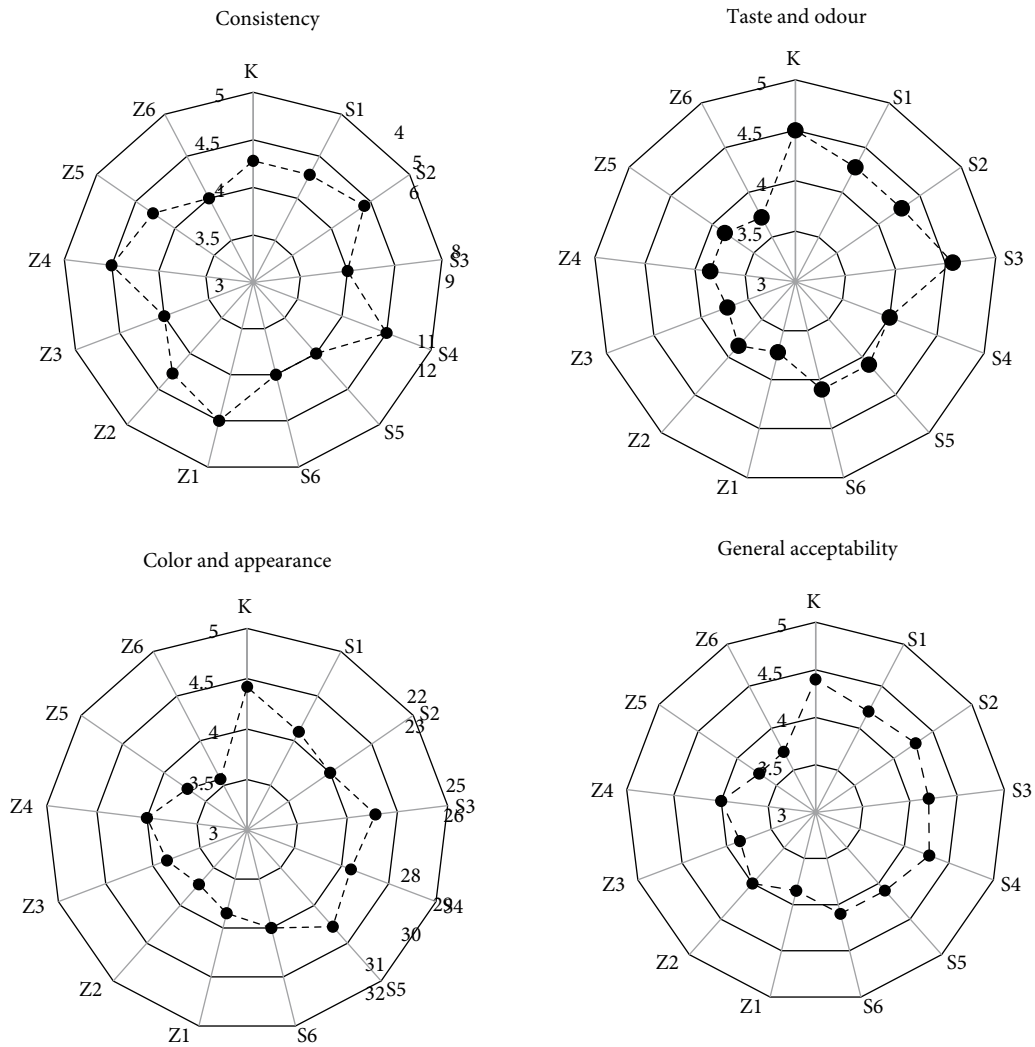


Figure 6. Sensory scores of the mellorine samples.

(Steffe, 1996; Rao, 1999; Sikora et al., 2007). An  $n$  value lower than unity also indicates shear thinning behavior in the samples. Shear thinning behavior results from the fact that hydrodynamic forces generated during shear break structural units in solutions (Bahnassey and Breene, 1994). In addition, partially broken-down micellar casein at the droplet surface in the ice cream mix might result in this behavior (Dickinson and Stainsby, 1984). The shear thinning behavior of ice cream mixes was reported in previous studies (Dogan and Kayacier, 2007; Dogan et al., 2013b; Toker et al., 2013c). There was a negative correlation between the  $K$  and  $n$  values ( $R = -0.901$ ), which is not compatible with previous studies (Toker et al., 2012a, 2012b; Arıcı et al., 2013; Dogan et al., 2013b; Goksel et al., 2013). As can be seen from Table 2,  $K$  values of the mellorine mix samples including olive oil were generally higher than those of the mellorine mix samples including soybean oil, which might have resulted from the higher

viscosity value of olive oil than soybean oil (Yalcin et al., 2012a, 2012b). After determination of flow behavior and  $\eta_{50}$  values of the mellorine mix samples, viscous synergy index ( $\eta_i$ ) values of the gum combinations were determined. The viscosity of the system caused by interaction between the gums is lower than the sum of each of the viscosities of each component considered separately, which is called viscous antagonism (Sarkar et al., 2012). In order to determine viscous synergy or antagonism, viscosity in the absence of the interaction,  $\eta_{calc}$ , was calculated using Eq. (10) (Sarkar et al., 2012). According to the results, the  $\eta_i$  value of the xanthan and locust bean gum combination was higher than 1.0, indicating synergy interaction between these 2 gums. Dogan et al. (2013c) also studied gum optimization for prebiotic hot chocolate beverages and their results were comparable with this study; this was expected, since both of the products were milk-based and this value was the lowest for the sample including alginate,

which is agreement with the previous studies (Dogan et al., 2013a, 2013c). Synergic interaction between xanthan and locust bean gum was also proven by combined design. Tako et al. (1984) reported the interaction of xanthan gum with galactomannans to form high viscosity at low total-polysaccharide concentrations. This interaction is more pronounced in locust bean gum than in the other galactomannans (Dea et al., 1977). These results support our findings. The mechanisms of the interaction between xanthan and locust bean gum have been investigated (Tako et al., 1984; Cairns, 1987; Wang et al., 2002). The side chains of xanthan and the locust bean gum backbone interacted with each other as in the lock-and-key model, in which 1 xanthan chain could associate with 1, 2, or more locust bean gum molecules (Tako et al., 1984). Similar to the present study, the steady rheological parameters or apparent viscosity values were satisfactorily predicted by the ANFIS model in previous studies (Karaman and Kayacier, 2012; Toker et al., 2012b, 2013b).

As can be seen from Table 7, there was a negative correlation between the overrun value and melting concentration or time of the mellorine samples. Similar to the overrun values, melting time values were significantly affected by the gum type used in the formulation of the mellorine. The variation of the melting rate and overrun value based on stabilizer and emulsifier type was reported in different studies (Güven et al., 2003; Keçeli and Konar, 2003; Moeenfarid, 2008). The increase in water absorption capacity of the gums increased the resistance of mellorine to melting.

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