


## Original article

**Using spray-dried sugar beet molasses in ice cream as a novel bulking agent**Betül Gizem Acan,<sup>1</sup> Omer Said Toker,<sup>1</sup> Tugba Aktar,<sup>2</sup>  Faruk Tamturk,<sup>1,3</sup> Ibrahim Palabiyik<sup>4</sup> & Nevzat Konar<sup>5\*</sup> <sup>1</sup> Chemical Metallurgical Engineering Faculty, Food Engineering Department, Yıldız Technical University, Istanbul 34210, Turkey<sup>2</sup> Faculty of Engineering, Food Engineering Department, Alanya Alaaddin Keykubat University, Antalya 07450, Turkey<sup>3</sup> Döhler Food and Beverage Ingredients R&D Center, Karaman 70100, Turkey<sup>4</sup> Agricultural Faculty, Food Engineering Department, Namik Kemal University, Tekirdağ 59030, Turkey<sup>5</sup> Faculty of Engineering, Food Engineering Department, Siirt University, Siirt 56100, Turkey*(Received 10 June 2019; Accepted in revised form 23 September 2019)*

**Summary** In this study, it is aimed to investigate and evaluate the use of molasses which is a by-product of sugar production as a novel bulking agent in ice cream as a sugar replacer. Sugar beet molasses (75%) and 12 DE maltodextrin (25%) were converted into powder form by spray drying. Spray-dried sugar beet molasses (SDSM) was then used as a substitute of sugar in different ratios (0:100, 25:75, 50:50, 75:25 and 100:0) in the ice cream products. The increased amount of SDSM decreased the overrun,  $L^*$ , whiteness index (WI) and melting behaviours and increased  $a^*$ ,  $b^*$ , total phenolic content, consistency and viscosity. Meanwhile, thermal properties have not been affected by the use of SDSM ( $P < 0.05$ ). The sensory findings have been particularly interesting especially when replacing above 50% sugar with SDSM, aroma, flavour and general acceptability decrease. According to the results of this study, substituting 25% of total sugar with SDSM as a bulking agent can decrease cost of the product and improve total phenolic content and some quality parameters without compromising sensorial properties.

**Keywords** Ice cream, maltodextrin, molasses, sugar.

**Introduction**

Approximately one-third of the foods consumed in the world become waste which causes many adverse consequences on world economy and ecosystem as well as social problems (Wadhwa, & Bakshi, 2013; Martin-Rios *et al.*, 2018). Therefore, the researchers shifted their focus on waste management, more specifically food waste management in order to decrease the acceleration of the waste (Corrado, & Sala, 2018). Food waste issue was addressed in the United Nations Sustainable Development Goals, where the UN suggested an objective 'to reduce the food waste to halve per capita the global food waste at the retail and consumer level, and reduce food losses along production and supply chains by 2030' (United Nations, 2015). In order to achieve the objective of the UN, it is necessary to increase the rates of recycling, and reuse and prevention as well as attracting the attention of the

users on the importance of food waste and waste management.

Molasses is a by-product of crystal sugar manufacturing which can be defined as unrefined natural sweetener made up of either sugar cane or sugar beet. It naturally contains high amount of fermentable sugars (sucrose, glucose, fructose and raffinose) as well as some organic nonsugar compounds such as betaine, lactic acid, amino acids, minerals, vitamins, phenolic compounds and dark colour compounds such as melanin. Those compounds are naturally presented in sugar cane and sugar beet and can be obtained during the processing of the sugar thanks to the high temperature and pH levels as well as the interactions of organic nonsugar compounds (Djordjević *et al.*, 2018). Sugar beet molasses was specifically included in this study due to its high nutritional value and low calorie content and due to being a by-product. In the literature, molasses was added to some food formulations as biscuits and cookies (Šimurina *et al.*, 2008; Filipčev *et al.*, 2012; Filipčev *et al.*, 2014), bread (Filipčev *et al.*, 2010) and gluten-free baked foods (Filipčev *et al.*, 2015, 2016;

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Šimurina *et al.*, 2017). Despite the product trials, molasses was used in the literature previously as a honey replacer (Filipčev *et al.*, 2014), flavour and nutritional value improver (Šimurina *et al.*, 2008) and a sweetener (Šimurina *et al.*, 2017). In our knowledge, there is not any study in which sugar beet or cane molasses is dried by spray drying for using as a food ingredient, for example as a bulking agent.

Today, consumer expectations and trends have changed significantly. Sugar-free, saturated fat-reduced and low-calorie foodstuffs are increasingly produced and preferred. However, these innovative products are also expected to exhibit similar quality characteristics to conventional ones (Konar *et al.*, 2016). In the present study, ice cream was selected as the modal food due to its high acceptability and wide consumer range all around the world. Ice cream as a product has a colloidal structure that contains air bubbles, fat globules, ice crystals, unfrozen serum phase, sweeteners, stabilisers, casein micelles and proteins which may affect the component change quality for molasses and sugar replacement procedure (Toker *et al.*, 2013). The primary quality indicator for a colloid structure like ice cream is mainly the texture which changes with various factors including viscosity of the aqueous phase, state of aggregation of the fat globules, overrun value, air cell size and aggregation size of the ice crystals (Kus *et al.*, 2005). These expectations and demands should be taken into consideration in ice cream reformulation studies. Textural and sensory properties, melting and flow behaviour, and aroma release can be mentioned as the main ice cream quality parameters (Abdel-Haleem, & Awad, 2015). Ice cream quality characteristics are strongly affected by ingredients (Adapa *et al.*, 2000; Soukoulis *et al.*, 2010a, 2010b). The structure and textural properties of ice cream are influenced by the size and distribution of air cells and ice crystals, and the use of oil and stabiliser components (Baer *et al.*, 1999). In the case of low-calorie product development studies, quality characteristics will be significantly affected by the type and level of substitute used. For example, Laura Rolon *et al.* (2017) found that maltodextrin usage rate and reduction in fat level resulted in a decrease in consumer acceptance of ice cream. Hyvönen *et al.* (2003) reported that the use of this component increases the rate of aroma release and melting in the fat-free ice cream samples prepared by the use of maltodextrin at the level of 2.1%, and found that the perceived 'fattiness' and 'creaminess' properties become apparent. In addition, sugar substitutes have a significant effect on the sensory acceptance of fat level in ice cream (Laura Rolon *et al.*, 2017). This is because the increase in the fat level can lead to a decrease in the volume of ice crystals. The increase in ice crystals may result in a harder texture (Akbari *et al.*, 2016). Therefore, the

choice of sugar substitution should take into account changes in the perception of fat level and accordingly quality characteristics.

In the present study, it was aimed to valorise food waste in a worldwide popular food product in order to evaluate a waste as well as replacing the valuable and health-threatening food component (sugar) with healthier and cost-effective options. Objectives of this study can be summarised as (i) replacing relatively unhealthy component (sugar) with a healthier one (molasses), (ii) reusing the food waste (molasses) in ice cream formulation and (iii) decreasing the sugar intake. Meanwhile, by using molasses the authors will investigate the possibility of its usage as a sugar substitute. Overrun value, melting profile, thermal properties, rheological properties, colour and sensory properties of ice creams were investigated when spray-dried sugar beet molasses was used instead of sugar in different ratios (0:100, 25:75, 50:50, 75:25 and 100:0).

## Materials and methods

### Materials

In this study, novel ice cream formulation by using sugar beet molasses was aimed in order to see its potential use as a sugar replacer. In the formulation, sugar beet molasses (Pak Maya, Kocaeli, Turkey), whole milk (purchased from a local market, Istanbul, Turkey), salep (purchased from a local market, Istanbul, Turkey), 12 DE maltodextrin (Dohler Food, Karaman, Turkey) and vanilla (purchased from a local market, Istanbul, Turkey) were used. Salep which is a traditional milk and orchid root drink was used as a stabiliser, where vanilla was used as a flavour delivering agent. Salep composition was previously determined as 56.1% glucomannan, 36.31% starch, 4.60% protein and 2.07% ash (Kurt & Kahyaoglu, 2015). Meanwhile, the protein, fat, dry matter and nonfat dry matter content of the milk was 3.0%, 3.3%, 11.0% and 7.7%, respectively.

Molasses made up of sugar beet was used in the powdered form in the ice cream formulation for this study. Sugar beet molasses was powderised by spray drying technique. The maltodextrin and molasses ratio was set as 25:75. The mixture was initially homogenised at 711 g for 10 min at room temperature (Ika, ULTRA-TUR-RAX T25), which was followed by drying at the set parameters: 1.0 µm nozzle diameter, 90 °C outlet temperature, 8 mL min<sup>-1</sup> feeding rate, 600 L h<sup>-1</sup> air flow rate and 170 °C inlet temperature of the spray drier. Those parameters were selected according to the pretests. Obtained SDSM was stored at 20 ± 2 °C in a container that blocks the air and sunlight. Final SDSM samples were standardised to have 6.52 ± 0.15% dry matter and 91.84 ± 0.32% ash content.

## Methods

### *Ice cream formulation and sample preparation*

Ice cream formulation and sample preparation method were adapted from Kurt *et al.* (2016) and Soukoulis & Tzia (2018) with some modifications by using an ice cream maker machine (Delonghi, II Gelataio, ICK 5000, China). The formulation consisted of sugar (22%), milk (77.1%), salep (0.43%) and vanilla (0.2%). Various amounts of sugar beet molasses were added to that mixture to achieve five different concentrations. Total sugar: spray-dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively. The ingredients were all added to heated milk (85 °C) stirred for 1 min. This step was followed by mixing at 12 g for 3 min followed by rapid cooling to 4 °C. The samples were aged for 24 h at 4 °C. The aged ice cream mixes were whipped at 0 °C for 10 min (Delonghi, II Gelataio, ICK 5000, China, mixing speed approximately 225 r.p.m.) and were frozen and hardened using a batch freezer (Arcelik, Gebze, Turkey) at −18 °C for 24 h. Samples were stored in a deep freezer (Arcelik) at freezing conditions of −18 °C prior to analysis.

$$\text{Overrun\%} = \frac{\text{weight of a unit volume of the ice cream mix} - \text{the weight of a unit volume of the ice cream}}{\text{weight of a unit volume of the ice cream}} \quad (2)$$

### *Sugar profile determination*

Sugar profile of spray-dried sugar beet molasses was determined by the method previously applied by Karaman *et al.* (2014). An HPLC system (Shimadzu, Kyoto, Japan) equipped with manual injection pump, refractive index detectors and ZORBAX carbohydrate column (4.6 × 250 mm, 5-µm particle size), which was thermostated at 25 °C, was used for the determination of sugar composition of the ice cream samples.

### *Rheological properties of ice cream samples*

Rheological properties of the samples that contain SDSM were determined by the method applied previously by Dertli *et al.* (2016). The rheometer was strain/stress-controlled rheometer (Anton-Paar, MRC 302, Graz, Austria) and was equipped with a Peltier temperature controller, and steady shear rheological characteristics of the samples were tested. The testing probe was parallel-plate configuration (plate diameter 35 mm, angle 4°, gap size 0.5 mm) within the shear rate range of 0.1–100 s<sup>−1</sup> at a constant measurement temperature (5 °C). 1 mL of the ice cream mix sample was placed between the plates, and the measurement was started immediately. A total of 30 data points were recorded at 10-s intervals during the shearing. Each measurement was repeated three times with two

replications (each 1 mL). The shear stress values vs. shear rate were plotted by increasing the shear rate. Obtained data were fitted to Ostwald de Waele model using a software (Toolmaster™, Graz, Austria), and consistency coefficient (*K*) and flow behaviour index (*n*) values were calculated according to the following model (1) used to describe shear-induced behaviour of the ice cream samples.

$$\delta = K \cdot \gamma^n, \quad (1)$$

where  $\delta$  is the shear stress (Pa),  $\gamma$  is the shear rate (s<sup>−1</sup>), *K* is the consistency coefficient (Pa·s<sup>*n*</sup>), and *n* is the flow behaviour index (dimensionless).

### *Overrun value determination of the ice cream samples*

Overrun value of the samples was measured according to the proportion of ice cream volume to the volume of mix according to the method applied previously by Soukoulis *et al.* (2010a). The overrun value was calculated according to the following formula (2) which identifies the decrease in weight of a given volume of mix due to the addition of air:

### *Physicochemical properties*

Dry matter and ash content of ice cream samples and sugar beet molasses were estimated by gravimetric method at 105 and 600 °C, respectively, according to AOAC (2000). Meanwhile, colorimetric properties of the samples were measured using a Minolta Chromameter (CR-400, Minolta Camera Co., Osaka, Japan) with *L*\*value (lightness), *a*\* value (redness/greenness) and *b*\* value (yellowness/blueness). For each sample, the whiteness index (WI) was calculated based on the following equation (3):

$$\text{WI} = 100 - \sqrt{(100 - L^*)^2 + (a^*)^2 + (b^*)^2} \quad (3)$$

Total phenolic content of the ice cream samples was measured according to the method applied by Singleton & Rossi (1965). Extraction of the phenolic content was carried out by methanol:water (80:20 V/V). Specifically, 5 g of ice cream sample was extracted with 45 mL of solvent right before the 3 h of incubation with continuous shaking. Samples were then centrifuged (Hettich, Universal 320R, Tuttlingen, Germany) at 10 303 g for 15 min and filtered through the 0.45-µm filter. The extracts were mixed with 0.2 N Folin–Ciocalteu phenol (1:5) with the addition of 7.5% Na<sub>2</sub>CO<sub>3</sub> [ratio of extract:7.5% Na<sub>2</sub>CO<sub>3</sub> is (1:4)].

The mixture was stored at the dark condition at room temperature ( $20 \pm 2$  °C) for 30 min. Absorbance of those samples was measured for the calculation of total phenolic content at 760 nm using a UV-Vis spectrophotometer (Shimadzu, UV-1800) against the gallic acid standard.

#### Melting profile determination

Melting profile of the ice cream samples was determined according to the methods used by Góral *et al.* (2018) and Kurt & Atalar (2018). For this purpose, 25 g of ice cream samples was left to melt at  $20 \pm 2$  °C. The dripping time (time of first drop from ice cream), complete melting time and melting profile were observed every 15 min until complete liquefaction.

#### Thermal properties

Thermal properties of the samples were measured by differential scanning calorimeter (DSC Q20, TA Instruments, Newcastle, WA, UK). Specifically, heat of ice melting  $\Delta H$  ( $\text{J g}^{-1}$ ), temperature offset, onset and mid-point (°C) were assessed at the closed-off aluminium pans of the DSC after the equilibration at  $-40$  °C and the measurement were done while the heating process continues between  $-40$  and  $10$  °C at  $5$  °C  $\text{min}^{-1}$ .

#### Hedonic evaluation of sensory properties

Hedonic evaluation of ice cream samples was determined based on protocols described previously by Soukoulis *et al.* (2010b) with some modifications. Basically, 100 mL ice cream samples were presented and served at certain intervals in odourless, randomly three-digit-coded glass beakers of 100 mL in a covered glass dish. Sensory evaluation was performed in a room with an appropriate temperature ( $25$  °C) and lighting in an open sitting condition. The panel ( $n = 46$ ) was composed of staff and graduate students of Food Engineering Department at Yıldız Technical University. The age composition of the panellists was 63% (20–25) and 37% (35–52), whereas the gender composition was 24% male and 76% female. Each panellist was trained before evaluation in order to familiarise with the sensory analysis, samples and methodology. They were asked to cleanse their palates with water between each sample and without break. Degree of liking of samples was evaluated for appearance, texture, flavour, aroma, melting resistance and general acceptance in a scale ranging from 0 to 10 points where 0 reflected unacceptable (not like at all), 5 acceptable (like moderately) and 10 excellent (like extremely). Panellists evaluated all samples in nine sessions consecutively in four days. The manner in which the treatment combinations were divided between the sessions and the order in which the samples were presented were randomised to minimise the carryover effects.

#### Statistical analysis

JMP 6 software was used to analyse the data that were compared using a Tukey's test ( $P < 0.05$ ) using the analysis of variance (ANOVA).

## Results and discussion

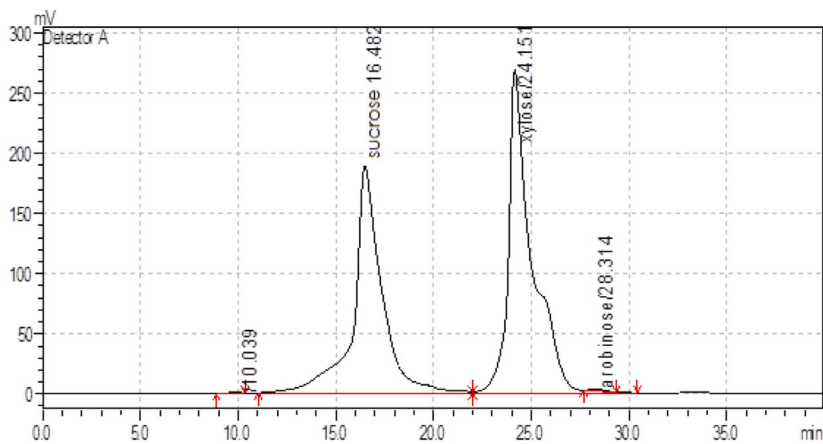
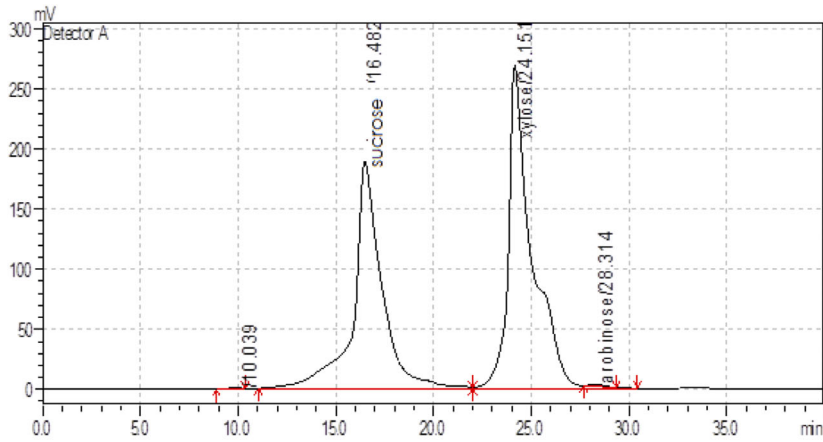
### Sugar profile determination

Sugar profile of spray-dried sugar beet molasses samples is shown in Fig. 1. According to the results in the spray-dried sugar beet molasses, samples consisted of sucrose ( $294.23 \pm 17.90$  ppm), xylose ( $385.90 \pm 26.24$  ppm) and arabinose ( $0.65 \pm 0.12$  ppm). According to results, sucrose contents of ice cream mixes of Ice100, Ice75, Ice50, Ice25 and Ice0 were determined as 0.006 g per 100 g, 5.504 g per 100 g, 11.003 g per 100 g, 16.501 g per 100 g and 22 g per 100 g, respectively.

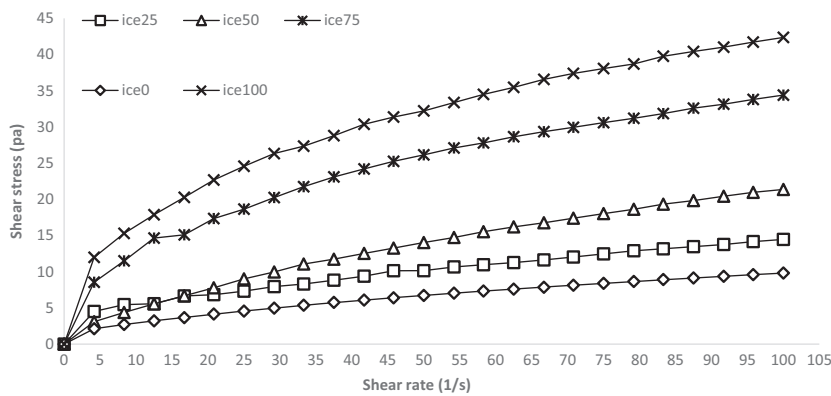
### Rheological properties of ice cream samples

Rheological properties were determined according to the Ostwald de Waele model, where they were assessed for flow behaviour index ( $n$ ) and consistency ( $K$ ) as shown in Fig. 2. For ice cream, it is well reported to be non-Newtonian character, specifically shear-thinning pseudoplastic flow behaviour by the previous studies (Velez Ruiz *et al.*, 1997; Dertli *et al.*, 2016; Mariano & Alamprese, 2017; Rolon *et al.*, 2017; Soukoulis, & Tzia, 2018). On the other hand, addition of different ingredients such as maltodextrin (Laura Rolon *et al.*, 2017) or molasses (Soukoulis, & Tzia, 2018) to the formulation of ice cream did not affect the pseudoplastic flow behaviour. As found in this study, using spray-dried sugar beet molasses coated by maltodextrin in order to replace sucrose did not influence flow behaviour properties. More specifically, in the rheological analysis  $R^2$  value was found to be very close to 1, which indicated the appropriateness of the model selection (Table 1).

Addition of SDSM to the ice cream showed an increase in the viscosity of all sample sets. This increase might be due to the substituting of the sucrose with SDSM which could possibly cause an interaction between protein and polysaccharide and/or protein and oligosaccharides. Intermolecular interactions were found to be the source of limited water molecules' mobility which is caused by the protein-oligosaccharide interactions that result in the thickening and increase in the viscoelasticity (Patmore *et al.*, 2003). Another reason for the viscosity increase, on the other hand, could be due to the water-binding capability of the carbohydrate components. Additionally, sucrose and corn syrup that contains a high amount of fructose which is commonly used as sweeteners in the ice cream process have also high water-binding capacity



**Figure 1** Sugar profile of spray-dried sugar beet molasses. [Colour figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



**Figure 2** Flow behaviour of the ice cream samples. Sugar:Spray dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively.

(Adapa *et al.*, 2000). Noteworthy, sucrose has higher water molecule affinity compared to fructose or glucose (Sato, & Miyawaki, 2016), where molasses has very similar affinity values to sucrose (Soukoulis, & Tzia, 2018). This information is critical since the high potential of rheological influence is significant due to the maltodextrin in the structure of SDSM.

Increase in the water mobility, macro viscosity increase and concentration of the ice cream mixes are the factors that are likely to influence the protective effect of polysaccharides against the formation of ice particles by binding water (Soukoulis, & Fisk, 2016). Previous studies illustrated that viscosity increases with the maltodextrin addition to low-fat ice cream mixes

**Table 1** Rheological properties of ice cream samples

Samples	<i>N</i>	<i>K</i> (Pa·s <sup><i>n</i></sup> )	<i>R</i> <sup>2</sup>
Ice0	0.50 ± 0.01	1.03 ± 1.12	0.997
Ice25	0.44 ± 0.03	1.87 ± 0.30	0.960
Ice50	0.65 ± 0.02	1.09 ± 0.09	0.997
Ice75	0.49 ± 0.10	3.87 ± 1.52	0.990
Ice100	0.47 ± 0.12	5.69 ± 2.96	0.960

Sugar:spray-dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively.

(Aykan *et al.*, 2008; Laura Rolon *et al.*, 2017). Likewise, interactions between the dextran and globular milk proteins were found to have an increasing effect on the viscosity. This interaction is likely to cause loss or at least a decrease on the emulsification capability due to adsorption effect of the water–oil interface as well as due to the hydrophobic character of the protein–polysaccharide complex (Adapa *et al.*, 2000). On the other hand, using the macromolecular sweeteners, such as corn starch hydrolysates and oligosaccharides, on the ice cream recipe increases the consistency constant and viscosity (Soukoulis *et al.*, 2010a). This kind of effect of those sweeteners on the viscosity is found to be highly associated with polymerisation degree, water-binding/holding capacity and their branching properties (Soukoulis *et al.*, 2010a). Thus, depending on the value of maltodextrin DE, possible changes on the DP values of the substances and the use of maltodextrin with different DE values may have the potential to affect the rheological properties with different intensities. For instance, fructooligosaccharide (FOS) addition to ice cream mixture affects the rheological properties by modulating the water-binding capability due to the degree of branching and polymerisation (Soukoulis, & Fisk, 2016).

Depending on the polar and nonpolar properties and other chemical properties of the affinity, the biopolymer should interfere with the water molecules of varying severity and should be expected to affect the flow properties of the ice cream mix. Viscosity and yield value has a negative relation with the overrun value (Velez Ruiz *et al.*, 1997). A similar relationship was observed between the viscosity and overrun values of the products obtained in our study.

According to our findings, *n* (flow behaviour index) and *K* (flow consistency index) values of ice cream were determined to vary between 0.44–0.65 and 1.03–5.69 Pa·s, respectively. Use of SDSM seems to have an increasing effect on the *K* value ( $P < 0.05$ ). This might be due to the effect of the maltodextrin on the rheological properties by contributing to water binding (Udomrati & Gohtani, 2015). Hydrocolloid stabilisers increase the *K* value (Varela *et al.*, 2014). Depending

on dextrose equivalent value, maltodextrins may act as a stabilising agent (Pycia *et al.*, 2017). Furthermore, a significant increase in the consistency value is observed due to the substitution of sugar with molasses (Soukoulis, & Tzia, 2018). In addition, in this study, a hydrocolloid-like behaviour of powder molasses in ice cream mix was found to be remarkable. The flow index, *n* value, in pseudoplastic liquids is an indicator of the departure from Newtonian behaviour. In this context, it was reported that the use of various hydrocolloids in the ice cream mix decreased the flow index and the presence of polysaccharide hydrocolloids enhanced the pseudoplastic properties of ice cream (Varela *et al.*, 2014).

It can be stated that both main components of SDSM have an effect on the change of *K* values of ice cream samples. Therefore, it may be necessary to take into account the DE value of maltodextrin and to use it for the production of powder molasses and ice cream. For instance, previous research done by Soukoulis *et al.* (2010a) mentioned that the use of 12 and 17 DE maltodextrin as a bulk sweetener with different molecular weight, degree of polymerisation, degree of branching, water-holding capacity and sweetening power, including DE maltodextrin, showed a decrease in *K* value of ice cream with the increase in DE value. For the future studies, the investigation of the effects of each component on the flow behaviour with the use of different levels of maltodextrin:molasses will be useful in determining the optimum composition and component composition of powder molasses as a sugar alternative.

### Overrun

Overrun is defined as the per cent of the expansion of ice cream following the air incorporation process during the freezing operation (Baer *et al.*, 1999). According to our results (Table 2), the substitution of sucrose with different levels of molasses powder resulted in a significant decrease in overrun values due to the increase in the level of substitution ( $P < 0.05$ ). This can be mentioned as a disadvantage for the complete substitution of sucrose. However, although the difference in overrun value in the samples between 25% and 50% sucrose-substituted sample and the control sample can be considered as statistically significant, it is in tolerable level. It is suggested that the changes in the ice cream sugar composition affect the properties of air-holding capacity (Soukoulis, & Tzia, 2018), whereas the decrease in the overrun value is predicted to be caused by maltodextrin which is one of the main components of SDSM. The observation was also supported by previous research done by Soukoulis *et al.* (2010a) which determined that maltodextrin, which especially had a low DE value, had a negative effect on the overrun value of ice cream. It has been

determined that the liquefied molasses use with various amounts as a sucrose substitution may affect the aeration of the ice cream mix positively due to the pH buffering effect (Soukoulis, & Tzia, 2018). Considering the results obtained, it can be stated that our study is compatible with previous studies due to the fact that this effect is exacerbated by the usage rate of maltodextrin. Furthermore, maltodextrin with low DE values (<10) and the use of maltodextrin at lower rates (<25) are expected to reduce the effect on the overrun.

Since sugars have a negligible surface activity, they cannot be adsorbed to the water–lipid or air–serum interface, but these binding capacities may affect the stability of the air cells of the relevant components (Soukoulis, & Tzia, 2018). This may lead to differences in the quality characteristics of overrun and freezing, because besides stability, texture, melting speed and sensory properties are also affected by the overrun. Increase in overrun value also results in increased foam stability and inhibition of ice crystals (Dertli *et al.*, 2016).

Texture and hardness of ice cream are affected by a number of factors, including overrun (Varela *et al.*, 2014). High overrun can reduce thermal diffusion which will reduce the melting speed (Akbari *et al.*, 2016). This may also result in an increase in adhesiveness and cohesiveness values and decrease in yielding value for viscosity (Velez Ruiz *et al.*, 1997). Therefore, in studies of reformulation of ice cream with the use of powder molasses, it will be useful to reduce the deviation of the overrun property compared to conventional samples.

The effect of the sample preparation method on the overrun was also investigated in this study. In

particular, freezing with low overrun values can be achieved by the use of batch freezers. This finding was supported by the results of Dertli *et al.* (2016) who found overrun values to be between 22.5% and 33.3% and 47.6% overrun value (Mariano & Alamprese, 2017) for the sucrose-containing samples and batch-type freezer. Based on the control sample (52.2%) for this study, it can be stated that there is no overrun drop from the source of the freezer type and the results are consistent with the previous studies.

### Melting properties

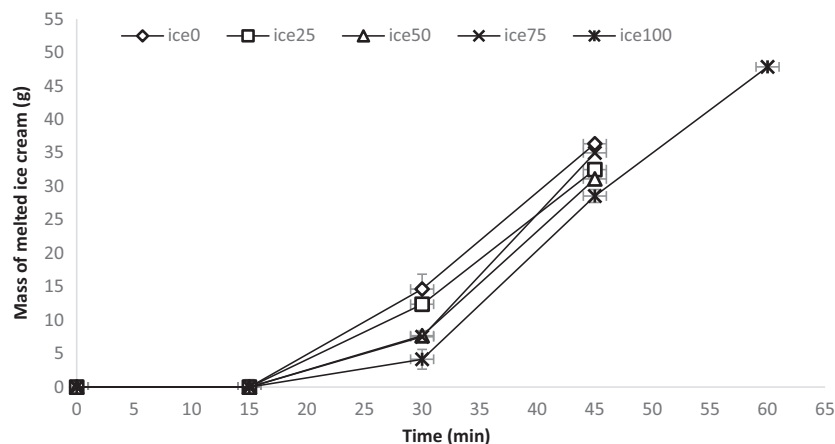
The effects of sucrose substitution with different levels of molasses powder were investigated by examining the rate of melting, first dropping time and the exact melting time of the ice cream samples (Table 2). First dropping time values were found between 1013 and 1217 s in all samples, and no significant difference was observed in comparison with each other (Fig. 3). However, with the increase in sucrose substitution level, it showed an increase in complete melting time ( $P < 0.05$ ). Therefore, it was determined that the melting speed decreased with the use of powder molasses. It should be noted that slow and uniform melting behaviour in ice cream is among the desired properties (Baer *et al.*, 1999). This situation shows that the use of powder molasses has an effect on improving ice cream melting properties.

It has been reported in previous studies that different levels of substitution of sucrose and glucose syrup components with other bulk sweeteners or bulking agents affect their melting behaviour and properties (Ozdemir *et al.*, 2008; Cadena *et al.*, 2012; Mariano &

**Table 2** Overrun, colour and melting properties of ice cream samples

Parameters	Ice0	Ice25	Ice50	Ice75	Ice100
Overrun (%)	52.22 ± 0.71 <sup>a</sup>	44.50 ± 3.18 <sup>b</sup>	46.74 ± 1.57 <sup>ab</sup>	42.30 ± 0.48 <sup>b</sup>	24.53 ± 4.09 <sup>c</sup>
Colour					
<i>L</i> <sup>*</sup>	81.74 ± 3.05 <sup>a</sup>	63.17 ± 0.41 <sup>b</sup>	57.63 ± 0.58 <sup>c</sup>	53.66 ± 0.54 <sup>d</sup>	50.94 ± 1.35 <sup>d</sup>
<i>a</i> <sup>*</sup>	−2.63 ± 1.03 <sup>a</sup>	2.19 ± 0.07 <sup>b</sup>	4.70 ± 0.11 <sup>c</sup>	5.72 ± 0.23 <sup>cd</sup>	6.55 ± 0.41 <sup>d</sup>
<i>b</i> <sup>*</sup>	4.30 ± 0.58 <sup>a</sup>	16.61 ± 0.20 <sup>b</sup>	19.15 ± 0.31 <sup>c</sup>	18.12 ± 0.58 <sup>c</sup>	18.17 ± 0.8 <sup>c</sup>
WI	81.05 ± 3.21 <sup>a</sup>	59.54 ± 0.31 <sup>b</sup>	53.26 ± 0.45 <sup>c</sup>	49.91 ± 0.70 <sup>cd</sup>	47.27 ± 1.48 <sup>d</sup>
First dropping time (s)	1013.55 ± 55.27 <sup>a</sup>	1069.5 ± 48.79 <sup>a</sup>	1208 ± 2.97 <sup>a</sup>	1227 ± 89.10 <sup>a</sup>	1114.05 ± 143.04 <sup>a</sup>
Complete melting time (s)	3213.6 ± 71.50 <sup>a</sup>	3364.8 ± 6.79 <sup>ab</sup>	3435.6 ± 15.30 <sup>b</sup>	3538.5 ± 70.00 <sup>bc</sup>	3699.3 ± 66.29 <sup>c</sup>
Total phenolic content (mg GAE kg <sup>−1</sup> )	68.19 ± 7.96 <sup>a</sup>	349.95 ± 6.50 <sup>b</sup>	494.05 ± 3.82 <sup>c</sup>	537.30 ± 3.82 <sup>c</sup>	1012.97 ± 31.36 <sup>d</sup>
Thermal properties					
<i>T</i> <sub>onset</sub> (°C)	−6.28 ± 0.76 <sup>a</sup>	−6.64 ± 0.64 <sup>a</sup>	−6.29 ± 0.42 <sup>a</sup>	−5.95 ± 0.52 <sup>a</sup>	−6.59 ± 0.21 <sup>a</sup>
<i>T</i> <sub>midpoint</sub> (°C)	0.96 ± 0.99 <sup>a</sup>	0.93 ± 0.25 <sup>a</sup>	0.21 ± 0.59 <sup>a</sup>	1.34 ± 1.76 <sup>a</sup>	0.50 ± 0.21 <sup>a</sup>
<i>T</i> <sub>offset</sub> (°C)	10.66 ± 0.35 <sup>a</sup>	10.66 ± 0.35 <sup>a</sup>	9.77 ± 1.61 <sup>a</sup>	11.74 ± 2.96 <sup>a</sup>	11.31 ± 1.07 <sup>a</sup>
Δ <i>H</i> (J g <sup>−1</sup> )	119.83 ± 12.19 <sup>a</sup>	100.95 ± 1.06 <sup>a</sup>	95.06 ± 2.85 <sup>a</sup>	116.35 ± 15.79 <sup>a</sup>	117.11 ± 27.71 <sup>a</sup>

All experiments were performed in triplicate. All data are reported as the mean ± SD. Different letters within the same rows are significantly different ( $P < 0.05$ ). Sugar:spray-dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively.



**Figure 3** Melting properties of ice cream samples. Sugar:Spray dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively.

Alamprese, 2017). However, the severity of this effect varies according to the used substitution. For example, in ice cream reformulation studies, use of liquid molasses as a partial sucrose substitute showed that ice cream samples with similar melting behaviour compared to the original recipe can be obtained and that melting behaviours do not show significant differences in samples of ice cream containing sucrose and partial molasses (Soukoulis, & Tzia, 2018).

Although maltodextrin from the components of SDSM has a lower concentration than liquid molasses, it may affect the melting properties. Some factors can lead to a decrease in the rate of freezing of the ice cream such as the increase in the amount of frozen water in the ice cream, low solubility of the sweeteners used or relatively slow melting of amorphous solids by the sugars during the freezing process (Mariano & Alamprese, 2017). Melting speed is affected as maltodextrin causes a decrease in the freezing point (Hyvönen *et al.*, 2003). The effect of oil level on melting speed in ice cream is higher than that of sugar. Maltodextrin is a suitable component for low-fat and fat-free ice cream formulation studies. For example, a recent study found that maltodextrin, used for fat substitution, slowed down the rate of melting speed (Laura Rolon *et al.*, 2017). However, in the literature, there are diverse results according to the melting rate reduction based on the sugar alternative additive usage. It has been reported that inulin, which is another volume agent and an oil substitute agent, increases the melting rate due to the aid of concentration increase (Tiwari *et al.*, 2015).

In addition, other factors that have an effect on melting speed should be considered. For example, high overrun value reduces the thermal diffusion capacity, which can lead to a reduction in melting speed (Akbari *et al.*, 2016). Therefore, in our study, the samples with lower overrun values should be able to complete faster melting and should be taken into consideration when

evaluating the effect of powder molasses on melting properties. However, using an alternative filler may provide a reduction in the rate of melting which may have a positive effect on other quality factors. Varela *et al.* (2014) stated that viscosity of the ice cream mix has a strong correlation with the decrease in the rate of melting and the increase in the shape retention.

#### Thermal properties

Thermal properties of ice cream are among the effective parameters on quality. Those thermal properties are well correlated with each other (Soukoulis *et al.*, 2010a). The formation of structural elements during the maturation of ice cream, thermodynamic stability and thermal properties can be affected by different process stages and conditions such as freezing and hardening (Soukoulis, & Fisk, 2016). However, the effect of the composition on thermal properties has greater importance. Thermal behaviour in the ice cream mix is difficult to predict due to the presence of various gum substances and sugars with different water-binding properties (Adapa *et al.*, 2000). For example, the change in the sweetener composition, the levels of monosaccharide and low molecular weight polyols present in the composition are effective on ice crystallisation (Soukoulis, & Tzia, 2018). In this study, some thermal properties of ice cream samples were examined by DSC technique (Table 2).  $T_{\text{onset}}$ ,  $T_{\text{mid-point}}$ ,  $T_{\text{offset}}$  and  $\Delta H$  values were determined between  $(-5.95) - (-6.64)$  °C,  $0.21-1.34$  °C,  $9.77-11.7$  °C and  $95.1-119.8$   $\text{J g}^{-1}$ , respectively.

The freezing point of ice cream should ideally be high enough to provide suitable and small crystal formation. This property is generally dependent on the purity of the solutions, and the decrease in the freezing point is expected with the increase in solute concentration (Adapa *et al.*, 2000; Akbari *et al.*, 2016). The main ingredients which are effective on this property



include sugars, and the effects of components such as milk proteins, flavouring agents, emulsifiers and stabilisers can be expressed as minor (Baer *et al.*, 1999). Achieving low temperatures during ice cream production results in the non-dissolution of the sweetener and reduces the freezing point by reducing the soluble sugar value (Mariano & Alamprese, 2017).

Hyvönen *et al.* (2003) stated that the addition of maltodextrin to ice cream mix caused a drop in melting point. Molasses have the potential to affect the thermal properties of the ice cream mix depending on the presence of complex protein and oligosaccharide composition. In addition, when polysaccharides are used as stabilisers, they can be effective in reducing the size of ice crystals by reducing the diffusion kinetics of water molecules to the nonfrozen phase, and this effect occurs at a certain level depending on the concentration. It should be noted that the ice crystal size is not affected after certain levels (Baer *et al.*, 1999). However, as a result of our study, it was determined that the use of powder molasses at different levels did not have a significant effect on the thermal properties that were examined. In a recent study (Soukoulis, & Tzia, 2018), it was reported that the effects of suppressing the glass transition temperature of sucrose and molasses were similar and that there was no difference in  $T_g$  values between samples containing sucrose and molasses in ice cream mix. This may be a desirable result since the primary purpose is not to alter the quality of a conventional component to an alternative and cost-reducing alternative, as it does not cause a change in quality characteristics.

### Colour properties

Colour properties are among the quality parameters that affect the sensory properties of foods. In this study, the colour properties of the ice cream samples were obtained using the CIE-Lab system and colorimeter. For this purpose,  $L^*$  (brightness),  $a^*$  (red-green),  $b^*$  (yellow-blue) and WI (whiteness index) values of all ice cream samples were determined (Table 2). With the use of SDSM, it was determined that all of the ice cream colour characteristics differed significantly, and the severity of this difference increased due to the powder molasses concentration ( $P < 0.05$ ). While  $L^*$  and WI values decreased with the use of powder molasses, it was also determined that due to the increase in  $a^*$  value, the redness increased. In addition,  $b^*$  value increased significantly and therefore yellowness increased. Roland *et al.* (1999) investigated the effects of some oil substitutes, including maltodextrin on the use of nonfat ice cream and their effects on product quality, and reported that only maltodextrin-containing samples had the same colour properties as 10% fat freeze. Thus, colour of the samples is affected

especially due to the liquid molasses content which is a component of SDSM. Some pigments contained in the plant source during sugar production also take part in the structure of molasses and are not separated and therefore become the main cause of the observed colour change. With further studies on this area, (i) masking this colour change caused by the use of different colorants or (ii) application of purification processes for the separation of pigments in molasses composition before powder molasses production and consequently developing of colour properties can be achieved.

### Total phenolic content

Molasses is a high content of sucrose, as well as fructose, glucose, protein and minerals. These substances contain essential elements (Fe, Ca, Na, K, Zn, Mn and Mg) as well as natural antioxidants and phenolic compounds (Jaffe, 2015; Soukoulis, & Tzia, 2018). On the basis of ice cream, the presence of phenolic compounds should be considered in particular for two reasons. The first is the positive development and change in the potential bioactivity and functional properties of the product due to the level and composition of the phenolic compounds. The second reason is the changes in the sensory and technological properties of the product as a result of the interaction of nitrogenous compounds, especially proteins, with phenolic substances.

The total phenolic content of the samples in this study was found to vary between 68.2 and 1013.0 mg GAE kg<sup>-1</sup> (Table 2). When the replacing ratio of sucrose with powder molasses increased in the ice cream composition, the total phenolic substance level increased considerably ( $P < 0.05$ ). The data show that powder molasses is an important source of phenolic substances.

### Sensory properties

The reformulation of the ice cream composition can cause significant changes in both physical and sensory properties (Laura Rolon *et al.*, 2017). The sense of pleasure during the consumption of ice cream is one of the most important quality parameters that affect market success. Fat content and types affect these parameters (Roland *et al.*, 1999; Hyvönen *et al.*, 2003; Akbari *et al.*, 2016; Laura Rolon *et al.*, 2017). In sugar substitution studies, the choice of bulking agent, which can also act as fat substitute, may be advantageous. Thus, the calorie decrease in the product as well as the pleasure taken from the product increases. This leads to an increase in consumer acceptance and appreciation. In this study, the sensory properties of ice cream samples were specifically determined by the appearance, texture, flavour, aroma, melting behaviour and general

acceptance (Table 3). Previous studies show that different levels of sucrose and glucose syrup with other bulk sweeteners affect sensory properties (Ozdemir *et al.*, 2008; Cadena *et al.*, 2012; Mariano & Alamprese, 2017). These bulking agents include maltodextrin, which is also used as a fat substitute. It was determined that the nonfat ice cream samples obtained by using maltodextrin are the most suitable fat substitution components in terms of sensory properties (Roland *et al.*, 1999).

There was a significant decrease as a result of using SDSM in appearance, flavour, aroma and general acceptance parameters ( $P < 0.05$ ). However, increase in SDSM usage level did not cause significant effect on the appearance. This is thought to be due to the effect on the aromatic properties of molasses, in particular, in reducing the sweetness level. Astringent off-flavour and aroma properties of sugar beet molasses are disadvantages of using a food component (Filipčev, 2011). Maillard browning carbohydrate–amino acid condensation products, formed during sugar processing, are also in very high concentration in molasses and range from low organic compounds to complex aromatic polymers. They are strongly involved in the colour and aroma of molasses (Valli *et al.*, 2012). Besides the flavour, the aroma properties of the samples other than Ice25 were also graded by panellists with low scores, with an increase in the level of SDSM and a decrease in the level of sucrose. In the previous studies, it was reported that there was an increase in sweetness with the use of maltodextrin as a fat substitute; however, Laura Rolon *et al.* (2017) could not determine this effect with the sensory analysis. A similar situation is also present for the effect of flavouring agents used in the ice cream composition. Also, the structural components of the ice cream have an effect on the release of aroma (Soukoulis *et al.*, 2010a). In a previous experiment maltodextrin usage for the nonfat ice cream at about 2% of the whole ingredients, was found to have a decreasing effect on the fattiness and creaminess properties while increasing the aroma release (Hyvönen *et al.*, 2003). Additional to their

effects on sweetness, sweeteners are substances that are important for the development of retronasal olfactory in the oral process (Soukoulis, & Tzia, 2018). In addition, some hydrocolloids, including polysaccharides, can exhibit aroma masking effect (Varela *et al.*, 2014). However, there are studies showing various results. For example, Soukoulis *et al.* (2008) reported that the presence of hydrocolloid caused an increase in perceived aroma level. Therefore, in the interpretation of the results, used components with similar properties, type, chemical composition or concentration should be carefully examined with the consideration of the complex colloid system. The use of bulking agents with higher sweetness levels can improve aroma severity, and macromolecular sweeteners may play a role in suppressing aroma (Soukoulis *et al.*, 2010a). For example, the substitution of sucrose from different origins with molasses resulted in changes in some sensory properties such as flavour and aroma. Sweetness was reduced in ice cream samples, especially with the use of sugar cane molasses, which was associated with invert sugar levels (Soukoulis, & Tzia, 2018). As a result, the use of powder molasses can be stated to be more advantageous with the use of SDSM up to 50% in order to obtain the product having the flavour, aroma and general acceptance levels closest to the sample containing only sucrose.

In the evaluation of the melting behaviour of the ice cream samples, using SDSM and its usage level were not associated with degree of likings. It has been observed that melting behaviour does not make differences in sensory perception due to differences in samples consistency values. However, in parallel with the increase in the value of consistency, the feeling of coating on the mouth/palate is expected to increase (Varela *et al.*, 2014). Intraoral behaviour has a significant effect on the acceptance of ice cream, which is perceived by the movement and friction of the tongue and other oral structures during the melting of the product, and by the increase in temperature; it is expected to become smoother, creamier and viscous fluid (Varela *et al.*, 2014).

**Table 3** Sensory properties of ice cream samples

Samples	Appearance	Texture	Flavour	Aroma	Melting resistance	General acceptance
Ice0	9.17 ± 1.00 <sup>a</sup>	7.91 ± 2.06 <sup>a</sup>	8.57 ± 1.56 <sup>a</sup>	8.43 ± 1.52 <sup>a</sup>	7.43 ± 2.37 <sup>ab</sup>	8.74 ± 1.36 <sup>a</sup>
Ice25	7.78 ± 1.90 <sup>b</sup>	7.35 ± 1.84 <sup>a</sup>	7.35 ± 2.73 <sup>b</sup>	7.96 ± 2.44 <sup>a</sup>	7.96 ± 2.44 <sup>a</sup>	7.96 ± 1.81 <sup>ab</sup>
Ice50	7.57 ± 2.02 <sup>b</sup>	7.57 ± 1.78 <sup>a</sup>	7.52 ± 2.03 <sup>ab</sup>	7.57 ± 1.57 <sup>ab</sup>	6.96 ± 2.10 <sup>ab</sup>	7.35 ± 1.64 <sup>b</sup>
Ice75	7.83 ± 1.92 <sup>b</sup>	7.17 ± 1.66 <sup>a</sup>	5.48 ± 2.00 <sup>c</sup>	6.57 ± 2.30 <sup>bc</sup>	6.57 ± 2.45 <sup>b</sup>	6.17 ± 1.45 <sup>c</sup>
Ice100	7.78 ± 2.12 <sup>b</sup>	7.61 ± 1.24 <sup>a</sup>	3.91 ± 1.84 <sup>d</sup>	5.57 ± 2.60 <sup>c</sup>	6.96 ± 2.34 <sup>ab</sup>	5.00 ± 1.92 <sup>d</sup>

0: unacceptable (not like at all); 5: acceptable (like moderately); 10: excellent (like extremely). All data are reported as the mean ± SD. Different letters within the same rows are significantly different ( $P < 0.05$ ). Sugar:spray-dried sugar beet molasses ratios were 0:100, 25:75, 50:50, 75:25 and 100:0 for samples coded as Ice100, Ice75, Ice50, Ice25 and Ice0, respectively.

A good ice cream texture is determined only if the components are in a balanced state and form a uniform structure (Varela *et al.*, 2014). Therefore, it is useful to consider the effect of all components on the texture. It has been found that the use of sugar cane molasses instead of sucrose causes an increase in the sensory properties of ice cream such as coarseness and iciness, as well as increased instrumental hardness related to a strong correlation with consistency (Soukoulis, & Tzia, 2018). The textural properties of the ice cream samples had a low effect, and this effect was considered to be tolerable. Polysaccharide and oligosaccharides provide structure formation and stability development as a result of their effects on the formation of macromolecular networks, phase separation, and cryogenisation thanks to the water-bonding property (Soukoulis, & Fisk, 2016). For this reason, the use of maltodextrin can be predicted to affect the sensory properties, specifically the texture. Previous studies have shown the increase in instrumental hardness with the use of maltodextrin (Soukoulis *et al.*, 2010a), but no significant change was determined in the use of fat substitution (Laura Rolon *et al.*, 2017). In another study, it was concluded that the use of maltodextrin as an fat replacer in fat-free ice creams showed a decrease in hardness (Roland *et al.*, 1999). Therefore, there are studies showing that maltodextrin exhibits different results on the ice cream's textural properties.

Hydrocolloids or other components that act as hydrocolloids retain free water and reduce the growth of ice crystals. Therefore, when the ice cream has undergone a structurally good stabilisation, the ice crystals remain small (Varela & Ares, 2014), which may affect the textural perception positively. Because the size of ice crystals is one of the major factors affecting the textural properties of ice cream, where especially coarse and icy texture is the primary reason for the large and unhomogeneous size distribution (Baer *et al.*, 1999). The size of the ice crystals in ice cream affects the texture and causes rough texturing if longer than 40–50  $\mu\text{m}$ , while the presence of ice crystals below 20  $\mu\text{m}$  is among the conditions required for smooth texture (Velez Ruiz *et al.*, 1997). In addition, an increase in the unfrozen water levels causes a decrease in the ice crystals which leads to lower hardness (Akbari *et al.*, 2016). Powder molasses-containing substances with different solubility properties in their composition are predicted to have different levels of solubility and some may influence the sensory texture due to their influence on the number and size of the free water amount and ice crystals, as some may exhibit stabiliser-like behaviour. However, there was not a significant effect as a result of using SDSM on texture properties of samples ( $P < 0.05$ ).

In addition to sugar substitutes, agents which can also be used as fat substitutes are alternative

components that have an effect on the general acceptance level of the consumer. For instance as Laura Rolon *et al.* (2017) reported, there is a decrease in consumer acceptance, although substitution of fat by maltodextrin showed minimal changes in the physical and sensory properties of ice cream. At the end of this study, with the increase in the use of SDSM as sucrose substitution, a significant decrease was observed in general acceptance. The main source of this decrease might be the negative impact of SDSM on ice cream flavour and aroma properties.

## Conclusion

Studies on food reformulation and identification of alternative food components have been accelerating along with the change in food resources as well as industrial competition. Experts in the field of food technology primarily focus on waste and by-products in this area. Molasses of different origin are also among these by-products. However, in order to improve the possibilities of use in different foodstuffs, it is of utmost importance to pretreat and alter the physical properties. In this study, the processing and usage possibilities of molasses, which is a by-product, in powder form were examined. As a result of this study, it has been concluded that a common ingredient such as maltodextrin and a widely used technique such as spray drying can be used for the purpose of sucrose substitution in the ice cream composition by the powder sugar beet molasses. With this method of use, it is also possible to obtain a cost-reducing component as well as sugar substitution. Particularly with the replacement of 25% sucrose with powdered sugar beet molasses, it will be possible to observe tolerable changes in the main ice cream quality parameters such as flow behaviour, sensory and thermal properties. In addition, in further studies, the use of maltodextrins with different DE values, the optimisation of the spray dryer conditions and the components that can mask the negative effects on the aroma and flavouring can improve the quality parameters and cause more replacement of sugar.

## Conflicts of interest

The authors have no conflicts of interest to disclose.

## Ethical approval

Ethics approval was not required for this research.

## Data availability statement

Research data are not shared.

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