



# The Effect of Gypsophila Extract as Natural Emulsifier on the Steady, Dynamic Rheological Behavior and Microstructural Properties of the Ice Cream Mix

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## Abstract

In this study, it was aimed to use the gypsophila extract as an emulsifier in ice cream production. For this purpose, the effect of different ratios of gypsophila extract on the steady, dynamic rheological properties and microstructural properties of the ice cream mix was aimed. All of the ice cream mix samples showed non newtonian shear thinning character. K and n values were calculated by modeling the rheological properties of the steady shear exponent with the law model. The K value of the frozen samples decreased to a certain level with the addition of gypsophila extract, but showed the highest value in the sample containing 0.3% gypsophila extract and 0.1% lecithin. All of the ice cream mix samples exhibited viscoelastic solid character. In the ice cream mix samples produced with gypsophila extract, the oil droplets showed a more homogeneous distribution in the form of small particles. The results of this study showed that the gypsophila extract could be used successfully as an emulsifier in ice cream production.

**Keywords:** Rheology, steady shear, microstructural, gypsophila extract.

## Çöven Estraktının Dondurma Miksinin Steady, Dinamik Reolojik Davranışları Ve Mikroyapısal Özelliklerine Etkisi

### Öz

Bu çalışmada çöven ekstraktının dondurma üretiminde emülgatör olarak kullanım potansiyeli amaçlanmıştır. Bu amaçla, farklı oranlarda çöven ekstraktının dondurma miksinin steady, dinamik reolojik özellikleri ve mikroyapısal özellikleri üzerine etkisi amaçlanmıştır. Dondurma miksi örneklerinin tamamı non newtonian shear thinning karakter göstermiştir. Steady shear reolojik özellikleri üssü yasa modeli ile modellenerek K ve n değerleri hesaplanmıştır. Dondurma örneklerinin K değeri çöven ekstraktı ilavesiyle belirli bir düzeye kadar azalmış ancak en yüksek değerini %0.3 çöven ve %0.1 lesitin içeren örnekte göstermiştir. Dondurma miksi örneklerinin tamamı viskoelastik katı karakter sergilemiştir. Çöven ekstraktı ile üretilen dondurma miksi örneklerinde yağ damlacıkları küçük partiküller halinde ve daha homojen dağılım göstermiştir. Bu çalışmanın sonuçları çöven ekstraktının dondurma üretiminde emülgatör olarak başarılı bir şekilde kullanılabileceğini göstermiştir.

**Anahtar Kelimeler:** Rheology, yatışkan faz, mikroyapısal özellik, gypsophila ekstraktı.

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## 1. Introduction

Gypsophila (soapwort) belongs to the genus Gypsophila of the Caryophyllaceae family and is a perennial herbaceous plant with stemless and many-branched leaves, white flowers, and pile-rooted (Yıldırımli, 2002). Plants of the genus, Gypsophila mainly grow in the Mediterranean region, especially Turkey, Caucasus, northern Iraq, and northern Iran (Acebes, Daz-Lanza, & Bernabé, 1998). In Turkey, 56 species belonging to 60 taxa (out of 126 species in the World) of the Caryophyllaceae family naturally grow. 46 of these species, provided that a few of them have commercial importance. The soapwort plant's commercially used roots and rhizomes are dried and sold. Soapwort extract is obtained by boiling soapwort roots and rhizomes in water. The main component of soapwort extract is saponin. Battal, Sarı, and Veliöğlü (2003) reported that total saponin was 11.58-19.58% in soapwort extracts.

Many food manufacturers are trying to replace synthetic food ingredients with more natural and sustainable alternatives. The word saponin is derived from the Latin word *sapo*, which means soap (SIROHI, SINGH, & PUNIYA, 2007). Saponins are natural surfactants and the physical properties of saponins in solution are due to their amphiphilic nature. The saponins act as emulsifiers, their surfactant is very strong, they form stable foams. All saponins have one or more sugar chains attached to the hydrophobic aglycon (triterpenoid or steroid) in common (Vincken, Heng, de Groot, & Gruppen, 2007).

A stable white foam is generated when the soapwort extract is stirred at high speed (Çağlayanlar, 2006). In Turkey, soapwort extract has been used for the foam formed in the food industry in the production of tahini halva, sultan (pasa) delight and nougat. This extract, which is turned into foam, bleaches the color of the product, prevents the separation of sesame oil from the halva as an emulsifier, ensures the desired structure of the product, increases the volume, and thus ensures the formation of characteristic features specific to the product (Çam & Topuz, 2018; Veliöğlü, 2001). Çelik, Yılmaz, Işık, and Üstün (2007) studied the effect of soapwort extract on physical and sensory properties of sponge cakes and rheological properties of sponge cake batters. In this study, the effect of soapwort extract on rheological properties of ice cream.

Ice cream is an oil-in-water emulsion obtained by blending milk with emulsifiers, stabilizers, sugar, and flavoring substances (Boor, 2001). Ice cream is an important food product since it is easy to digest, provides energy, and is high in vitamins and minerals, in addition to being delicious. However, ice cream has a very complex physicochemical system. The stability of this system is an important factor in the production of quality and suitable ice cream. For the quality of ice cream production, the ice cream mix must be balanced in mass and well-processed, as well as emulsifier and stabilizer substances added to the content. These substances are added to the mixture in small quantities and are effective in the physical quality of the ice cream and the formation of the viscous structure (Elife, ERDEM, & TEKİN, 2017).

The aim of this study is to reduce the cholesterol in the blood in terms of human health (Kim et al., 2003), hemolytic (facilitating oxygen transport), prevent cancer (Gurfinkel & Rao, 2003), and trigger bone development (Yamaguchi, Ono, & Ma, 2001), which is reported to have positive effects, as well as technically stable foam forming, bleaching, emulsifier in products. The aim of the study is to measure the changes in the

chemical, physicochemical, rheological, textural, and sensory properties of ice cream by incorporating the saponin extract containing the glycoside called saponin, which can act as an antiseptic and reduce the surface tension, in the production of ice cream, provided that the maximum dose that can be taken daily is not exceeded, and to determine whether it is beneficial to use the lye extract in ice cream production. For this purpose, it will be revealed how the foaming feature of the litter's effect on the volume increase of the ice cream, the emulsifier feature on the icing, oil release, microstructure, texture properties (especially the gumminess), and the bleaching feature on the color, as well as the effects of the use of lint extract on the first dripping time and total melting time of the ice cream.

## 2. Material and Method

The cow milk used in this study were obtained from local farm. XG and egg yolk powder (EYP) were obtained from Sigma-Aldrich (Sigma Chemical Co., St. Louis, MO), and the sugar, milk, and milk cream were purchased from the local market.

### 2.1. Preparation of ice cream samples

The formulation data to be used in ice cream production was shown in Table 1. Full-fat cow's milk is used in the production of ice cream. Milk dry matter was increased up to 14.5 percent by evaporation at 100 C. 40 g of Emulsifier, 30 g of Guar gum, 30 g of European salep and 30 g of Osmaniye salep were taken into an empty container. Afterwards, 1.8 kg of sugar was added to the same container. When the temperature of the mixture came to 85°C, pasteurization was performed for 30 minutes. The pasteurized mixes were cooled to 65-70°C and taken into hoppers. It was cooled a little more in the boxes and rested at +4°C for 1 night. For the trial production of gypsophila extract in ice cream, 1, 2.5, 5, 4 mL of gypsophila extracts per 1 kg of mix were separately whipped with 50 g of sugar in 50 ml of hot water for 10 minutes. Soup mixture, whose volume was increased by whisking, was poured into the ice cream machine with the mix. The ice cream mix was mixed in the ice cream machine for 10 minutes. Packaged ice creams were left to rest at -18°C for 1 day.

### 2.2. Rheological Analyzes

Flow behavior and dynamic rheological behavior of ice cream mixes were tested by a stress and temperature-controlled rheometer (MCR 302, Anton Paar, Australia). A parallel plate probe (PP50, Anton Paar, Australia) was utilized for steady and dynamic rheological analysis. All measurements were conducted in triplicated at 25 °C.

#### 2.2.1. Flow Behavior Rheological Properties

The steady shear rheological properties of ice cream mixes were determined using a parallel plate probe (plate diameter 50 mm, gap size 0.5 mm) with a shear rate in the range 0.1–100 (1/s). The measurement was conducted at a temperature of 25 °C, and three parallel measurement were performed for each sample. The data obtained from steady shear the rheological analysis were modeled by the power law model, and nonlinear regression was utilized to estimate K and n values;

$$\tau = K\dot{\gamma}^n \quad (1)$$

where, the  $\tau$  value shows the shear stress (Pa),  $K$  the consistency coefficient (Pa.sn),  $\dot{\gamma}$  the shearrate ( $s^{-1}$ ), and  $n$  the flow behavior index.

### 2.2.2. Dynamic Rheological Properties

Parallel plate configuration was applied for the dynamic rheological analysis of ice cream mix samples. Before the frequency sweep test, the amplitude sweep test was conducted at a range between 0.1% and 100% strain to observe the linear viscoelastic region. The frequency sweep test was carried out in the frequency range of 0.1–10 Hz and angular velocity of 0.1–64 ( $\omega$ ). Elastic modulus ( $G'$ ) and viscose modulus ( $G''$ ) corresponding to angular velocity and frequency values were tested.

### 2.2.3. Microstuructural Properties

A light microscope (Olympus BX41, Japan) at 100 time magnification was used to examine morphology of the emulsions before and after thermal loop test. One droplet of sauce sample without dilution was placed on microscope slide and covered by a coverslip. Different areas of slides were observed to evaluate the emulsion stability thoroughly.

### 2.2.4. Statistical anlysis

All Ice cream samples were prepared in three replications, and four parallel measurements were carried out from each replicate. The mean and standard deviation values were shown. Statistical analysis were performed wit the Statistica software package (IBM, USA). To compare the mean values of the rhepological parameters, the duncan multiple comparison test conducted ( $p < 0.05$ ). The Power Law model parameters of steady shear rheological analysis were estimated by using of nonlinear regression analysis. Statistica software program (Stat Soft Inc., Tulsa, UK) was utilized to conduct nonlinear regression analysis.

## 3. Results and Discussion

### 3.1. Steady Shear Rheological Properties

Flow behavior rheological properties of ice cream mixes at 4°C and 25 °C are shown in figure 1. The rheological properties of the ice cream mix are one of the most important parameters

affecting the quality of the final product. Having a certain level of consistency of the ice cream mix will affect the melting properties. As can be seen from the figure, a decreasing increase in the shear stress value was observed depending on the increasing shearrate. In this case, a decrease in the viscosity of the ice cream mixes was observed due to the increase in shear rate. All of the ice cream mixes showed a non-Newtonian shear thinning flow characteristic. This flow characteristic is typical flow behavior of ice cream mixes. Similar results have been reported in the literature (Karaman & Kayacier, 2012; Martin, Odic, Russell, Burns, & Wilson, 2008; Tekin, Sahin, & Sumnu, 2017). This type of flow behavior of the ice cream mixer can be explained by the deformation observed in the structure together with the weakening of the interaction between protein and stabilizers due to the increased shear rate (Sharma et al., 2017).

Steady shear flow rheological behavior of the ice cream mixer was modeled using the power low model to make a numerical comparison in order to make a numerical comparison. Power low model parameters are shown in Table 2. The fact that the  $R^2$  value is greater than 0.99 shows that the power low model can be used successfully in modeling the stady shear rheological properties of ice cream mixes. All of the  $n$  values of the samples were found to be less than 1. This result is a different indication that the shear thinning of the samples exhibited a flow behavior characteristic. The values for  $n$  of the samples varied between 0.258 and 0.290. These value ranges are expected values from ice cream mix samples. A value of  $n$  below 0.2 means that the samples exhibit excessive consistency or weak gel, which is undesirable in terms of ice crystals and melting profile in the final product. The  $K$  values of the samples were determined as 6.97-9.38  $\text{Pa.s}^n$  and 4.59-6.31  $\text{Pa.s}^n$  for 4°C and 25°C, respectively. A significant difference was observed between the  $K$  values of the samples. As the lecithin level decreased, the  $K$  value decreased, while the  $K$  value showed the highest value in the sample containing 0.3 gypsophila extract and 0.1 lecithin. The lowest  $K$  value was obtained from the sample containing purely gypsophila extract. These results indicate that gypsophila extract does not have as much effect as lecithin when used alone, but they can have a synergistic effect when used together. This synergy can be explained by the interaction observed between the hydrophilic groups of lecithin and saponin of the gypsophila extract. The  $K$  values of the samples at 4°C were found to be significantly higher than the  $K$  values at 25 °C. This result is expected. With the increase in temperature, the interaction of macromolecules with water weakened, and the viscosity and consistency value decreased.

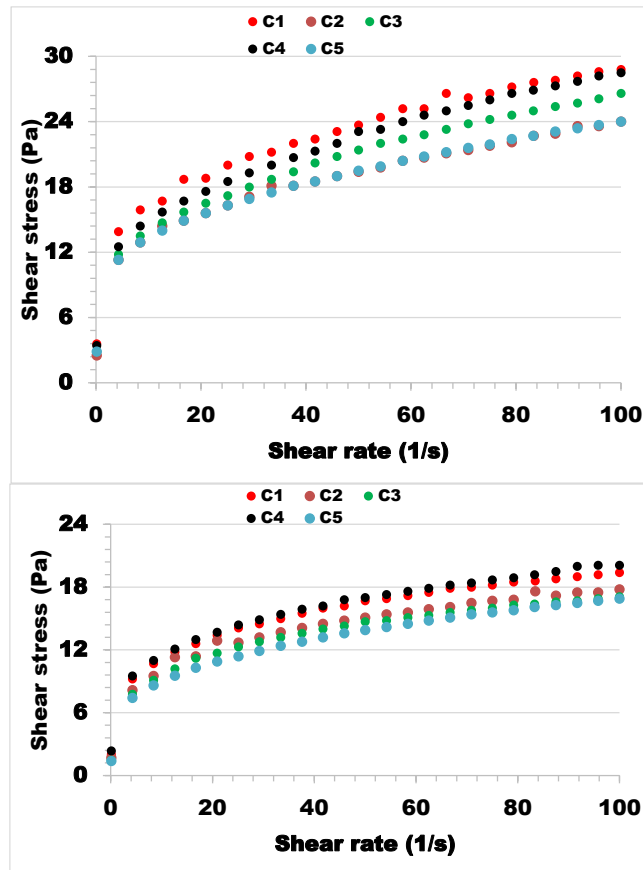


Fig1 Steady Shear rheological properties of ice cream mixes.

Table 1. Power law model parameters corresponding to steady shear rheological analysis.

Temperature	Samples	Power Law model parameters		
		K	n	R <sup>2</sup>
4 °C	S1	8.7273 <sup>b</sup>	0.2588	>0.99
	S2	7.1016 <sup>c</sup>	0.2611	>0.99
	S3	6.9773 <sup>d</sup>	0.2875	>0.99
	S4	9.3812 <sup>a</sup>	0.2909	>0.99
	S5	7.0181 <sup>cd</sup>	0.2643	>0.99
25 °C	S1	6.1842 <sup>b</sup>	0.2509	>0.99
	S2	5.5897 <sup>c</sup>	0.2539	>0.99
	S3	5.2452 <sup>d</sup>	0.2595	>0.99
	S4	6.3103 <sup>a</sup>	0.2531	>0.99
	S5	4.5948 <sup>e</sup>	0.2832	>0.99

S1, S2, S3, S4, S4 and S5 indicate the samples formulated with 0.4, 0.3, 0.2, 0.1 and 0.0 lecithin respectively. The different t lowercase letter in same column shows statically differences.

### 3.2. Dynamic Rheological Properties

Fig 2 demonstrated the dynamic rheological behavior of the ice cream mix samples. Steady shear rheological properties, as well as dynamic rheological properties, is an important rheological property for ice cream mix. Since it contains stabilizer in the ice cream mix structure, it is expected to exhibit viscoelastic solid behavior. As a result of the interaction of the stabilizers with water and other macromolecules, the mobility of the continuous phase is restricted and the rheological character emerges in the solid structure. As can be seen from the figure, the  $G'$  value is higher than the  $G''$  value in the entire frequency range. This result shows that all of the ice cream mix samples exhibit viscoelastic solid character. The similar results were reported from previously

published studies (Atik, Cakmak, Avci, & Karasu, 2021; Toker et al., 2013). A decrease was observed in  $G'$  and  $G''$  values as the slurry ratio in the stuffing increased. These results are in agreement with steady shear rheological properties. Another result to be explained based on the figure is that the difference between  $G'$  and  $G''$  values is low in the low frequency range. As the frequency value increases, the difference between  $G'$  and  $G''$  values increases. This increase in the  $G'$  value, which appears due to the increase in the frequency value, shows the deformation in the structure. These results showed that all of the samples could meet the rheological properties expected from ice cream mixes.

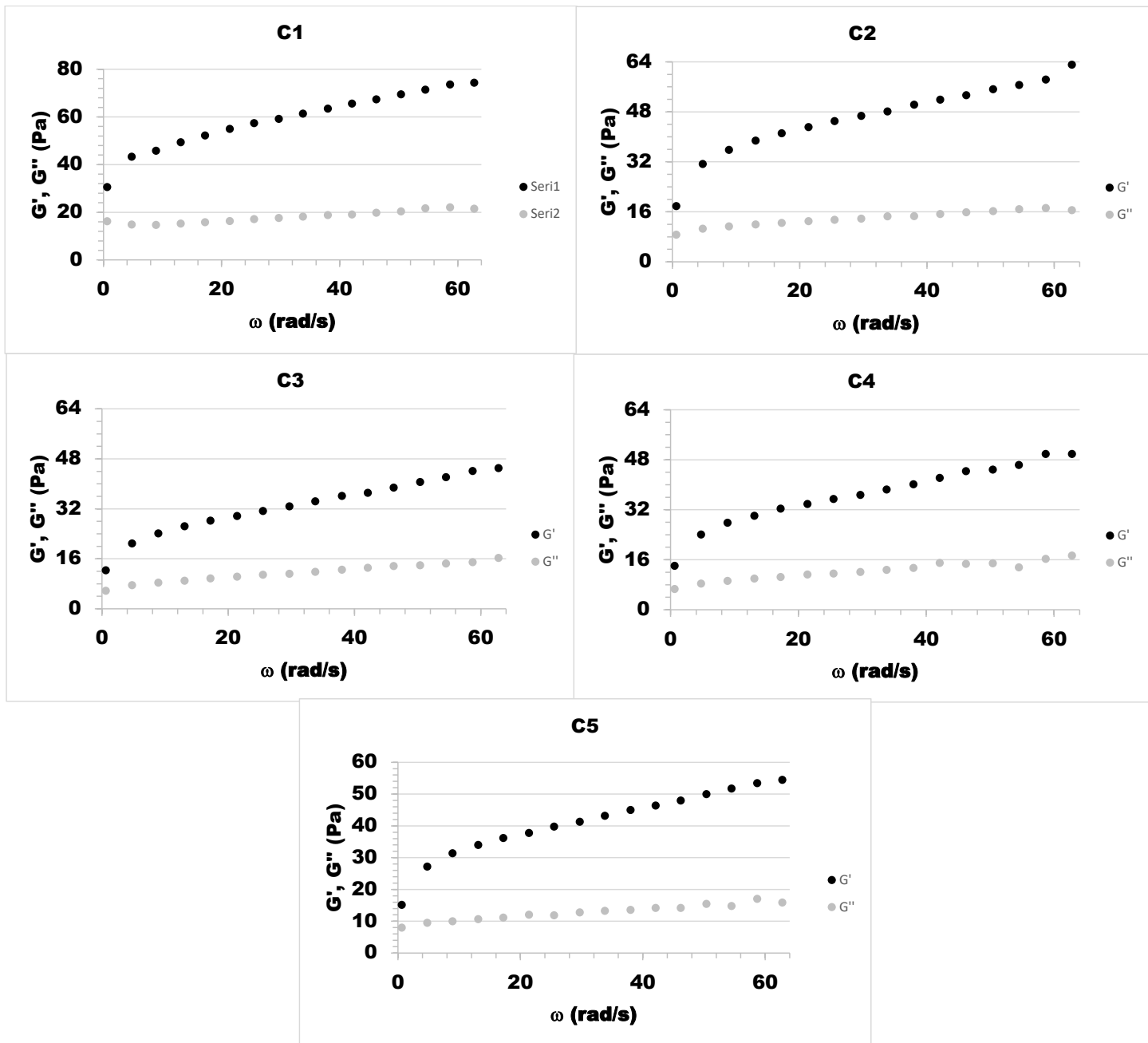


Fig 2. Dynamic rheological properties of ice cream mix.

### 3.2. Micro Structural Properties

Ice cream mix is an emulsion in which oil droplets are dispersed in the continuous phase of water. In this type of emulsion, the size of the oil droplet and the distribution of the droplet size is an important factor that determines the stability of the emulsion. For ice cream mixes, it is desired that the oil droplets were homogeneously dispersed in the water as small particles. Thus, an Oil/Water emulsion with high emulsion stability is obtained. Emulsifiers are one of the important factors affecting oil droplet size and distribution. Figure 3 shows the oil droplet distribution. It has been observed that the oil droplet size exhibits different behavior according to the emulsifier type used. It has been clearly seen that the oil particle sizes of the samples containing the gypsophila extract showed a lower and more homogeneous distribution. This indicates that the gypsophila

extract can be used to improve the emulsion properties of the ice cream mix. The lower droplet size and homogeneous distribution of the oil droplet can be explained by the saponin richness of the gypsophila extract. The difference between hydrophilic and lipophilic balance values of saponin and lecithin may have caused this result. The saponin showed a high HLB value, resulting in higher adsorption kinetics at the oil-water interface and ultimately a decrease in oil droplet size (Salminen, Bischoff, & Weiss, 2020). The easy dissolution of saponin in water through its hydrophilic groups and its interaction with other components may have had a positive effect on the oil droplet distribution in the ice cream mix. Similar results have also been reported in the literature (Jarzebski et al., 2020; Salminen et al., 2020).

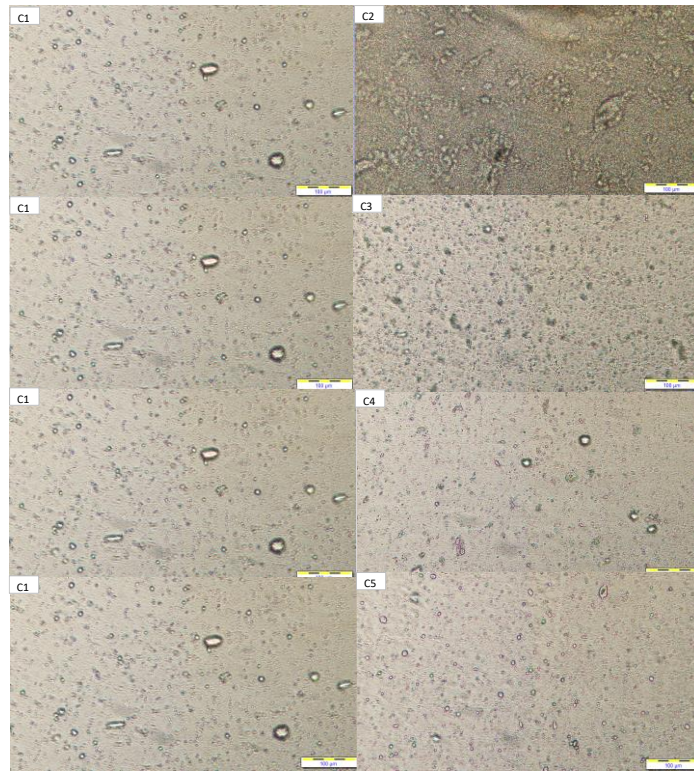


Fig 3. Microstructural properties of ice cream mix.

### Conclusion

In this study, the potential of using gypsophila extract as an emulsifier in ice cream production was investigated. All products showed shear thinning and viscoelastic characteristics expected from ice cream. The oil droplets of the products containing Çöven extract were lower and the oil droplets were more homogeneously distributed. The results of this study showed that the cayenne extract can be used successfully in ice cream production.

### References

- Acebes, B., Daz-Lanza, A. M., & Bernabé, M. (1998). A saponin from the roots of *Gypsophila bermejoi*. *Phytochemistry*, 49(7), 2077-2079.
- Atik, I., Cakmak, Z. H. T., Avcı, E., & Karasu, S. (2021). The Effect of Cold Press Chia Seed Oil By-Products on the Rheological, Microstructural, Thermal, and Sensory

- Properties of Low-Fat Ice Cream. *Foods*, 10(10). doi:10.3390/foods10102302
- Battal, H., Sarı, F., & Velioglu, S. (2003). Çöven ekstraktı üretimi üzerine bir araştırma.
- Boor, K. J. (2001). ADSA foundation scholar award fluid dairy product quality and safety: looking to the future. *Journal of Dairy Science*, 84(1), 1-11.
- Çağlayanlar, E. (2006). The effect of soapwort extract on starter performance, dough rheological properties and bread quality.
- Çam, İ. B., & Topuz, A. (2018). Production of soapwort concentrate and soapwort powder and their use in Turkish delight and tahini halvah. *Journal of Food Process Engineering*, 41(1), e12605. doi:https://doi.org/10.1111/jfpe.12605
- Çelik, İ., Yılmaz, Y., Işık, F., & Üstün, Ö. (2007). Effect of soapwort extract on physical and sensory properties of sponge cakes and rheological properties of sponge cake batters. *Food Chemistry*, 101(3), 907-911. doi:https://doi.org/10.1016/j.foodchem.2006.02.063

- Elife, K., Erdem, T. K., & Tekin, F. B. (2017). Maraş Dondurması Üretimi ve Üretilen Dondurmanın Fizikokimyasal Niteliklerinin Belirlenmesi. *Caucasian Journal of Science*, 4(1), 45-56.
- Gurfinkel, D. M., & Rao, A. V. (2003). Soyasaponins: The Relationship Between Chemical Structure and Colon Anticarcinogenic Activity. *Nutrition and Cancer*, 47(1), 24-33. doi:10.1207/s15327914nc4701\_3
- Jarzebski, M., Siejak, P., Smulek, W., Fathordoobady, F., Guo, Y., Pawlicz, J., . . . Pratap-Singh, A. (2020). Plant Extracts Containing Saponins Affects the Stability and Biological Activity of Hempseed Oil Emulsion System. *Molecules*, 25, 2696. doi:10.3390/molecules25112696
- Karaman, S., & Kayacier, A. (2012). Rheology of Ice Cream Mix Flavored with Black Tea or Herbal Teas and Effect of Flavoring on the Sensory Properties of Ice Cream. *Food and Bioprocess Technology*, 5(8), 3159-3169. doi:10.1007/s11947-011-0713-5
- Kim, S.-W., Park, S.-K., Kang, S.-I., Kang, H.-C., Oh, H.-J., Bae, C.-Y., & Bae, D.-H. (2003). Hypocholesterolemic property of *Yucca schidigera* and *Quillaja saponaria* extracts in human body. *Archives of pharmacal research*, 26(12), 1042-1046.
- Martin, P. J., Odic, K. N., Russell, A. B., Burns, I. W., & Wilson, D. I. (2008). Rheology of commercial and model ice creams. *Applied Rheology*, 18(1). Retrieved from <Go to ISI>://WOS:000254452800009
- Salminen, H., Bischoff, S., & Weiss, J. (2020). Formation and stability of emulsions stabilized by *Quillaja saponin*-egg lecithin mixtures. *Journal of Food Science*, 85(4), 1213-1222. doi:https://doi.org/10.1111/1750-3841.15104
- SIROHI, S., SINGH, N., & PUNIYA, A. (2007). Promising Plant Secondary Metabolites for Enteric Methane Mitigation and Rumen Modulation.
- Tekin, E., Sahin, S., & Sumnu, G. (2017). Physicochemical, rheological, and sensory properties of low-fat ice cream designed by double emulsions. *European Journal of Lipid Science and Technology*, 119(9). doi:10.1002/ejlt.201600505
- Toker, O. S., Karaman, S., Yuksel, F., Dogan, M., Kayacier, A., & Yilmaz, M. T. (2013). Temperature Dependency of Steady, Dynamic, and Creep-Recovery Rheological Properties of Ice Cream Mix. *Food and Bioprocess Technology*, 6(11), 2974-2985. doi:10.1007/s11947-012-1005-4
- Velioğlu, S. (2001). Çöven Ekstraktı Üretim Koşullarının Belirlenmesi ve Standardize Edilmesi Üzerine Araştırma. TÜBİTAK, Tarım Ormancılık ve Gıda Teknolojileri Araştırma Grubu (TOGTAG), Proje(2467).
- Vincken, J.-P., Heng, L., de Groot, A., & Gruppen, H. (2007). Saponins, classification and occurrence in the plant kingdom. *Phytochemistry*, 68(3), 275-297. doi:https://doi.org/10.1016/j.phytochem.2006.10.008
- Yamaguchi, M., Ono, R., & Ma, Z. J. (2001). Prolonged intake of isoflavone-and saponin-containing soybean extract (Nijiru) supplement enhances circulating  $\gamma$ -carboxylated osteocalcin concentrations in healthy individuals. *Journal of health science*, 47(6), 579-582.
- YILDIRIMLI, Ş. (2002). The chorology of the Turkish species of Caryophyllaceae, Casuarinaceae, Celastraceae, Ceratophyllaceae and Cercidiphyllaceae families. *Ot Sistematik Botanik Dergisi*, 9(2), 175-199.