Food Research 7 (2) : 186 - 193 (April 2023)
Journal homepage: https://www.myfoodresearch.com
FOOD RESEARCH
FULL PAPER

Development of a direct observation method and the influence of formulation parameter on frozen ice cream microstructure

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Article history:
Received: 7 September 2021
Received in revised form: 17 October 2021
Accepted: 20 February 2022
Available Online: 7 April 2023

Keywords:
Ice cream,
Fat content,
Food microstructure,
Microscopy,
Dairy processing

DOI:
https://doi.org/10.26656/fr.2017.7(2).696

Abstract

Ice cream microstructure is influenced by the ingredients used and processes conducted during production. The equipment required to observe the microstructure of this temperature-sensitive material is very expensive and might not be affordable for most small-scale ice cream business entrepreneurs. This study aimed to develop an economical and affordable device setup and method to capture images of ice cream microstructure in sub-freezing conditions. The arrangement comprises a portable Dino-Lite microscope placed in a freezer with a surrounding temperature of -21.5°C. The commissioning of the setup was conducted by viewing the microstructure of ice cream made with different percentages of oil content i.e., 0%, 4%, 5%, and 6% at 500× magnification. Several analyses were conducted to correlate the microstructure with the physical quality of ice cream i.e., overrun and melting rate. It was found that ice cream with higher fat content had smaller air cell sizes and well-distributed microstructural components. The air cell radius (*r*) decreased from 0.089 mm (0% oil) to 0.056 mm (6% oil). Ice cream with lower fat content had higher overruns and vice versa. Overrun of ice cream with 0% and 6% oil were 55.12% to 33.38%, respectively. The overrun decreased linearly with fat content (R^2 = 0.9859). The melting rate of ice cream with 0% and 6% oil contents were 1.82 g/min and 1.66 g/min, respectively. Ice cream with higher fat content melts slower and decreased linearly with oil content (R^2 = 0.9413). The device and method developed are reliable in characterizing the size of the microstructure of ice cream, which provides a fundamental understanding of the relationship between the ingredients used with the microstructure of ice cream for small-scale ice cream business entrepreneurs and even researchers.

1. Introduction

Ice cream is known as a complex food that consists of tiny air cells distributed in a continuous aqueous matrix that is partially frozen. It is important to choose the ingredients to make ice cream such as milk, cream, sugar, stabilizer, and emulsifier as the ingredients affect the development of structure, texture, and palatability desired. In ice cream production, all of these ingredients have their functions, which will affect the quality of the ice cream produced.

Ice cream is comprised of four main distinct phases: solid ice crystals, fat globules, air bubbles, and liquid unfrozen serum phase (Rahman *et al.*, 2013). Approximately 30% water, 50% air, 5% fat, and 15% matrix (sugar solution) by volume are the components of standard ice creams (Clarke, 2004). Ingredients are not the only factor that contributes to the quality of ice cream. The processing of ice cream also plays a fundamental and significant role in creating the perfect ice cream with good physical properties. Mixing the food, pasteurizing, homogenizing, ageing, freezing, and

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eISSN: 2550-2166 /
Published by Rynnye Lyan Resources

hardening are the main techniques for making ice cream. Each of the processes has its role and importance in the development of ice cream structure and texture.

Ingredients and processes create the microstructure of ice cream. The behaviour of the main components of ice cream i.e., ice crystals, air cells, and fat globules contribute to the texture of ice cream. Observing the microstructure of ice cream is very crucial in order to understand the relationship between the ingredients and processes with the structure and physical quality of ice cream. The study of ice cream microstructure requires a high-quality and effective microscopy technique to obtain images and information. Low-temperature scanning Electron Microscopy (LT-SEM) and cryo-scanning electron microscopy (cryo-SEM) are examples of the devices used in studying the microstructure of ice cream (Caldwell *et al.*, 1992; Wildmoser *et al.*, 2004). However, these devices are very expensive and are not readily available in most institutions, and limit the researcher's ability to further assess the quality of frozen materials, not to mention the small-scale ice cream business entrepreneurs.

A direct observation approach by optical microscopy with episcopic coaxial lighting was developed by physicists studying polar ice structures (Arnaud *et al.*, 1998). The method was then adopted by Faydi *et al.* (2001) to characterize the structure of the ice cream mix, and also by Fiol *et al.* (2017) to characterize the microstructure of their new ice cream product. The observation approach is based on the light flux reflected by the surface of the sample i.e., the air bubbles (Goff, 1997; Caillet *et al.*, 2003). Optical microscopy was also used to observe ice recrystallization in ice cream (Donhowe and Hartel, 1996a; Donhowe and Hartel, 1996b). Ice cream is a temperature-sensitive material where it needs to be stored at a cold temperature below -15°C to maintain its structure and texture. Hence, the process of observing the microstructure of ice cream needs to be conducted in a cold freezing environment. At some point in freezing conditions, condensation might occur on the ocular which will affect the observation process. A good handling technique is required to overcome the situation. It is of paramount importance for anyone dealing with ice cream be it in the research and also business to have a good understanding of the relationship between ingredients and processes with the microstructure and quality of ice cream.

This study aimed to develop and evaluate the performance of a cost-effective portable microscopy device arrangement that can be handily placed inside a freezer and can be used to conveniently check the microstructure of ice cream. A handheld digital stereomicroscope (Dino-Lite, Model AM4113, New Taipei City, Taiwan), equipped with a digital video camera and a LED light source was chosen to be used in the arrangement due to the portability that makes it easier to be navigated inside a freezer and capture the image of ice cream microstructure *in-situ*. The arrangement would help in correlating the effects of ingredient change with the microstructure and consequently towards the physical quality of ice cream i.e., overrun and melting rate. The setup is expected to be able to give economic and fast checking of the microstructure of temperature-sensitive frozen materials, although the image generated might not be at par with the quality of images captured using high-end microscopy equipment. This study was led by the success of the previous work conducted by the team i.e., in-situ viscoelastic characterization and modelling of ice cream via a customized compression test device in sub-freezing conditions (Rahman *et al.*, 2019).

2. Materials and methods

2.1 Preparation of ice cream mix

Ice cream samples were prepared based on the formulation stated by Parid *et al.* (2018) as shown in Table 1. Full cream milk, sugar, whey powder, creamer, and flavouring were obtained from local suppliers. The emulsifier used was Olein PK-10 (BIS Chemicals, Shah Alam, Malaysia) and the commercial CMC used was purchased from Warisnove Sdn. Bhd. (Gambang, Pahang, Malaysia). For this experiment, 4 different samples were prepared by manipulating the oil content which was 4%, 5%, 6% oil, and also a control sample (0% oil).

Table 1. Formulations of ice cream samples with different percentages of oil contents (% w/w)

Ingredients	Composition (% w/w)
Water	60.9
	56.9
	55.9
	54.9
Oil	0
	4
	5
	6
Skimmed Milk Powder	18.1
Sugar	16.3
Creamer	3.6
Emulsifier	0.4
Stabilizer	0.3
Flavouring	0.4

The dry ingredients were dispersed under agitation into the wet ingredients at room temperature by manual stirring. The mixture was then batch pasteurized at 80°C for 15 s and consequently made to go through the two-stage homogenization process using a laboratory-scale

https://doi.org/10.26656/fr.2017.7(2).696

homogenizer. Then, the liquid ice cream mixture was rapidly cooled at a constant temperature of 4°C overnight for ageing. The aged mixture was then frozen using a home-style batch ice cream machine (Breville, Model BC1600, Sydney, Australia) which has a maximum capacity of 1 L. The ice cream was further hardened and stored in a deep freezer under quiescent freezing conditions at -18°C.

2.2 Preparation of the device

Ice cream samples of approximately 1 mm thickness were prepared and put in a slide box as shown in Figure 1. The box was then placed in a −18°C freezer for 24 hrs until the ice cream matured.

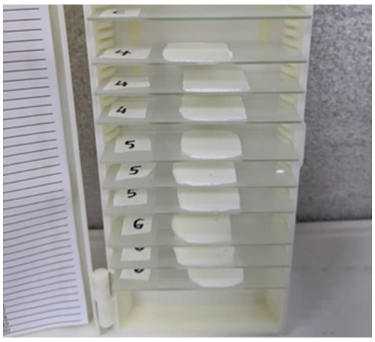

Figure 1. Ice cream samples placed on slides and stored for 24 hrs in -18°C freezer

A digital stereomicroscope (Dino-Lite, Model AM4113, New Taipei City, Taiwan), equipped with a digital video camera and a LED light source providing the episcopic coaxial lighting was positioned inside another freezer (initially set at -40°C). The ice cream sample was then placed on a platform inside the freezer and the microscopic observation was conducted with the freezer door opened. Therefore, the temperature inside the freezer was measured to be −21.5°C which is an optimal temperature for the experiment to be conducted. Images were stored in a laptop located outside the freezer which was connected to the digital microscope via a USB cable. The information is simplified in Figure 2.

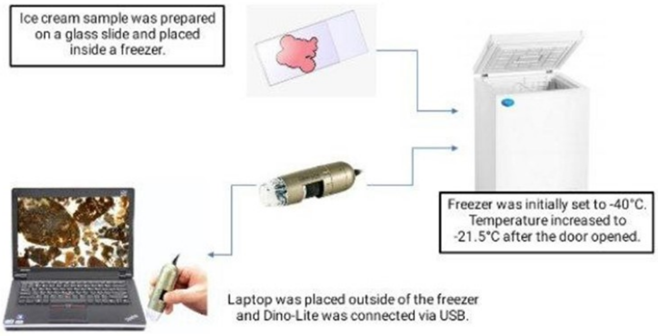

Figure 2. The setup arrangement to observe ice cream microstructure: a glass slide (with approximately 1 mm thick sample on it) and a Dino-Lite microscope were placed inside a freezer, and the microstructure image was recorded in a laptop connected to the Dino-Lite microscope.

2.2.1 Image adjustment

The images obtained were adjusted for a better view of the microstructure. Figure 3 shows the adjustments that the author made to the parameters in DinoCapture 2.0 software that came together with the Dino-Lite device.

This setting was flexibly adjusted based on the

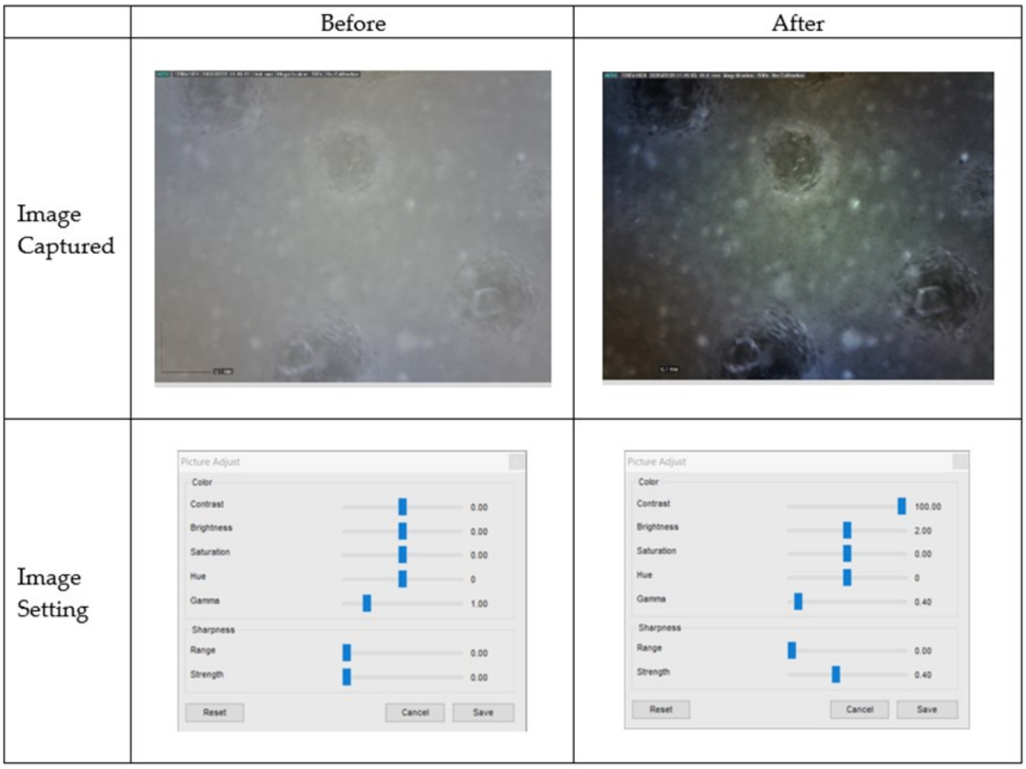

Figure 3. Image adjustment in DinoCapture 2.0 software to obtain a clearer image of the ice cream microstructure

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image obtained. The important part of adjusting the image set was to get the clearest image of the structure by adjusting the contrast and sharpness strength.

2.3 Overrun measurement

In order to relate the microstructure image with physical parameters, ice cream overrun was determined according to the method described by Marshall *et al.* (2003) by using the formula shown in Equation 1:

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

where, M_1 is the mass of the ice cream mixture (g), and M_2 is the mass of the ice cream (g).

2.4 Melting characteristics measurement

The melting rate of the samples was also determined. A hard ice cream block (200 g) was removed from the containers, put on a 1 mm wire screen mesh, and allowed to melt under controlled room temperature. The time required for the dripping of the first drop of melted ice cream was recorded. The weight of the material passed through the screen was recorded at 5 mins time intervals for 120 mins duration. The melting rate was determined as the slope of the graphs of the dripped portion as a function of time and expressed in $g.g^{-1}$ min. A uniform distribution of small ice crystals and air cells results in gradual melting, a phenomenon that can be attributed to a reduced rate of heat transfer, since the trapped air functions as thermal resistance layers (Muse and Hartel, 2004).

3. Results and discussion

3.1 Analysis of the microstructural characterization of ice cream

Figure 4 shows the captured images of the ice cream microstructure for samples with different oil percentages. The ice crystals, air cells, and fat globules are dispersed in a semi-frozen liquid matrix. It can be observed that as the percentage of oil increased, the air bubble radius (*r*) decreased from 0.089 mm (0% oil) to 0.056 mm (6% oil). The curvature (*C*) and area (*A*) of the air cell also decreased with the increase in fat content. During hardening, the sizes of air cells are in the range of 20-60 µm as observed by Warren and Hartel (2014). Wildmoser *et al.* (2004) measured the ice crystal and air bubble sizes using cryo-SEM for ice cream (8% fat) processed using a conventional freezer and the combination of the conventional freezer and low-temperature extrusion (LTE) to be 36 µm and 16 µm,

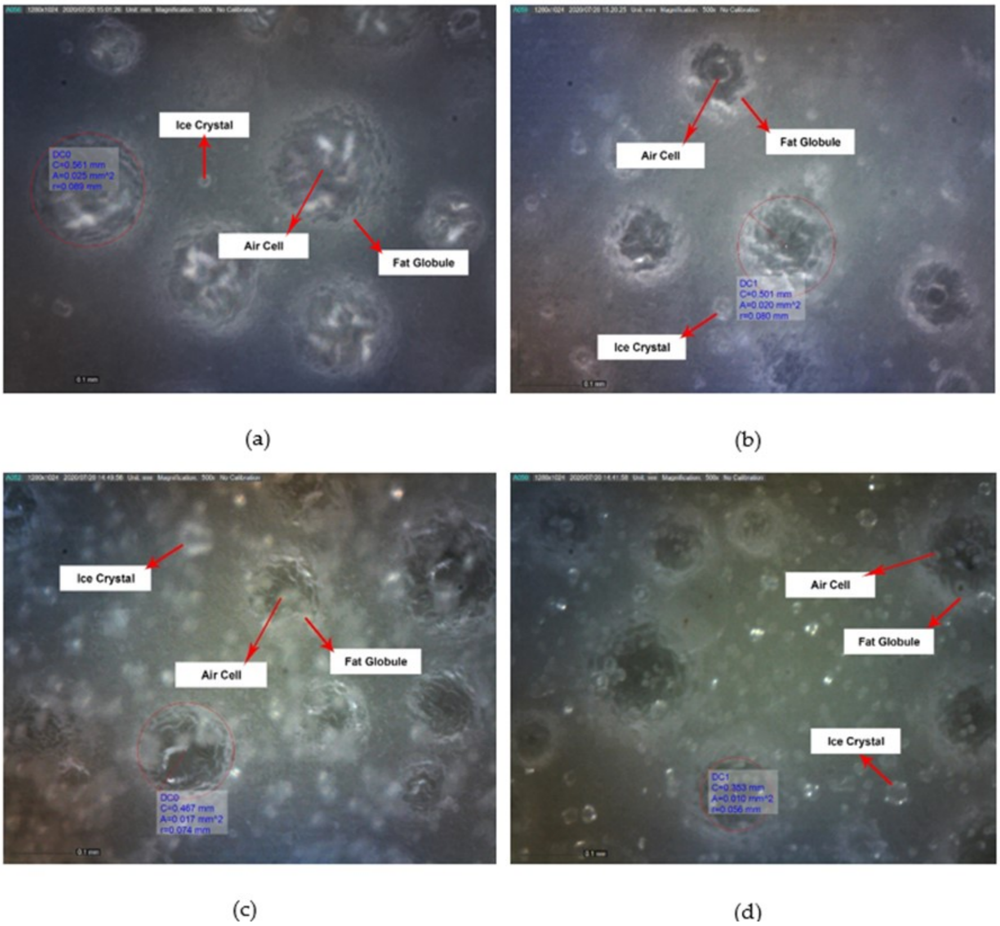

Figure 4. The microstructure of ice cream made with (a) 0%, (b) 4%, (c) 5% and (d) 6% oil contents at 500× magnification. It can be seen that as the percentage of oil content increases, the air bubble size decreases and the distribution of the microstructure becomes more regular.

https://doi.org/10.26656/fr.2017.7(2).696

respectively. This confirms that as fat content increases, air cell size decreases.

It can also be seen that as oil content increased, the ice crystals' size decreased. At freezing temperatures, the viscosity of ice cream is high. Higher fat content increases the viscosity of the ice cream mixture during processing (Adapa *et al.*, 2000). As temperature decreases, the texture becomes denser and it would be difficult to incorporate air inside the mixture as the temperature goes down towards the end of the process. Hence, the mixture with high viscosity holds air better than the one with lower viscosity (low-fat content). Even though the overrun is lower, the air bubbles are more stable in ice cream with higher fat content. Fat globules attach to the air cell to stabilize it, while ice crystals are distributed in the matrix. The air cells, ice crystals, and fat globules inside the ice cream with 6% oil have a regular distribution compared to the other samples. A study by Fiol *et al.* (2017) captured a more regular distribution of microstructure in ice cream produced using sunflower oil compared to the one produced without oil.

Figure 5 shows the correlation between the percentage of oil contents in the samples with the respective radius of the air cell in the microstructure. A negative parabolic relationship was obtained between the percentage of oil content and the air cell size with an R^2 of 0.979.

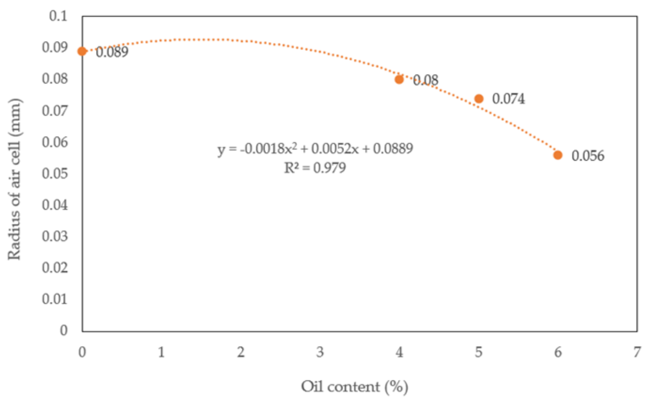

Figure 5. Correlation between oil content (%) and radius of air cell in ice cream microstructure. The radius of the air cell decreases as the oil content increases.

3.2 Analysis of overrun of ice cream

Air content in ice cream influences the melting rate of ice cream. Figure 6 shows the overrun of the ice cream samples. Overrun was known as the amount of air added in ice cream by comparing the mass of a fixed volume of ice cream mix before and after the ice cream undergoes the freezing process. From the bar graph, it can be seen that the control sample (0% oil) had the highest overrun which was 55.12% compared to the other samples. Samples with 4%, 5% and 6% oil contents had overrun of 38.10%, 35.82% and 33.38%, respectively. It can be seen that the overrun decreased as the oil content increased. Apart from fat content, the use of stabilizers in ice cream mix led to increased viscosities which in turn resulted in lower overrun but more stable foam (Stanley *et al.*, 1996).

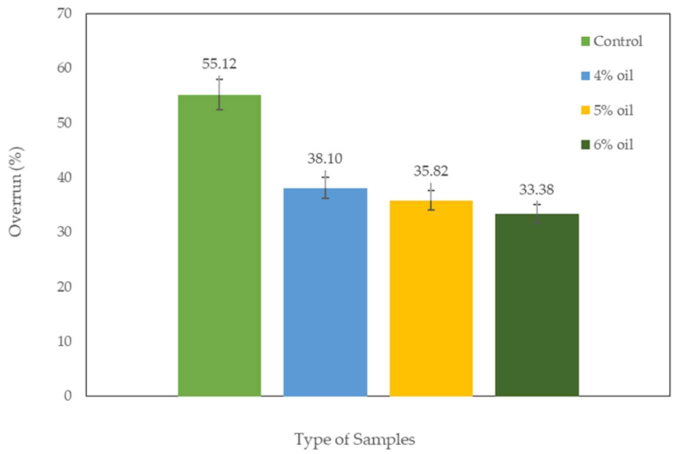

Figure 6. The decrease in the overrun of ice cream samples as the oil content increases.

Figure 7 shows the correlation between the percentage of oil contents in the samples with the respective overrun. Overrun decreased proportionally with oil content (R^2 = 0.9859). This reflected the bigger air cell size in ice cream with less fat content.

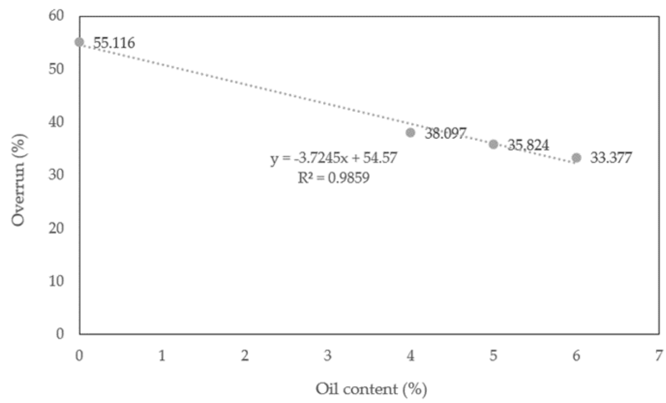

Figure 7. Correlation between oil content (%) and overrun of ice cream. The overrun decreases as the oil content increases.

3.3 Analysis of the melting rate of ice cream

The melt-down rate of ice cream is affected by many factors, including the amount of air incorporated, the nature of the ice crystals, and the network of fat globules formed during freezing (Parid *et al.*, 2021). The melt-down rate involves the relationship between the mass of the melted ice cream with time. Figure 8 shows the melting rate of ice cream with different oil contents. The results showed that the ice cream prepared using 0% oil had the fastest melting rate, while the sample made using 6% oil content had the slowest melting rate.

Ice cream that has high-fat content tends to melt slower. Roland *et al.* (1999) analyzed ice cream

https://doi.org/10.26656/fr.2017.7(2).696

formulated with various fat percentages and found that high-fat content reduces the melting rate. This has been verified by Alamprese *et al.* (2002) who reported a softer ice cream with higher fat content and a slower melting rate. Hyvonen *et al.* (2003) found similar results and reported that a slightly retarded melting of ice cream in the mouth was due to the increase in fat content.

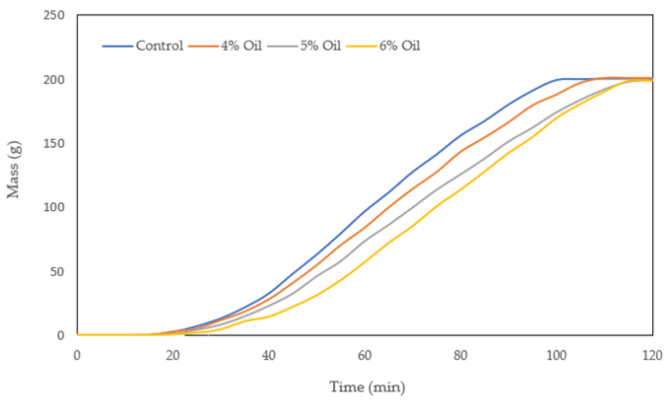

Figure 8. Effect of oil content on the melt-down rate of ice cream. Ice cream with higher oil content has a slower melting rate than the ones with lower oil content.

During dynamic freezing, fat undergoes partial coalescence or destabilization. Partial coalescence occurs when a protruding fat crystal from one fat globule pierces another globule's interfacial film, creating a largely irreversible connection between the inner phases of the globules (Walstra, 2003). These partly coalesced globules are essential to the production of smooth texture and meltdown resistance (Goff, 1997). Meltdown levels typically decrease as fat destabilization grows (Muse and Hartel, 2004; Goff and Hartel, 2013). Hence, higher fat content leads to more partially coalesced globules that reduce the melting rate. This also reflects the better distribution of particles in the ice cream microstructure of ice cream with 6% oil content as shown in Figure 4 (d). Figure 9 shows the correlation between the percentage of oil contents in the samples with the respective melting rate. The melting rate decreased

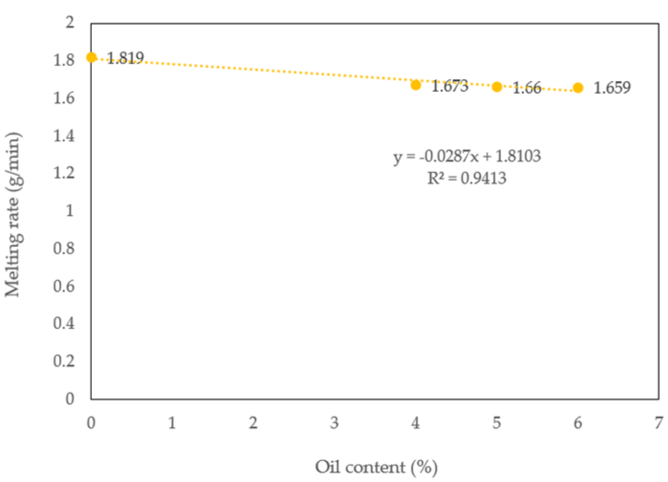

Figure 9. Correlation between oil content (%) and melting rate of ice cream. Ice cream with higher oil content has a slower melting rate.

linearly with oil content (R^2 = 0.9413). Altering the formulations change the microstructure of ice cream which leads to various meltdown behaviours. The level of fat destabilization, ice cream mixture viscosity, and overrun was found to affect meltdown (Amador *et al.*, 2017).

4. Conclusion

Observing the microstructure of ice cream requires efficient equipment with proper handling and technique due to the temperature sensitivity of ice cream. This study managed to set up a device arrangement and method to capture the microstructure of ice cream. The commissioning of the arrangement was conducted by evaluating the ability of the microscopic device arrangement to capture ice cream made from a different percentage of oil contents i.e., 0%, 4%, 5%, and 6%. Results showed that different percentages of oil content in ice cream affected the microstructure of ice cream i.e., the air cells, ice crystals, and fat globules. It was observed that ice cream with a higher percentage of oil content had smaller air cells and ice crystal sizes with clearly visible fat globules. A parabolic correlation was obtained between the percentage of fat content and the air cell size with an R^2 of 0.979.

Ice cream with lower fat content had higher overruns and vice versa. The overrun decreased linearly with fat content (R^2 = 0.9859). Ice cream with higher fat content melts slower due to more partial coalescence between fat globules occurring which creates extra meltdown resistance. The melting rate decreased linearly with oil content (R^2 = 0.9413). Overall, images of the microstructure of ice cream were successfully obtained using the device developed and the physical properties were well correlated. The device and method developed are reliable in characterizing the microstructure of ice cream, which helps researchers and small business entrepreneurs to understand the fundamental relationship between the ingredients in ice cream with the microstructure of ice cream, and consequently towards its physical quality. The device arrangement setup is simple, requires less cost, and its usefulness could be extended to other frozen product characterization.

Conflict of interests

The authors declare no conflict of interest.

Acknowledgements

The authors were grateful for the support from the laboratory staff who helped in handling the equipment for the analyses involved in the research. This study was conducted in the laboratory of the Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia.

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