Rheological Evaluation of the Structure of Ice Cream Mixes Varying Fat Base

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Abstract:
This paper discusses the influence of fat type in the structure of ice cream, during its production by means of rheo-optical analysis. Fat plays an important part in the ice cream structure formation. It's responsible for the air stabilization, flavor release, texture and melting properties. The objective of this study was to use a rheological method to predict the fat network formation in ice cream with three types of fats (hydrogenated, low trans and palm fat). The three formulations were produced using the same methodology and ratio of ingredients. Rheo-optical measurements were taken before and after the ageing process, and the maximum compression force, overrun and melting profile were calculated in the finished product. The rheological analysis showed a better response from the ageing process from the hydrogenated fat, followed by the low trans fat. The formulation with palm fat showed greater differences between the three, where through the rheological tests a weaker destabilization of the fat globule membrane by the emulsifier was suggested. The overrun, texture measurements and meltdown profile has shown the distinction on the structure formation by the hydrogenated fat from the other fats.

Zusammenfassung:
In diesem Artikel wird der Einfluss eines Fett-Typs in der Struktur von Eiscreme während der Herstellung mit rheo-optischen Methoden untersucht. Fett spielt eine wichtige Rolle bei der Bildung der Struktur von Eiscreme. Es ist verantwortlich für die Stabilisierung an Luft, die Geschmacksabgabe, die Textur und die Schmelzeigenschaften. Das Ziel dieser Untersuchung war, eine rheologische Methode anzuwenden, um die Bildung von Fettnetzwerken in Eiscreme anhand drei unterschiedlicher Fett-Typen zu erforschen (hydrogeniert, Magertransfette und Palmenfett). Diese drei Formulierungen wurden mit derselben Methode und derselben Zusammensetzung hergestellt. Rheo-optische Untersuchungen wurden vor und nach der Alterung durchgeführt. Die maximale Druckkraft, das Überlaufen und das Schmelzprofil bei dem hergestellten Produkt wurden bestimmt. Die rheologische Analyse wies eine bessere Antwort bzgl. des Alterungsprozesses des hydrogenierten Fettes auf, gefolgt von dem Magertransfett. Die Formulierung mit dem Palmenfett wies größere Unterschiede zwischen den drei auf. Durch die rheologischen Messungen wurde eine schwächere Destabilisierung der Fettglobul-Membran durch den Emulgator angedeutet. Das Überlaufen, die Texturmessungen und das Aufschmelzprofil zeigten die Unterschiede der Strukturbildung der hydrogenierten Fette im Vergleich zu den anderen beiden Fetten.

Résumé:
Cet article discute l'influence du type de graisse sur la structure de la crème glacée, lors de sa production, au moyen d'une analyse rhéo-optique. La graisse joue un rôle important dans la formation de la structure de la crème glacée. Elle est responsable de la stabilisation de l'air, de la libération de l'arôme, et des propriétés de texture et de fondant. L'objectif de cette étude est d'utiliser une méthode rhéologique afin de prévoir la formation du réseau de graisse dans la crème glacée pour trois types de graisse (graisse hydrogénée, de palme et de trans bas). Les trois formulations ont été produites en utilisant la même méthodologie et le même ratio d'ingrédients. Les mesures rhéo-optiques ont été effectuées avant et après le processus de vieillissement, et la force de compression maximum et les profils de fusion ont été calculés pour les produits finis. L'analyse rhéologique présente une meilleure réponse pour le processus de vieillissement associé à la graisse hydrogénée, suivi de la graisse trans bas. La formulation avec la graisse de palme présente la plus grande différence entre les trois types, et les tests rhéologiques suggèrent pour cette graisse une déstabilisation plus faible de la membrane globulaire de la graisse par l'émulsifiant. Les mesures montrent une distinction de la graisse hydrogénée par rapport aux autres à propos de la formation de la structure.

Key words: food rheology, structure, ice cream, palm fat, low trans fat

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1 INTRODUCTION

The technological advances in the food industry gave rise to the hydrogenated vegetable fat. The ice cream industry has used this fat in the manufacture of its products for the effects that provides to the texture and its low cost. After trans fatty acids have been included among dietary lipids that act as risk factors for health, fat industry have sought to replace the use of hydrogenated vegetable fat in their products. Many studies have been made to find substitutes for these fats with low levels of trans fatty acids. The challenge has been to make this change without the loss in sensory and appearance quality, items essential to the final consumer.

The importance of fat in the formation of structure and the physical and sensory aspects of the ice cream is widely recognized today. It has an essential role in the texture and flavor, which are the main aspects to consider for its success in the market. It is present in the mixture as a fine emulsion that partially coalesces during the freezing process [1]. The ageing process of an ice cream production is of great importance to the fat phase where many changes occur in the emulsion that will determine the formation of the structure of the fat network [2]. During this process, the emulsifier displaces the protein in the fat globule membrane, allowing the fat to suffer a better partial coalescence during freezing. The crystallization of fat also takes place during this step which also influences the fat network formation. When choosing a replacement for milk fat or hydrogenated fat, the structure, the profile during dynamic conditions of crystallization temperature, melting profile, content of triacilglicerols of high melting point, taste and purity should be researched [3, 4].

One of the candidates as a substitute for hydrogenated fat is the palm fat (*Elaeis guineensis*), which is 100 % natural and have gained its place in the market by having a melting point around 40°C, requiring no changes through processes of transformation, which makes it extremely versatile. The refining is done in a natural way, is highly stable to oxidation and contains about 50 % of saturated fatty acids, 40 % monounsaturated and 10 % polyunsaturated. It is free of trans fatty acids because it has not been subjected to the hydrogenation process [5]. Another solution found by the industry to reduce trans fatty acid content of vegetable fats, was by chemical or enzymatic interesterification of a mixture of fats and / or liquid or fully hydrogenated vegetable oils. This process has been much used and is usually done with mixtures of highly saturated fats with liquid oils to produce fats with intermediate characteristics.

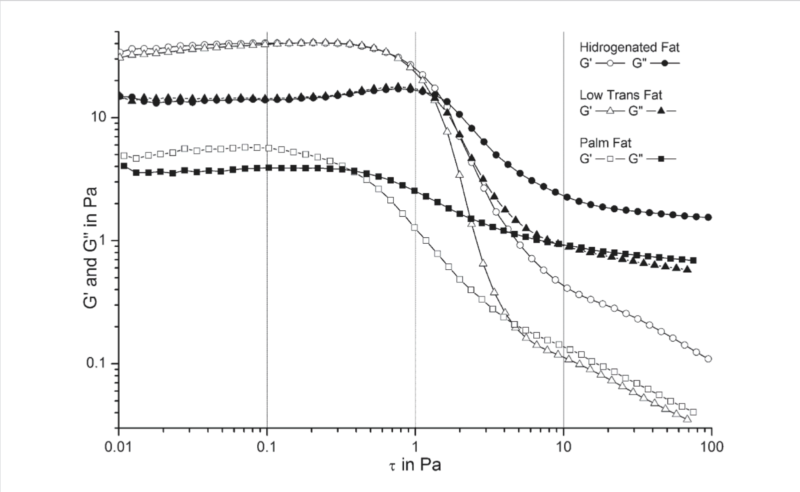
In a study of ice cream formulations using different types of fat, rheology provides information to analyze the effect of each studied fat in the ice cream structure. In evaluations of oscillatory rheology, the material deformation is low, where the structure remains intact. In those tests it is possible to evaluate both viscous parameters of liquids as elastic parameters of solids. The knowledge of the rheological behavior of foods is useful not only to control the product quality, but mainly for the design of piping systems, heat exchangers, filters, pumps, among others. It allows you to discuss the structure and composition of the tested food, since the rheological behavior is dependent on these factors [6]. Oscillatory rheology has been used to study the structure of food emulsions like mayonnaise before [9]. This method shows great potential to study not only the finished food product, but to study food during its processing, analyzing the transformations occurring in its structure.

The aim of this paper is to study the structure of ice cream mixes as well as the production of ice cream varying the type of fat in the formulation, and to use rheo-microscopy to analyze the ageing process of ice cream production to predict the ice cream structure formation in the freezing/aeration process.

2 MATERIALS AND METHODS

2.1 COMPOSITION AND PREPARATION OF SAMPLES

Three formulations were prepared varying the fat base. The formulations contained 12 % sucrose (União), 14 % milk powder (Itambé), 4 % dried glucose (CornProducts – MorRex 1940), 0.5 % emulsifier/stabilizer (Prozin – Palsgaard 5924) and 6 % fat base, varying from hydrogenated vegetable fat (Bunge – ProDulce 37), low trans fat (Bunge ProMult 44LT) and palm fat (Agropalma 370SE). The emulsifier/stabilizer used contains mono and diglycerides of fatty acids E-471, carboxymethyl cellulose sodium chloride E-466, guar gum E-412 and carrageenan



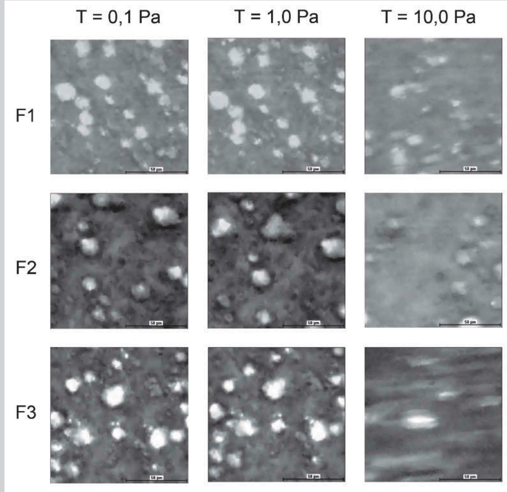


Figure 1 (left):
Stress amplitude sweep test results of ice cream samples at 10°C before the ageing process.

Figure 2:
Microscopic images (20x) of the ice cream samples during the stress amplitude sweep test under different stresses (F1 = Formulation with hydrogenated vegetable fat, F2 = Formulation with low trans fat, F3 = Formulation with palm fat).

E-407. All fats used are commercial fats suitable for ice cream.

The production was made in pilot scale. After weighting the ingredients, they were mixed and pasteurized at 75° C for 15 minutes under constant homogenization at 1200 rpm using a mixer (Fisaton 713D, Brazil). The mixture was cooled and stored in a refrigerator at 10°C for the ageing process for 20 hours. The mixture was then frozen and aerated in a vertical ice cream producer (Conservex/Skysen, Brazil) with a cooling bath of - 25° C for 10 minutes, until the ice cream reached a temperature of - 6° C. The ice cream was packed and hardened at a - 28° C freezer for 2 hours before it was stored at a - 20° C freezer.

2.2 RHEOLOGY

The rheological properties of samples were determined by oscillatory tests. The tests were performed on a MARS rheometer (Thermo Fisher Scientific-Haake, Germany) at 10° C to simulate the temperature of the refrigerator used in the ageing process. The cone-plate sensor (C35 / 1 Ti polished) with a 0.024 mm gap was used. Analysis were performed at least in triplicate, with a stress amplitude sweep ranging from 0.01 to 100 Pa, with a constant frequency of 1 Hz and frequency sweeps varying from 0.1 – 10 Hz, constant stress of 0.1 Pa. The micro - structural deformations were followed by an optical microscopy system coupled to the equipment (Rheoscope module) and images were captured by a CCD camera. The sample placed on the plate of the equipment was enough to cover an area of 2 cm². The amount of sample used was standardized with a plastic dropper ranging between 5 to 7 drops depending on the sample. Analysis were performed in two stages, immediately after preparation of the mixture before the ageing process and 20 hours later, after ageing.

2.3 TEXTURE

The extrusion force determination of the formulations was carried out in a TA-XT2 (Stable Micro System, UK) with the AB/E back extrusion probe (4 cm piston diameter). The tests were performed in triplicate for all formulations with a penetration probe speed of 2 mm/s and a covered distance of 15 mm. The characteristic curves were generated in the program "Texture Expert Exceed" – version 2.6, where the maximum force required to extrude the samples of all the ice cream formulations were determined. The tests were performed soon after the freezing and aeration process, where the mass temperature was at - 5.5° C. The cup used in the test was filled to about 1 inch from the top for all samples.

2.4 OVERRUN

To determine the overrun a defined volume of the mixture was weighed after the ageing process in a 10 mL glass beaker. Soon after the freezing and aeration process of the ice cream, the same volume was weighed in the same glass beaker. The overrun was calculated using the equation proposed by Goff & Hartel [7] as shown in Equation 1:

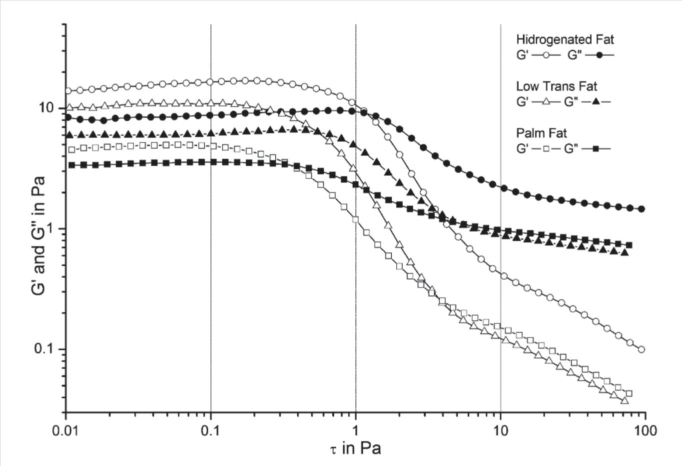
$$\%_{overrun} = \frac{m_{mixture} - m_{Ice\ creme}}{m_{mixture}} \quad (1)$$

2.5 MELTING TEST

Samples weighing 100 ± 1 g stored at - 20° C were placed on a metal grid for 45 minutes at controlled room temperature of 25 ± 0.5° C. The weight of the dripped portion of the samples was recorded every 5 minutes.

2.6 STATISTICAL ANALYSIS OF DATA

The mean values and curves from the rheological tests were calculated using the program Ori-



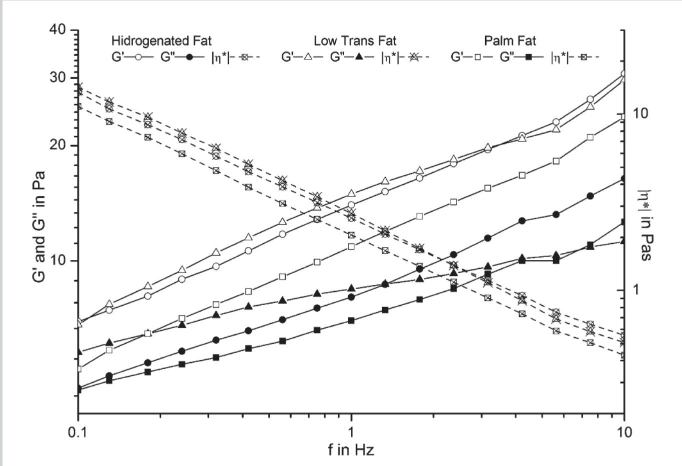


Figure 3 (left): Stress amplitude sweep test results of ice cream samples at 10°C after 20 hours of ageing process.

Figure 4: Frequency sweep test results of ice cream samples at 10°C after 20 hours of ageing process.

gin version 8. The data obtained in the texture analysis were statistically analyzed using the program Microsoft Excel 2007 and determined by the Tukey test (with a significance level of 5 %) with the Statistica v.6 Software.

3 RESULTS AND DISCUSSION

3.1 RHEOLOGY

The rheological analysis showed that all samples had a predominance of elastic behavior over the viscous behavior ($G' > G''$) within its linear viscoelastic region (LVR) (Figure 1). The formulations made with hydrogenated fat and low trans fat showed similar curves until the LVR indicating a similar structural behavior. The formulation made with palm fat showed lower values of G' and G'' when compared with the other fats. It also showed structural change to a lower stress, the same results found by Silva Junior & Lannes to chocolate ice cream [8]. The effect of stress on the structure of the formulations can be seen in Figure 2, where a structural deformation can only be seen at a tension of 10 Pa. The structural changes observed through the results with the palm fat formulation at 1,0 Pa could not be noted visually through the microscopy.

Generally, a longer LVR indicates a weakly flocculated and stable dispersion [9]. The longer LVR observed in the formulations of hydrogenated and low trans fats can be explained due to their β' type of crystallization, which forms smaller crystals than β form. The hydrogenated fat particularly favors the occurrence of β' type crystals, due to its trans fatty acid content. The presence of a percentage of the trans isomer affects the fat crystallization kinetics, which assists and directs the formation of crystals in the β' form even when the natural tendency of crystallization of the fat is β [10]. The presence of β' crystals leads to a higher viscoelasticity in the product as smaller crystals allow greater incorporation of liquid oil, increasing the elasticity [11]. The palm fat shows polymorphism in its crystallization profile [12], making it possible that at the moment of the ageing process there could be a mixture of crystals in the α, β' and β form. For those reasons, the formulation with palm fat showed a smaller LVR, associated with the fact that the crystals in the palm fat tend to agglomerate and form large round or spherical crystals, which reduce the interaction of the crystals formed with the liquid fraction [13].

After ageing, the three formulations kept the characteristics of viscous modulus prevalence, as observed by Bazmi, et al. [14]. The hydrogenated fat showed the most resistant structure from the 3 formulations (Figure 3). The formulation with hydrogenated fat, just as the low trans fat showed an increase in the loss factor value (tan δ) after ageing, indicating that the ratio between the elastic and viscous modules has decreased, where the formulation of palm fat, the elastic and viscous modules did not show major changes after the ageing process. The palm fat presents a profile of slower crystallization [15], and it requires a longer ageing time than the other formulations for the crystallization of its fat. The highest values observed for the formulation of hydrogenated fat can be attributed to a faster crystallization due to the presence of trans fatty acids that tend to crystallize more rapidly than its counterpart, the cis isomer, explaining the difference found with the low trans fat [16]. It was also suggested that the palm fat globules suffered less destabilization on its membrane surface from the emulsifier when compared with the use of hydrogenated vegetable fat.

From the frequency sweep test (Figure 4), we could see that all formulations showed a drop in the curve of complex viscosity and an increase in both elastic and viscous modulus with an increase in frequency within the tested parameters, attributing the samples a typical behavior

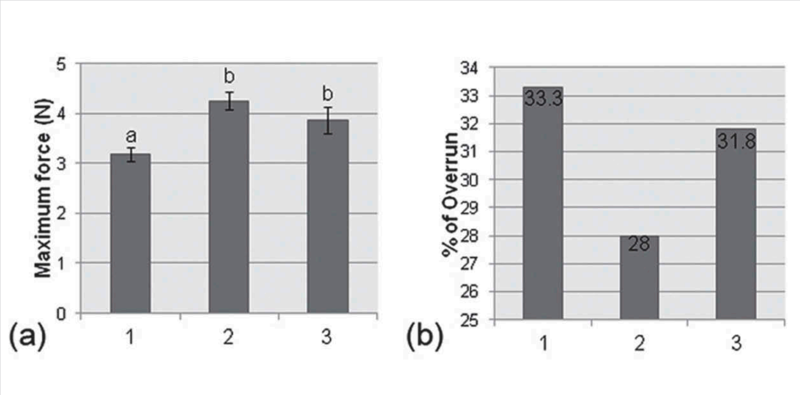


Figure 5:
Results from texture and overrun analysis to ice cream formulations produced with: 1 = Hydrogenated Fat, 2 = Low Trans Fat, and 3 = Palm Fat Error bars indicate standard errors; same letters indicate values that do not differ statistically (α = 0.05), different letters indicate values that do differ statistically (α = 0.05).

of pseudoplastic materials [17]. The presence of small flakes of fat is responsible for the resistance to flow at low shear stresses [14]. Previous studies already shown that ice cream demonstrate a viscoelastic behavior due to its microscale aspects of its structure [18]. Among the formulations analyzed, the one which contained hydrogenated fat showed the highest viscosity values. The fact could be attributed to the β' crystals, which leads to a higher viscosity due to their small crystal size and greater surface area that should also lead to a greater interaction between particles [11]. The formation of large round/spherical crystals found in the palm oil, also accounts for the lower viscosity found in the formulation produced with that oil.

After the ageing period, the elastic and viscous modules of the formulations with hydrogenated fat and low trans fat showed a decline, indicating to be more fragile under stress. The destabilization of the interfacial membrane of fat globules by the action of the emulsifier, which occurs during ageing, may have caused the decrease in the parameters. The formulation with hydrogenated fat had the greatest changes in its parameters after ageing while the palm fat had the smallest changes. This reinforces the theory that the palm fat suffers less destabilization of the membrane. The palm fat has a large number of palmitic acid (16:0) ranging from 35 to 47 % [19]. This high percentage of saturated fatty acids is disadvantageous to the destabilization of fat globules, since the more unsaturated and the longer are the chains of vegetable fat used in the formulation, the more pronounced will be the destabilization of fat. In addition, emulsifiers mono and diacilglicerol can interact better through the hydrophobic region of molecules, where the palm fat has less hydrophobic regions when compared with other vegetable fats used in the hydrogenated fat [20]. In a study conducted by Bazmi et al. [21] it is shown that the destabilization effect is more significant in emulsions containing a lower content of saturated fat in the triacilglicerol.

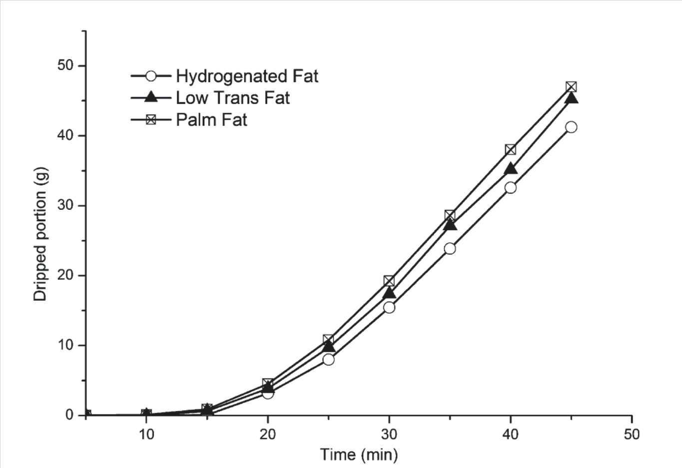
3.2 TEXTURE

According to the data obtained from the texture analysis, it can be observed that the formulation with hydrogenated vegetable fat has a value of maximum force required for extrusion of 3.19 ± 0.13 N, the value was lower than those found for formulations produced with low trans fat of 4.25 ± 0.18 N and palm fat of 3.87 ± 0.27 N (Figure 5). This indicates that upon completion of the process of freezing/aeration, the formulations with low trans fat and palm fat had a firmer texture. The formulation with low trans fat was found to be more resistant to extrusion of all three. The presence of the large round/spherical crystals could explain the firmness found in the formulation of palm fat [13].

3.3 OVERRUN

Among the samples tested, the highest overrun was observed in the formulation made with hydrogenated fat. The formulation with low trans fat resulted in less incorporation of air (Figure 5b). The low value of overrun presented by the formulation with low trans fat and palm fat could explain the harder texture found for the ice cream made with these fats because, as noted by Sofjan & Hartel [22], a decrease in overrun causes an increase in the firmness of ice cream. The fat industry has been working on the development of low trans fat formulations that have the same technological performance that hydrogenated fat. The lower overrun associated with the formulation of palm fat could also be explained by the lesser destabilization of fat globules, which does not allow the formation of a stable fat network, hindering the stabilization of air bubbles incorporated into the mass of ice at the time of freezing/aeration.

Although the results of the rheological tests suggested that the formulation produced with palm fat had a worse response from the ageing process, it had better results from the texture and overrun analysis when compared with the low trans fat. Sung & Goff [23] analyzed ice creams with different solid fat contents and noted that a 55 % solid fat content at 5° C was better than a 60 – 70 % solid fat content for the fat globule membrane destabilization but the overrun was lower. That could happen due to oil wetting and spreading at the moment of freezing/aerating process, which could compromise foaming sta-



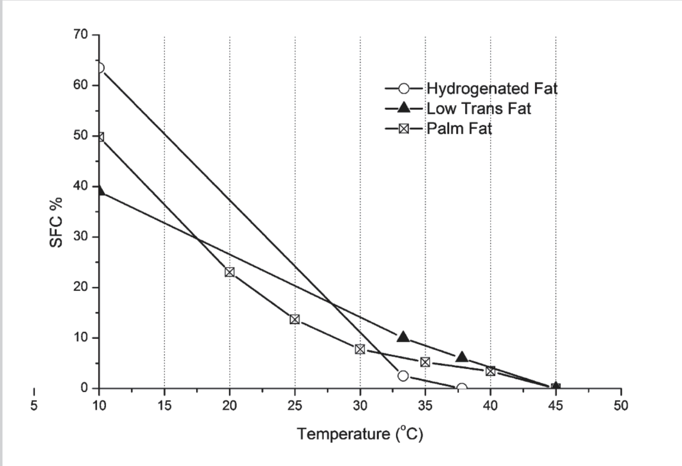


Figure 6 (left): Meltdown curves of ice creams with different fats.

Figure 7: Solid fat content of the tested fats.

bilization. The low trans fat used in this experiment had a lower solid fat content at 5° C than the palm fat, which could explain the lower overrun observed.

3.4 MELTING TEST

The melting profile allows us to evaluate the fat network formation since the fat globule aggregation by partial coalescence seems to be the largest contributor to the ice cream melting resistance. [23]. As the ice crystals melt and the ice cream structure starts collapsing, it's expected the fat network to keep its form during the meltdown [24]. The melting curves of each formulation can be observed in Figure 6, in which the hydrogenated fat showed the slowest melting rate and palm fat the fastest melting rate. As it was seen in the rheological results, the hydrogenated fat presented the best results to the ageing process, which would allow the formation of a better fat network through partial coalescence. The presence of small crystals is also responsible for a better fat network formation since it causes a higher emulsification which is related to a higher partial coalescence [11, 24]. The meltdown is also influenced by the solid fat content of each fat. Palm fat has the lowest solid fat content at the room temperature tested (25 ± 0.5° C) as can be seen in Figure 7.

4 CONCLUSION

All data obtained from the rheological analysis with images suggested that palm fat suffers a lower destabilization of fat globules by the emulsifiers during ageing, with hydrogenated vegetable fat presenting the best results to this parameter. The hydrogenated fat shows less firmness and greater overrun on ice cream formulations when compared to formulations with low trans fat and palm fat. In this experiment, palm fat and commercial low trans fat could not substitute with the same pattern the hydrogenated fat in ice cream manufacture while maintaining its beneficial properties to the product. Further studies must be done to achieve an ideal substitution, in particular the interaction between palm fat and the emulsifier. The rheological analysis during the ice cream production proved to be useful to predict the final characteristics of the ice cream since the results from the overrun, melting test and textural measurements agreed with the findings of the rheological analysis.

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