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Enrichment of ice cream with dietary fibre: Effects on rheological properties, ice crystallisation and glass transition phenomena

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ABSTRACT

In the present study, the effects of four dietary fibre sources (oat, wheat, apple and inulin) on the rheological and thermal properties of model sucrose–polysaccharides solutions and ice cream mixes were investigated. The content of fibre in insoluble compounds increased significantly the viscosity and the shear thinning behaviour of the model solutions and ice creams, due to the increase of total solids and the formation of networks comprised of hydrated cellulose and hemicellulose. The increase of soluble material did not alter significantly the rheology of the samples but limited the freezing point depression and elevated the glass transition temperatures, indicating a potential cryoprotective action. The use of oat and wheat fibre favoured viscosity development due to water-binding, whereas inulin caused a remarkable increase of glass transition temperature (T_g) in model solutions and ice cream mixes, indicating the reduction of water molecule mobility from the bulk aqueous phase to the ice crystals' surface. Apple fibre addition greatly increased viscosity and elevated the T_g values, particularly in the presence of proteins. Thus, our results suggest the potential use of dietary fibres as crystallisation and recrystallisation phenomena controllers in frozen dairy products.

1. Introduction

The term dietary fibre is referred to polysaccharides, oligosaccharides and their hydrophilic derivatives, which cannot be digested by the human digestive enzymes, to absorbable components in the upper alimentary tract (Thebaudin, Lefebvre, Harrington, & Bourgeois, 1997). Chemically defined, dietary fibres include a group of heterogeneous substances such as celluloses, hemicelluloses, lignins, pectins, and seeweed or bacteria-derived gums. A large number of studies dealing with the physiological and nutritional aspects of dietary fibre has led to their incorporation in a great number of food products, such as bakery, breakfast cereals, baby foods, meat products, pasta and yogurts (Gelroth & Ranhotra, 2001, chap. 23).

The physiological actions promoted by fibre addition in foods include the maintenance of gastrointestinal health, reduction of intestine transit time, protection against colon cancer, lowering of total and low-density lipoprotein cholesterol in the blood serum, reduction of postprandial blood glucose levels, increase of calcium bioavailability and reinforcement of the immunological system (Thebaudin et al., 1997; Tungland & Meyer, 2002). The recommended daily intake for total fibre for adults has been set at 38 g for men and 25 g for women (Trumbo, Schlicker, Yates, & Poos, 2002).

Dietary fibres can provide a multitude of functional properties when they are incorporated in food systems. Thus, fibres addition contributes to the modification and improvement of the texture, sensory characteristics and shelf-life of foods due to their water-binding capacity, gel-forming ability, fat mimetic, antisticking, anticlumping, texturising and thickening effects (Dello Staffolo, Bertola, Martino, & Bevukaqcua, 2004; Gelroth & Ranhotra, 2001; Thebaudin et al., 1997, chap. 23). The type, as well as the extent of the furnished functional effects is undoubtedly related to the fibres plant origin, the insoluble to soluble fibre ratio, the fibre–fibre synergy and interactions with other food components.

Ice cream is a complex colloidal frozen system that consists of partially coalesced fat droplets, air cells, ice crystals and a continuous aqueous phase, in which the polysaccharides, proteins, lactose and mineral salts are dispersed (Marshall, Goff, & Hartel, 2003). Due to the thermodynamic instability of the ice cream, recrystallisation phenomena occur during storage, leading to the gradual increase of the ice crystals mean size and the deterioration of the product quality characteristics (Regand & Goff, 2003). As a common practice, recrystallisation is effectively controlled by the addition of hydrocolloids, due to their ability to control water diffusion from the serum to the ice crystal interface or form cryogels, which diminish water mobility (Goff, Ferninando, & Schorsch, 1999; Hagiwara & Hartel, 1996; Regand & Goff, 2003). Moreover,

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hydrocolloids also contribute to the improvement of textural, melting and flavour characteristics of ice cream (Soukoulis, Chandrinos, & Tzia, 2008; Soukoulis & Tzia, 2008; Marshall et al., 2003; Baer, Krisnawamy, & Kasperson, 1999). Though stabilisers may be defined as dietary fibre, due to their polysaccharide origin, the low levels at which they are added in frozen dairy desserts does not allow them to furnish the desired physiological and nutritional effects in ice cream. On the other hand, dietary fibres due to their water-binding properties and gel-forming ability, could promote the effective control of ice crystallisation and ice crystals' growth during freezing and storage, respectively.

There are little data dealing with the study of the functionality of dietary fibres in ice creams. The use of citrus fibre has led to significant improvement of melting quality of ice cream but failed to improve viscosity, overrun and texture (Dervisoglu & Yazici, 2006). In a similar study, the addition of rice flour was found to be a satisfactory fat replacer, though it imparted a powdery mouthfeel (Cody, Olabi, Pettingell, Tong, & Walker, 2007). However, there are limited data concerning the impact of dietary fibre on ice crystallisation and rubbery to glassy state transition phenomena.

The aim of this study, was to evaluate the effects of four types of dietary fibre on the rheological properties, ice formation and glass transition phenomena of ternary sucrose–polysaccharides–fibre aqueous model systems and fibre-enriched ice cream mixes.

2. Materials and methods

2.1. Ice cream mixes preparation

The composition of the ice cream mix was 6% fat, 11% milk solids non fat (MSNF), 16% sugar solids, 0.2% stabiliser, 0.2% emulsifier and 2 or 4% dietary fibre. The types of dietary fibre used in the study were oat fibre (VITACEL 201, 3% soluble fibre, 93% insoluble fibre (70% cellulose and 25% hemicelluloses), water-binding capacity (WBC) 8.0 g H_2O/g of fibre, JRS, Rosenberg, Germany), wheat fibre (VITACEL 201, 3% soluble fibre, 93% insoluble fibre (70% cellulose and 24% hemicelluloses), WBC 8.0 g H_2O/g of fibre, JRS, Germany), apple fibre (VITACEL 401, 15% soluble fibre of which 9.3% pectin, 45% insoluble fibre, WBC 5.0 g H_2O/g of fibre, JRS, Germany) and long chain inulin (FIBRULINE XL, 90% soluble fibre, Cosucra, Warcoing, Belgium). For the ice cream mix preparation stabilisers (guar gum) (Danisco, Grindsted, Denmark) and microcrystalline cellulose (Avicel, FMC Biopolymers, Drammen, Norway), added at 1:1 ratio), emulsifier (mono-diglycerides of fatty acids, 60% monoester content, Rikemal P-150S, Rikevita, Malaysia), MSNF (skim milk powder, Epiim S.A., Latvia) and dietary fibres were blended with sucrose (Hellenic Sugar Industry, Larissa, Greece), then dispersed under agitation (at 1000 rpm) into the liquid materials (cream, 35% milkfat, and full-fat fresh milk (3.5% milkfat), Fage SA, Athens, Greece) at 50 °C for 10 min using a mechanical stirrer (Ika-Werke GmbH, Konigswinter, Germany). The mix was then batch pasteurised at 76 °C for 20 min using a water bath and consequently two-stage (200 and 30 bar, respectively), two-fold homogenised using a laboratory single piston homogeniser (APV Gaulin, Abvertslund, Denmark). Then, the ice cream mixes were rapidly cooled at 4 °C and remained at constant temperature for 18 h to be aged.

2.2. Sucrose model systems preparation

In order to investigate and evaluate the individual effects of fibre addition on the rheological and thermal properties of ice cream mixes, sucrose–polysaccharides model systems were prepared based on the composition the ice cream mixes (16% sucrose, 2% or 4% fibre and 0.2% stabiliser). Sucrose, polysaccharides blend and fibres were dispersed in distilled water at 40 °C for 20 min to achieve a homogenous solution. The sucrose solutions were homogenised under the previously mentioned conditions, cooled at 4 °C and remained at the same temperature for 18 h. Before analyses all sucrose solutions were well agitated, in order to achieve an adequate dispersion of the non-soluble materials.

2.3. DSC measurements

Thermograms were obtained with a Perkin–Elmer calorimeter (DSC6, Perkin–Elmer, Norwalk, PA) and Pyris software for Windows. The DSC instrument was calibrated with pure indium standard before analysis. Aliquots (15 mg) of each sample (solution or ice cream mix) were sealed into aluminium pans (50 μl, Perkin–Elmer) and placed into the DSC. A protocol similar to that proposed by Blond (1994) was implemented including the following steps: (a) cooling to −80 °C at 10 °C/min, (b) heating from −80 to −40 °C and annealing at the same temperature for 30 min to promote maximal ice formation, (c) cooling to −80 °C at 10 °C/min and isothermal holding for 5 min and (d) heating from −80 to 20 °C at 5 °C/min.

Freezing points of the formulations were calculated from the DSC melting curves by determining the temperature at which the steepest slope was observed. The amount of ice formed per gram of sample (*IC*) was determined by integrating the melting curves and dividing the melting enthalpy with the pure ice fusion latent heat ($\Delta H = 334\ J\ g^{-1}$). The percentage of unfreezable (bound) water (*UFW*) was calculated using the formula (Alvarez, Wholters, Vodovotz, & Ji, 2005):

$$UFW\ (\%) = \text{moisture content}\ (\%) - IC\ (\%)$$

The glass transition temperatures were calculated by constructing the tangents to the DSC curve baselines before and after the glass transition. The intersection of these tangents to the tangent of inflection point gives the extrapolated onset, midpoint and endpoint temperatures. During DSC measurements two transitions were reported: rubbery to glassy state (T'_g), and Arrhenius to Williams Lander Ferry (WLF) diffusion kinetics (T'_m) (Goff, 1997).

2.4. Quantification of serum protein concentration

Samples (50 ml) were placed in centrifuge tubes and centrifuged at 10,000*g* for 15 min at room temperature. Centrifugation produced an aqueous phase on the bottom (serum phase), an emulsion phase in the middle and an oil phase on the top. In all analyses the upper oil phase was negligible, and macroscopically only the emulsion and serum phases were detected. The serum was carefully collected with a syringe and precisely weighted. Protein content of the subnatant phase (serum) was determined according to the method of Bradford (1976).

2.5. Rheological measurements

The rheological behaviour of the aged ice cream mixes, model sucrose systems and serum phases was determined using a rotational viscometer (RC1 Rheometer, Rheotec Meßtechnic GmbH, Raderburg, Germany) coupled with a circulating cooling bath (RE312, Lauda GmbH, Keuda-Konigshofer, Germany). The measurements were carried out at 4 °C using a MS-CC48 DIN/FTK cylinder. Samples (80 ml) were allowed to equilibrate at 4 °C for 10 min prior to measurement. Shear stress sweeps from 10–300 s^{-1} were performed after loading of the samples on the viscometer. In order to explain the ice cream mixes rheological behaviour data were fitted to the Ostwald–de Waale model:

$$\sigma = K\gamma^n \quad (1)$$

where σ is shear stress (Pas), γ = shear rate (s^{-1}), K is the consistency index (Pas^n) and n is the flow behaviour index. Apparent viscosity of ice cream mixes was calculated at a shear rate of 50 s^{-1} (Kokini viscosity), which represents the sensing shear rate in the mouth of low viscosity foods (Akhtar, Murray, & Dickinson 2006). The thixotropic character was calculated according to the formula:

$$\% \text{ thixotropy} = 100 \times \frac{\eta - \eta'}{\eta} \quad (2)$$

where: η is the viscosity at 50 s^{-1} during the upward shearing step, and η' is the viscosity at 50 s^{-1} during the downward shearing step.

2.6. Determination of effective molecular weight

Effective molecular weight is a very useful parameter for evaluating the effects on freezing point depression and glass transition temperature in the case of complex multicomponent matrices, such as ice cream. Effective molecular weight (M_s) was determined according to the method proposed by Chen (1986):

$$\ln\left(\frac{\frac{X_w - X_b}{M_w}}{\frac{X_w - X_b}{M_w} - \frac{X_s}{M_s}}\right) = \frac{L_f T_f M_f}{R}\left(\frac{1}{T_0} - \frac{1}{T_f}\right), \quad (3)$$

where T_f, T_0 the freezing point temperature as it was determined by DSC and the freezing point temperature of pure water, respectively (in K), X_w is the mass fraction of water, X_b is the mass fraction of bound water, X_s is the mass fraction of dry matter, M_w is the molecular weight of pure water (kg mol^{-1}), L_f is the ice fusion latent heat (J kg^{-1}) and R is the perfect gas constant.

Ice fusion latent heat was calculated using the formula:

$$L_f = (333.802 + 2.1165T) \times 1000, \quad (4)$$

where T is the temperature expressed in °C.

2.7. Light microscopy

The microstructure of ice cream emulsions was visualised at a magnification of 40× using a digital camera (Sony, W50, Tokyo, Japan) attached to a light microscope (Olympus CH, Tokyo, Japan). Images were processed using Fovea Pro software (Reindeer Graphics, Asheville, CA) with Adobe Photoshop CS2.

2.8. Statistical analysis

In the experiments conducted the effects of fibre type and percentage on the rheological and thermal data were examined. All results were statistically processed using STATISTICA release 7, statistical software (Statsoft Inc., Tulsa, OK). All the results reported are an average of two replicates and the data were analysed using analysis of variance and the Duncan's multiple range test (statistical significance was determined at $p < 0.05$).

3. Results and discussion

3.1. Rheological behaviour of ice cream mixes

The values of rheological parameters of ice cream mixes are displayed in Table 1. It can be seen that the addition of dietary fibre significantly ($p < 0.001$) affected the rheological behaviour of ice cream mixes, enhancing viscosity development and strengthening shear thinning behaviour as depicted by the increase of Kokini viscosity and consistency coefficient and the decrease of flow behaviour index, respectively ($n < 1$). Due to sample complexity, rheology is affected by many factors including the presence of components and their concentration (e.g., fat, polysaccharides and proteins), hydration phenomena occurring during ageing, protein aggregation, fat crystallisation, fat droplets' coalescence or flocculation, etc. (McClements, 1999; Goff, Davidson, & Cappi, 1994; Nor Hayati, Che Man, Tan, & Nor Aini, 2007). The increased viscosity of the fibre-enriched ice cream mixes seems to be caused both by the contribution of the soluble matter to the composition of the aqueous phase and by the contribution of insoluble fibres to the increase of total solids, affecting the three dimensional conformation of the hydrated biopolymers. Comparing the rheological properties of the samples containing inulin, the increase of viscosity is caused by the increase of serum concentration, due to water retention by the soluble fibres. However, the differences in the thixotropic behaviour amongst control and samples with inulin was found to be non-significant ($p > 0.05$), which suggests that the fluidity of the biopolymers was not affected by the presence of soluble fibres. The contribution of the soluble fibres in the viscosity reinforcement of samples enriched with oat and wheat fibre was not important, particularly due to their very low content in soluble matter (3%) and β-glucan. However, all the samples con-

Table 1
Rheological parameters of model sucrose–polysaccharides systems and ice cream mixes with and without addition of dietary fibre after ageing at 4 °C for 18 h (means of two replicates ± standard error).

Treatment	Consistency coefficient K (Pas^{-n})	Rheological behaviour index n	Consistency coefficient K' (Pas^{-n})	Rheological behaviour index n'	Kokini viscosity η (Pas)	Kokini viscosity η' (Pas)	Thixotropy (%)
Sucrose	$0.01^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$0.01^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.00^{a} \pm 0.00$
Sucrose/Oat 2%	$0.03^{a} \pm 0.00$	$0.85^{de} \pm 0.02$	$0.01^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$0.83^{ab} \pm 0.02$	$0.50^{a} \pm 0.00$	$40.59^{f} \pm 2.25$
Sucrose/Oat 4%	$1.05^{bc} \pm 0.19$	$0.46^{ab} \pm 0.04$	$0.54^{c} \pm 0.08$	$0.56^{ab} \pm 0.03$	$6.35^{c} \pm 2.42$	$4.83^{c} \pm 1.41$	$23.54^{de} \pm 4.95$
Sucrose/Wheat 2%	$0.09^{a} \pm 0.02$	$0.63^{bc} \pm 0.03$	$0.02^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$1.06^{b} \pm 0.40$	$1.00^{b} \pm 0.00$	$5.51^{c} \pm 3.56$
Sucrose/Wheat 4%	$2.09^{d} \pm 0.11$	$0.38^{a} \pm 0.04$	$0.64^{c} \pm 0.09$	$0.57^{ab} \pm 0.05$	$9.24^{de} \pm 2.13$	$5.95^{cd} \pm 2.30$	$35.61^{ef} \pm 8.16$
Sucrose/Apple 2%	$0.24^{a} \pm 0.04$	$0.72^{cd} \pm 0.03$	$0.10^{ab} \pm 0.01$	$0.88^{e} \pm 0.04$	$4.01^{c} \pm 1.25$	$3.13^{c} \pm 0.90$	$22.08^{de} \pm 1.52$
Sucrose/Apple 4%	$5.79^{e} \pm 0.38$	$0.41^{a} \pm 0.02$	$3.21^{f} \pm 0.24$	$0.52^{a} \pm 0.03$	$28.79^{f} \pm 4.39$	$24.54^{g} \pm 5.12$	$14.74^{d} \pm 4.12$
Sucrose/Inulin 2%	$0.01^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$0.01^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.00^{a} \pm 0.00$
Sucrose/Inulin 4%	$0.09^{a} \pm 0.01$	$0.71^{cd} \pm 0.02$	$0.02^{a} \pm 0.00$	$1.00^{f} \pm 0.00$	$1.45^{b} \pm 0.29$	$1.00^{b} \pm 0.00$	$30.89^{ef} \pm 11.5$
Ice cream (control)	$0.16^{a} \pm 0.04$	$0.79^{de} \pm 0.04$	$0.15^{ab} \pm 0.02$	$0.79^{cd} \pm 0.03$	$3.52^{c} \pm 1.62$	$3.30^{c} \pm 0.99$	$6.25^{c} \pm 2.43$
Ice cream/Oat 2%	$0.15^{a} \pm 0.03$	$0.81^{de} \pm 0.02$	$0.13^{ab} \pm 0.03$	$0.83^{e} \pm 0.05$	$3.57^{c} \pm 1.09$	$3.34^{c} \pm 1.61$	$6.28^{c} \pm 0.91$
Ice cream/Oat 4%	$1.24^{c} \pm 0.12$	$0.59^{b} \pm 0.03$	$0.69^{c} \pm 0.07$	$0.68^{bc} \pm 0.02$	$12.47^{e} \pm 2.91$	$9.87^{e} \pm 1.89$	$20.87^{de} \pm 1.36$
Ice cream/Wheat 2%	$0.39^{ab} \pm 0.05$	$0.73^{cd} \pm 0.03$	$0.21^{b} \pm 0.03$	$0.83^{e} \pm 0.03$	$6.78^{cd} \pm 1.82$	$5.40^{cd} \pm 1.54$	$20.37^{de} \pm 0.31$
Ice cream/Wheat 4%	$2.71^{d} \pm 0.23$	$0.52^{ab} \pm 0.04$	$1.43^{e} \pm 0.14$	$0.62^{b} \pm 0.01$	$20.72^{f} \pm 5.57$	$16.17^{f} \pm 2.29$	$21.97^{de} \pm 3.92$
Ice cream/Apple 2%	$1.24^{c} \pm 0.11$	$0.65^{bcd} \pm 0.02$	$0.80^{d} \pm 0.10$	$0.72^{bc} \pm 0.04$	$15.77^{ef} \pm 2.80$	$13.38^{ef} \pm 3.31$	$15.16^{d} \pm 2.46$
Ice cream/ Apple 4%	$12.81^{f} \pm 0.87$	$0.45^{ab} \pm 0.05$	4.99 ± 0.47	$0.61^{b} \pm 0.05$	$74.49^{g} \pm 11.2$	$54.26^{h} \pm 9.82$	27.16 ± 4.98
Ice cream/Inulin 2%	$0.14^{a} \pm 0.02$	$0.84^{de} \pm 0.02$	$0.13^{ab} \pm 0.01$	$0.86^{e} \pm 0.02$	$3.76^{c} \pm 0.88$	$3.74^{cd} \pm 0.62$	$0.41^{b} \pm 0.13$
Ice cream/Inulin 4%	$0.36^{ab} \pm 0.03$	$0.81^{de} \pm 0.01$	$0.32^{bc} \pm 0.02$	$0.82^{de} \pm 0.00$	$8.56^{d} \pm 1.08$	$7.91^{de} \pm 0.49$	$7.56^{c} \pm 2.31$

[a–h]Different letters between the rows indicates significant difference ($p < 0.05$) amongst samples, according to Duncan's mean values comparison test.

taining oat and wheat fibres were characterised by significantly improved viscosities ($p < 0.001$) and strengthened shear thinning character ($p < 0.001$) likely induced by the presence of insoluble materials and high water retention ability, reaching up to 800%. The visualisation of the microstructure of the ice cream mixes with 4% oat or wheat fibres revealed the existence of an extended network consisting of hydrated fibres which constrains the serum areas and, thus, leads to increased viscosity (Fig. 1). Similar conformations, but less extended, were also observed in samples with 2% oat or wheat fibre. The thixotropic character of the former samples can be attributed to the gradual orientation of the fibre strands during shearing. The same effect was also observed in the case of ternary sucrose–polysaccharides fibre systems. The addition of apple fibres led to the most pronounced increase of viscosity, which seems to be caused by the synergistic effect of both soluble and insoluble fibres. The significant content of soluble matter in pectin, which is well known for its gel-forming ability (Luz Fernandez, 2001, chap. 30), can explain the intense enhancement of viscosity, which was 3–15 times greater than the control. By observing the microstructure of ice cream mixes with apple fibres, a dense, sandy-like emulsion was detected, resulting from the natively grainy morphology of these fibres (Fig. 1). Generally, the existence of grainy materials does not induce any time-dependent viscosity change, and thus, the thixotropy of the samples fortified with apple fibres may be influenced by the presence of pectin.

3.2. Effects of dietary fibre on freezing point depression

The extent of freezing point depression (FPD) is a critical parameter in ice cream production as it influences the initial mean size of the formed ice crystals and also their native thermodynamic instability, which leads to their gradual growth (Hartel, 2001). Freezing point temperatures of ice cream mixes are given in Table 2. The freezing point of the control ice cream mix was determined by DSC at −2.42 °C, which is very close to the literature data (Cognè, Andrieu, Laurent, Besson, & Nocquet, 2003). Generally, the freezing point is depressed as the serum phase concentration is increased or as the solutes molecular weight is decreased (Hartel, 2001). Although, sugars have a primary affect on freezing point, the addition of high molecular biopolymers, such as polysaccharides, does not induce significant FPD. The type and the percentage of the fibre used affected significantly ($p < 0.001$) the freezing point temperatures and effective molecular weights, as well as the temperature of bulk ice formation. The addition of wheat and oat fibre led to significant ($p < 0.001$) depression of freezing point temperatures, whereas apple fibre and inulin had the opposite effect, leading to slight increase of freezing point. The FPD was found to be more pronounced in the 2% fibre-enriched formulations. The former observations seem to be reasonable as the extent of FPD is the bilateral result of the increase in serum concentration and dry matter effective molecular weight. In the case of oat and wheat fibre addition, the significant decrease of freezing point temperatures is governed by the increase of serum concentration, due to their high water-binding capacity and their low content in soluble fibre which would enrich the serum in high molecular weight biopolymers. In contrast, the moderate effects on the FPD of mixes when apple fibre or inulin were added, are caused by the enrichment of the aqueous phase in high molecular weight polysaccharides, due to the higher contents of the former materials in water-soluble fibres. Thus, the addition of apple fibre or 4% inulin influences FPD in a similar way to hydrocolloids, which generally increase the freezing point temperature of ice cream mixes (Hag-

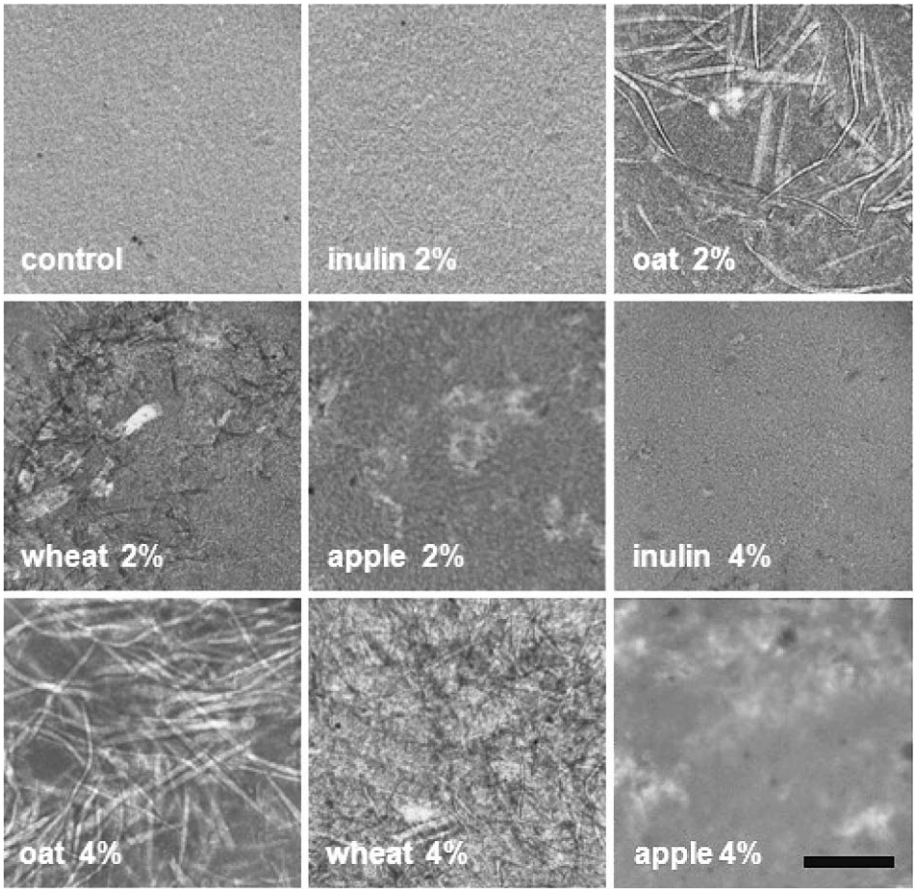

Fig. 1. Light micrographs of aged ice cream mixes with or without addition of dietary fibre taken at room temperature with a magnification of 40× (scale bar 50 μm).

Table 2
Freezing point temperature and equivalent molecular weights of ice cream mixes with and without dietary fibres addition (means of two replicates ± standard error).

Treatment	Freezing point temperature T_f (°C)	Effective molecular weight (kg mol^{-1})	Bulk ice crystallisation temperature T_{ice} (°C)
Control	$-2.42^{b} \pm 0.14$	$0.377^{c} \pm 0.023$	$-23.0^{a} \pm 0.7$
Oat 2%	$-4.31^{d} \pm 0.23$	$0.227^{a} \pm 0.014$	$-26.3^{b} \pm 0.5$
Oat 4%	$-2.96^{c} \pm 0.29$	$0.366^{bc} \pm 0.040$	$-25.9^{b} \pm 0.9$
Wheat 2%	$-4.27^{d} \pm 0.32$	$0.219^{a} \pm 0.018$	$-25.5^{b} \pm 0.8$
Wheat 4%	$-2.94^{c} \pm 0.13$	$0.368^{c} \pm 0.019$	$-24.6^{ab} \pm 0.3$
Apple 2%	$-2.31^{b} \pm 0.09$	$0.464^{d} \pm 0.027$	$-23.7^{a} \pm 0.4$
Apple 4%	$-1.89^{a} \pm 0.21$	$0.531^{de} \pm 0.062$	$-23.3^{a} \pm 0.2$
Inulin 2%	$-3.11^{c} \pm 0.23$	$0.319^{b} \pm 0.026$	$-29.7^{c} \pm 0.9$
Inulin 4%	$-1.78^{a} \pm 0.14$	$0.614^{e} \pm 0.049$	$-28.6^{c} \pm 0.6$

[a–e]Different letters between the rows indicates significant differences ($p < 0.05$) amongst samples, according to Duncan's mean values comparison test.

iwara & Hartel, 1996; Herrera, M'Cann, Ferrero, Zaritzky, & Hartel, 2007). This observation also supports the calculated values of the effective molecular weights (Table 1). The effective molecular weight of the control ice cream mix (0.377 kg mol^{-1}) was very close to that of the containing sugars, i.e., sucrose and lactose (0.342 kg mol^{-1}). The addition of apple fibre and 4% inulin was accompanied by a significant ($p < 0.001$) increase of effective molecular weight, suggesting the contribution of pectin and other polysaccharides in the compositional profile of solutes.

3.3. Effects of dietary fibre on ice crystallisation

The amount of ice formed per gram of sample, and the percentages of the crystallised and unfreezable (bound) water in sucrose–fibre systems and ice cream mixes are displayed in Table 3. The higher amounts of melting enthalpies in the case of model systems stems from their higher water content (ranging from 79.8% to 83.8%) than ice cream mixes (62.6–66.6%). The ice formation in model systems was more intense due to their lower solids content and the absence of proteins, which tend to hydrate and bind water (Alvarez et al., 2005). Considering the impact of dietary fibres addition on ice crystallisation, the addition of wheat or oat fibre led to significant ($p < 0.05$) increase in the percentage of frozen water. Contrariwise, the presence of apple fibre and inulin led to a significant ($p < 0.05$) decrease in the percentage of frozen water in both sucrose systems and ice cream mixes. However, the percentage of the fibre added did not influence the percentage of crystallised water. Ice crystallisation is strongly dependent on the extent of freezing point depression and the percentage of bound water (Hartel, 2001). In our experiments, there was no observed clear relationship between freezing point depression and ice fusion latent enthalpy values, although it might be expected. However, because during the freezing-thawing procedure annealing of the samples occurred, which allowed maximal ice crystallisation, the ice fusion latent enthalpies are primarily dependent on the bound (unfreezable) water and less on the FDP. Indeed, the correlation of the frozen water percentage with the percentage of bound water is strong ($r = 0.97$, $p < 0.001$), indicating that the water-binding capacity and the impact of the components on the steric inhibition of water molecules controlled the ice formation phenomena in the present study. Interpreting the data of unfreezable water percentage, inulin and apple addition led to an increase of bound water, which seems to have resulted by their high contribution to the serum composition, due to their high soluble matter content. Moreover, in the case of apple fibre, the presence of pectin which is able to form gel networks binding water (Luz Fernandez, 2001, chap. 30), is depicted by the high percentages of unfreezable water in both sucrose systems and ice cream mixes. On the other hand, the addition of wheat and oat fibre did not change significantly the percentages of unfreezable water as was expected. A reasonable explanation is that the water-binding capacity of the former fibres is mainly exhibited by the insoluble matter composed of cellulose and hemicellulose, and partially from the soluble matter, e.g., β-glucans being the most important gelling components, which increase the percentage of bound water. (Lazaridou, Biliaderis, & Izydorczyk, 2007, chap. 1).

The melting temperature range (ΔT) values revealed significant ($p < 0.05$) differences amongst sucrose and ice cream formulations, with the control samples showing the greatest range and samples containing inulin the narrowest (Table 3). However, samples

Table 3
Percentage of frozen water for the model sucrose–polysaccharides systems and ice cream mixes with and without addition of dietary fibre (means of two replicates ± standard error).

Treatment	Heat of ice melting ΔH (J g^{-1})	Temperature range (offset – onset) ΔT (°C)	Ice content *IC* (%)	Frozen water *FW* (%)	Unfreezable water *UFW* (%)
Sucrose	$239.9^{h} \pm 5.2$	$52.3^{g} \pm 0.8$	$71.8^{e} \pm 1.6$	$85.7^{f} \pm 1.9$	12.0
Sucrose/Oat 2%	$233.6^{gh} \pm 4.5$	$52.0^{g} \pm 0.5$	$69.9^{e} \pm 1.3$	$85.4^{f} \pm 1.6$	11.9
Sucrose/Oat 4%	$228.1^{fgh} \pm 3.3$	$49.1^{ef} \pm 0.6$	$68.2^{de} \pm 1.0$	$85.6^{f} \pm 1.1$	11.6
Sucrose/Wheat 2%	$238.2^{gh} \pm 9.1$	$51.6^{g} \pm 0.4$	$71.3^{e} \pm 2.7$	$87.2^{f} \pm 4.2$	10.5
Sucrose/Wheat 4%	$221.7^{fgh} \pm 8.3$	$49.0^{ef} \pm 0.7$	$66.4^{de} \pm 2.5$	$83.2^{ef} \pm 3.1$	12.5
Sucrose/Apple 2%	$231.2^{fgh} \pm 8.8$	$50.8^{fg} \pm 0.8$	$69.2^{de} \pm 2.6$	$84.6^{ef} \pm 3.1$	12.6
Sucrose/Apple 4%	$215.1^{f} \pm 6.2$	$49.2^{f} \pm 0.2$	$64.4^{d} \pm 1.9$	$80.7^{cde} \pm 2.4$	15.4
Sucrose/Inulin 2%	$226.6^{fg} \pm 4.2$	$48.9^{ef} \pm 0.3$	$67.8^{de} \pm 1.3$	$82.9^{ef} \pm 1.6$	14.0
Sucrose/Inulin 4%	$221.5^{fg} \pm 5.1$	$47.4^{cd} \pm 0.5$	$66.3^{de} \pm 1.5$	$83.1^{ef} \pm 1.9$	12.6
Ice cream (control)	$169.4^{cd} \pm 4.1$	$47.2^{cd} \pm 0.4$	$50.7^{c} \pm 1.2$	$76.1^{bcd} \pm 1.8$	15.9
Ice cream/Oat 2%	$159.9^{bcd} \pm 5.6$	$44.2^{ab} \pm 0.3$	$47.9^{bc} \pm 1.7$	$74.1^{bcd} \pm 2.7$	16.7
Ice cream/Oat 4%	$164.4^{cd} \pm 6.6$	$45.8^{bcd} \pm 0.5$	$49.2^{bc} \pm 2.0$	$78.6^{cde} \pm 3.2$	13.4
Ice cream/Wheat 2%	$167.3^{cd} \pm 2.3$	$44.1^{ab} \pm 0.5$	$50.1^{bc} \pm 0.8$	$75.6^{bcd} \pm 3.2$	14.5
Ice cream/Wheat 4%	$170.6^{d} \pm 3.7$	$44.8^{abc} \pm 0.7$	$51.1^{c} \pm 2.6$	$81.6^{def} \pm 4.2$	11.5
Ice cream/Apple 2%	$161.9^{cd} \pm 6.4$	$46.3^{cd} \pm 0.8$	$48.5^{bc} \pm 1.9$	$67.7^{a} \pm 2.2$	16.1
Ice cream/Apple 4%	$141.5^{a} \pm 4.2$	$45.6^{bcd} \pm 0.7$	$42.4^{a} \pm 1.3$	$75.1^{bcd} \pm 1.5$	20.2
Ice cream/Inulin 2%	$148.8^{ab} \pm 4.1$	$43.1^{a} \pm 0.6$	$44.6^{ab} \pm 1.2$	$69.0^{ab} \pm 1.9$	20.0
Ice cream/Inulin 4%	$152.2^{abc} \pm 2.9$	$43.3^{a} \pm 0.8$	$45.6^{bc} \pm 0.9$	$72.8^{bc} \pm 1.5$	17.0

[a–h]Different letters between the rows indicates significant differences ($p < 0.05$) amongst samples, according to Duncan's mean values comparison test.

Table 4
Glass transition and melting temperatures for the model sucrose–polysaccharides systems and ice cream mixes with and without addition of dietary fibres (means of two replicates ± standard error).

	T'_g (glass transition temperature)				T'_m (melting temperature)			
	Onset (°C)	Midpoint (°C)	Offset (°C)	ΔT (°C)	Onset (°C)	Midpoint (°C)	Offset (°C)	ΔT (°C)
Sucrose	$-54.8^{abcd} \pm 0.3$	$-51.3^{ab} \pm 0.4$	$-47.8^{ab} \pm 0.2$	7.0	$-41.1^{a} \pm 0.2$	$-37.3^{a} \pm 0.2$	$-35.1^{a} \pm 0.3$	6.0
Sucrose/Oat 2%	$-54.9^{abc} \pm 0.4$	$-50.8^{abc} \pm 0.2$	$-47.6^{ab} \pm 0.3$	7.3	$-40.6^{ab} \pm 0.4$	$-36.6^{ab} \pm 0.2$	$-32.4^{b} \pm 0.3$	8.2
Sucrose/Oat 4%	$-53.4^{efg} \pm 0.2$	$-50.4^{bcd} \pm 0.3$	$-47.1^{ab} \pm 0.2$	6.3	$-39.1^{cdf} \pm 0.4$	$-35.5^{de} \pm 0.3$	$-30.5^{fg} \pm 0.1$	8.6
Sucrose/Wheat 2%	$-54.9^{abc} \pm 0.5$	$-50.6^{bcd} \pm 0.4$	$-47.3^{ab} \pm 0.1$	7.6	$-39.9^{bc} \pm 0.2$	$-36.4^{bc} \pm 0.3$	$-31.3^{cd} \pm 0.2$	8.6
Sucrose/Wheat 4%	$-55.1^{ab} \pm 0.6$	$-49.9^{cd} \pm 0.3$	$-47.9^{a} \pm 0.4$	7.2	$-39.5^{cd} \pm 0.3$	$-35.7^{cd} \pm 0.4$	$-30.4^{fg} \pm 0.4$	9.1
Sucrose/Apple 2%	$-55.8^{a} \pm 0.4$	$-51.6^{a} \pm 0.1$	$-46.9^{bc} \pm 0.3$	8.9	$-40.7^{ab} \pm 0.4$	$-36.1^{bcd} \pm 0.4$	$-31.1^{de} \pm 0.5$	9.6
Sucrose/Apple 4%	$-55.9^{a} \pm 0.7$	$-51.1^{ab} \pm 0.1$	$-47.4^{ab} \pm 0.2$	8.5	$-40.0^{bc} \pm 0.2$	$-36.0^{bcd} \pm 0.3$	$-30.2^{fg} \pm 0.0$	9.8
Sucrose/Inulin 2%	$-53.2^{efg} \pm 0.4$	$-50.2^{cd} \pm 0.2$	$-45.9^{df} \pm 0.4$	7.3	$-39.7^{c} \pm 0.2$	$-34.6^{fgh} \pm 0.2$	$-30.4^{fg} \pm 0.1$	9.3
Sucrose/Inulin 4%	-52.6 ± 0.3	$-49.7^{cd} \pm 0.4$	$-44.7^{h} \pm 0.1$	7.9	$-39.3^{cd} \pm 0.4$	$-34.1^{ghi} \pm 0.1$	$-30.0^{g} \pm 0.2$	9.3
Ice cream (control)	$-53.7^{efg} \pm 0.4$	$-50.5^{bcd} \pm 0.5$	$-46.2^{cd} \pm 0.5$	7.5	$-40.2^{abc} \pm 0.2$	$-36.2^{bcd} \pm 0.3$	$-31.1^{de} \pm 0.0$	9.1
Ice cream/Oat 2%	$-52.5^{fgh} \pm 0.3$	$-49.8^{cd} \pm 0.2$	$-45.7^{dfg} \pm 0.2$	6.8	$-40.1^{abc} \pm 0.6$	$-35.7^{cd} \pm 0.2$	$-32.2^{bc} \pm 0.3$	7.9
Ice cream/Oat 4%	$-52.4^{gh} \pm 0.4$	$-48.6^{d} \pm 0.3$	$-44.8^{gh} \pm 0.3$	7.6	$-38.5^{df} \pm 0.2$	$-34.8^{efg} \pm 0.2$	$-31.5^{bcd} \pm 0.2$	7.0
Ice cream/Wheat 2%	$-52.7^{fgh} \pm 0.2$	$-49.6^{c} \pm 0.2$	$-45.3^{dfgh} \pm 0.1$	7.4	$-40.9^{ab} \pm 0.7$	$-35.5^{de} \pm 0.2$	$-32.4^{b} \pm 0.3$	8.5
Ice cream/Wheat 4%	$-51.8^{h} \pm 0.6$	$-48.1^{fg} \pm 0.1$	$-44.5^{h} \pm 0.0$	7.3	$-40.5^{ab} \pm 0.5$	$-34.9^{ef} \pm 0.1$	$-31.8^{bcd} \pm 0.3$	8.7
Ice cream/Apple 2%	$-53.9^{bcde} \pm 0.2$	$-48.4^{d} \pm 0.3$	$-45.9^{df} \pm 0.2$	8.0	$-39.9^{bc} \pm 0.2$	$-34.4^{fgh} \pm 0.3$	$-30.9^{dfg} \pm 0.3$	9.0
Ice cream/ Apple 4%	$-53.6^{defg} \pm 0.0$	$-47.7^{fg} \pm 0.4$	$-45.2^{fgh} \pm 0.2$	8.4	$-38.2^{f} \pm 0.4$	$-34.0^{hi} \pm 0.1$	$-30.5^{fg} \pm 0.5$	7.7
Ice cream/Inulin 2%	$-50.4^{i} \pm 0.1$	$-47.3^{g} \pm 0.2$	$-44.7^{h} \pm 0.1$	5.7	$-37.2^{g} \pm 0.2$	$-33.4^{ik} \pm 0.0$	$-30.2^{fg} \pm 0.2$	7.0
Ice cream/Inulin 4%	$-49.5^{i} \pm 0.2$	$-46.6^{i} \pm 0.0$	$-43.2^{i} \pm 0.1$	6.3	$-36.8^{g} \pm 0.1$	$-32.8^{k} \pm 0.1$	$-29.1^{h} \pm 0.6$	7.7

[a–k]Different letters between the rows indicates significant differences ($p < 0.05$) amongst samples, according to Duncan's mean values comparison test.

enriched with apple, oat or wheat exhibited a similar behaviour, reflected by their lower ΔT values ($p < 0.001$). Moreover, the increase of the fibres percentage led to significant ($p < 0.001$) decrease of the melting temperature range. Generally, the temperature range is an indicator of the homogeneity of ice crystals' size distribution. Thus, a narrow melting temperature range displays a more homogeneous distribution of ice crystals, which melt over a smaller temperature range (Alvarez et al., 2005). Concluding, it can be said that the fibre enrichment of ice creams may contribute to the improvement of texture perception and the stability of ice crystals during frozen storage, as it favours the formation of tiny ice crystals.

3.4. Effects on glass transition temperatures of sucrose solutions and ice cream mixes

The determination of glass transition temperature (T_g) is of critical importance in ice cream products, as it is strictly associated with their thermodynamic stability during storage (Hagiwara & Hartel, 1996). Proteins and polysaccharides, usually existing in a complex multicomponent form, are used to control the transition from the high viscous-rubbery to the glassy state. Although at temperatures above T_g the ice crystals growth is controlled by water diffusion kinetics, at temperatures below T_g, due to the tremendous increase of viscosity, the diffusion phenomena are effectively limited, leading to a viscosity-controlled thermodynamic stability (Herrera, M'Cann, Ferrero, Zaritzky, & Hartel, 2007; Kasapis, 2006, chap. 2; Regand & Goff, 2003; Slade & Levine, 1991). Many factors affect glass transition such as the plasticiser content (e.g., water), the concentration of the aqueous phase, the critical molecular weight of the solutes and the cooling rate (Hartel, 2001; Kasapis, 2006, chap. 2; Slade & Levine, 1991). Usually in multicomponent systems, e.g., sucrose–polysaccharides–protein solutions, two second-order thermodynamic transitions occur: at the higher temperature (T'_m) there is a transition from linear (Arrhenius) to non-linear WLF control of diffusion phenomena, with simultaneous increase of viscosity, whereas at the lower temperature (T'_g) the transition of the WLF-controlled rubbery state to the vitreous is induced (Sahagian & Goff, 1996, chap. 1).

The effects of dietary fibre addition on glass transition (T'_g) and melting (T'_m) temperatures are displayed in Table 4. In both sucrose systems and ice cream mixes the addition of dietary fibre led to significant increase of glass and melting midpoint temperatures ($p < 0.05$). The elevation of T'_g and T'_m, which occurred in model sucrose systems and ice cream mixes is associated with the restriction of the mobility of water molecules and thus with enhancement of the thermodynamic stability of the formulations. Statistical analysis revealed the significant impact ($p < 0.001$) of the percentage and type of fibres added, as well as the presence of proteins, on the glass and melting midpoint temperatures. The addition of wheat and oat fibre led to a similar behaviour indicating a slight elevation of T'_g and T'_m, respectively. The water-binding capacity of the former fibres which led to increased serum viscosities (Table 5), is the main reason explaining the elevation of T'_g. Concerning the effective molecular weights of the wheat and oat fibre-enriched formulations, one would expect a lower value to control T'_g values. However, the water retention, which seems to be more pronounced as freeze concentration occurs and as the fibres content is increased, combined with the molecular restriction, which is caused by the existence of the hydrated cellulosic network (Fig. 1), explain the gradual increase of T'_g values. If oat fibre had a more significant content of β-glucan, the elevation of T'_g values might be even more pronounced. The presence of proteins was also associated with the elevation of T'_g, though no significant differences in serum protein concentration were determined between control, wheat or oat fibre-enriched samples (Table 5).

The addition of apple fibre had a more complex effect on T'_g and T'_m values. In the absence of proteins only a slight elevation of T'_g and T'_m (about 1 °C) was achieved, obviously induced by the in-

Table 5
Effects of dietary fibres on serum phase characteristics (means of two replicates ± standard error).

Treatment	Serum protein content (g per g of sample)	Viscosity of serum at 4 °C (Pas)
Control	$0.019^{b} \pm 0.004$	$0.063^{a,*} \pm 0.004$
Oat 2%	$0.018^{b} \pm 0.004$	$0.072^{a} \pm 0.005$
Oat 4%	$0.023^{b} \pm 0.002$	$0.079^{a} \pm 0.002$
Wheat 2%	$0.020^{b} \pm 0.002$	$0.078^{a} \pm 0.003$
Wheat 4%	$0.021^{b} \pm 0.004$	$0.073^{a} \pm 0.005$
Apple 2%	$0.008^{a} \pm 0.002$	$0.206^{b} \pm 0.009$
Apple 4%	$0.006^{a} \pm 0.005$	$0.841^{c} \pm 0.013$
Inulin 2%	$0.020^{b} \pm 0.005$	$0.067^{a,*} \pm 0.002$
Inulin 4%	$0.022^{b} \pm 0.001$	$0.069^{a,*} \pm 0.003$

[a–c]Different letters between the rows indicates significant differences ($p < 0.05$) amongst samples, according to Duncan's mean values comparison test.
*Samples exhibited Newtonian behaviour.

crease of the solutes content and the aqueous phase viscosity, due to pectin gelation phenomena. When protein was added (real systems), the elevation of T'_g and T'_m was greater, indicating the contribution of the fibre–protein interactions towards the rubbery to glassy state transition. Again the gelation of pectin contributed to the increase of the T'_g value, but also the induction of phase separation, due to incompatibility of proteins with pectin, may have led to an increase in the local concentration of the soluble polysaccharides and, thus, to T'_g elevation. Indeed, the composition of unfrozen serum phase revealed in the at least two-fold decrease of protein material, supports the occurrence of phase separation. The phase separation in pectin–protein systems and the increase in heat capacity (ΔC_p) in protein–polysaccharides systems when phase separation is provoked is well-established amongst literature data (Rogers, Roos, & Goff, 2006; Tolstoguzov, 2006, chap. 17). The addition of inulin had the strongest elevating effect on T'_g and T'_m in all formulations evaluated. Inulin performed in a similar way to polysaccharides, with the augmentation of T'_g values correlated with the high effective molecular weight of the dry matter, leading to gradual hindering of molecular mobility, as the freeze concentration is proceeding. Inulin undoubtedly had the most significant impact on rubber to glass transition phenomena and, thus, the strongest barrier against water diffusion and recrystallisation.

4. Conclusions

The enrichment of ice cream with dietary fibres is an effective way to enhance nutritional and physiological aspects and to promote functionality by influencing rheological and thermal properties of the final product. The composition and the soluble to insoluble ratio are critical parameters for the exhibited functionality of the added fibres. High content in insoluble fibres led to reinforcement of the viscosity and thixotropy of ice cream mixes and also slight increase of glass transition temperatures, due to water retention and the molecular restriction caused by the presence of an extended network of hydrated cellulosic matter. The increase of insoluble matter affected much less the viscosity development (but still viscosity is increased) but significantly increased the freezing point temperatures, the effective molecular weights of the dry matter and the glass transition temperatures. The presence of protein did not show any noticeable interactions (apart from their contribution to T_g elevation) in the formulations with inulin, oat and wheat fibre, but was found to be a valuable parameter in the case of apple fibre, probably due to its incompatibility with pectin provoking phase separation.

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