

EFFECT OF ACID WHEY CONCENTRATE ON THERMOPHYSICAL-PROPERTIES OF MILK BASED ICE- CREAM

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Hydrolyzed, nanofiltered concentrate of the ultrafiltered permeate (HNF concentrate) of acid whey can be used as food additive in ice-cream production. However, quantity of acid whey in the product is limited by the tendency to crystallisation of ice, the melting properties and taste. Aim of the present study was to examine how the acid whey concentrate influences the quality characteristics of milk-based ice-cream. Thermophysical properties were determined by differential scanning calorimeter, consistency was measured by oscillatory rheometer, and sensorial quality was evaluated by scoring method. It was concluded that the acid whey did not increase the melting of the product, and reduced the freezing point. Cryoscopic temperature, onset point and glass transition temperature (T_g) gradually decreased as the quantity and proportion of acid whey increased in the ice-cream. Rheological results indicated that using HNF acid whey produced more creamy and smooth ice-cream. However, because of its characteristic taste, maximum 20% of milk could be replaced by HNF acid whey in milk-based ice-creams.

Keywords: HNF concentrate, acid whey, ice-cream, DSC, oscillation, rheology

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Whey is the liquid remaining part of milk, following the precipitation and removal of casein proteins from milk during cheese or curd cheese manufacture. It represents 85-89% of the milk and contains 55% of its nutriment. The most abundant components of the whey are lactose (4.5-5 g/100 g), soluble proteins (0.6-0.8 g/100 g), lipids (0.4-5 g/100 g), mineral salts and B vitamins. There are two main whey varieties: the acid and the sweet whey. Acid whey typically has higher mineral and lower lactose content, and it is resulted from the acid coagulation of milk (SISO, 1996). Large amount of milk nutrients remain in the whey, so it can be advantageously used for functional food production (RHICHA et al., 2007). Whey protein concentrate can be used to replace the nonfat dry milk in bakery products, or in sweets (PARSONS et al., 1985). LEE & WHITE (1991) could increase physical and nutritional value of vanilla ice-cream by using ultrafiltered retentates and whey concentrates to substitute SNF (solid non fat).

Quality of ice-cream is determined by the thermophysical, rheological and sensorial properties. Thermophysical properties (ie. heat flow curves, endothermic and exothermic peaks, onset point, transition temperature etc.) can be measured by DSC (Differential Scanning Calorimetry) method (FIGURA & TEIXERA, 2007, BROSTOW et al., 2008). Using this technique, ice crystallisation, glass transition temperature, oxidation and other chemical reactions are often examined (AZIS et al., 2011; DISSANAYAKE & VASILJEVIC, 2009) and quantity of bound, freezable and unfreezable water can also be determined (ALVAREZ et al., 2005). Unfreezable water content influences the instability of the system and alludes to the required storage temperature or temperature range of the ice-cream (HEYS et al., 2008).

Whey protein concentrate has significant effect on rheological properties of ice-cream as it changes the disperse microstructure of the product (AKTAS et al., 1997). The rheological properties (elasticity, viscosity, hardness etc.) of the ice-cream can be suitably analyzed by oscillation rheometry (ADAPA et al., 2000). Temperature sweep (or dynamic thermoanalysis

test) is a widespread method to examine changes in rheological properties of ice-creams at storage or consumption temperature and during melting (MEZGER, 2006).

Aim of the present study was to investigate the effect of HNF acid whey on thermophysical properties, rheological behavior and sensorial quality of milk-based ice-creams.

1. Materials and methods

1.1. Materials

Hydrolyzed and nanofiltered concentrate of acid whey permeate (HNF AWh) was developed and provided by the Hungarian Dairy Research Institute. HNF AWh had 25.2 g/100g dry material content, 0.9 g/100 g protein, 21.50 g/100 g carbohydrate, 11.5 g/100 g lactose, 2.65 g/100 g minerals, 1.53 g/100 g lactic acid, and pH of 4.45.

In addition, commercially available milk (2.8 g/100 g fat content), cream (30 g/100 g fat content), sugar, and ice-cream emulsifier (MEC3, Italy) and flavoring agent (vanilla paste, MEC3, Italy) were used for sample preparation.

1.2. Sample preparation

Six different milk-based ice-creams were produced using 0, 20, 40, 60, 80, 100 % of hydrolyzed and nanofiltered concentrate of acid whey permeate (HNF AWh) to replace milk. Samples are indicated as H0, H20, H40, H60, H80, H100, respectively, according to their HNF AWh content.

Control sample was produced without HNF AWh as follows: 66 g milk, 14 g cream, 15 g sugar, 1 g vanilla paste and 4 g emulsifier were mixed and whipped by an electric whisk for 5 minutes and then frozen by an ice-cream machine (CRM GEL5, Andretti, Italy) for 15 minutes and hardened in a shock freezer (Nortech QCF 103, Normann, Italy) at -35°C.

Samples containing 20, 40, 60, 80, 100 % HNF AWh were produced using 13.2, 26.4, 39.6, 52.8 and 66.0 g/100 g of HNF AWh; 12.0, 9.5, 6.5, 3.3, 0.8 g/100 g of sugar and 14.5, 15.0, 15.4, 16.2, 17.2 g/100 g of cream, respectively, in order to fix the dry material content at 32 g/100 g and the fat content at 6 g/100 g.

Sensory evaluation of ice-cream samples was performed immediately after production. Melted ice-cream samples were used for DSC measurements. For rheological measurements ice-cream samples were frozen using a circle shaped mould 50 mm in diameter and 2 mm in height in order to minimize deformation prior to measurement. Samples were put onto the measuring plate of the rheometer cooled to -10°C, and the measuring bob was kept on ice until starting the measurements.

1.3. Cryoscopic temperature

Cryoscopic temperature was determined based on the freezing curve of ice-cream mix using a refrigerated - heating circulator (Julabo F12, JULABO Labortechnik GmbH, Seelbach, Germany) and a Testo 452 data collector and Ni-Cr-Ni thermocouple controlled by Comsoft 4 software (Testo AG, Lenzkirch, Germany). Temperature was recorded in function of time in order to determine temperature of ice crystal nucleation. Samples were measured in three parallels.

1.4. DSC measurements

Thermophysical properties of samples were determined by a DSC 131 evo (Setaram Instrumentation, Caluire, France) differential scanning calorimeter and an immersion cooler (Flexi-Cool FC100, FTS, USA). Synthetic air (Messer Hungarogáz Kft., Hungary) was used as the purge gas at a flow rate of 50 cm³/min. A three-step calibration was used consisting of empty DSC pan, mercury and indium.

50-70 mg sample was weighed and an empty aluminium DSC pan was used as reference. Temperature program was as follows: cooling from +30°C to -50°C by cooling rate of 5°C/min, then keeping at -50°C for 15 min, and finally heating from -50°C to +30°C by a heating rate of 2 °C/min. Sample temperature (T, °C) and heat flow (H, mW) were recorded. Data collection and analyses was controlled by Callisto Processing 1.076 software (AKTS AG, Switzerland). Glass transition (T_g, °C), onset melting temperature (T_o, °C), and latent heat of melting (peak area, ΔH, J/g) were determined using the heating interval of the curve and fitting tangential sigmoid baseline.

Ratio of ice content (freezeable water) compared to the total water content of the sample was calculated at -50°C, -18°C (storage) and at -10°C (consumption) temperature as follows:

$$IC\% = \frac{\Delta H_{\text{sample}}}{(\Delta H_{\text{water}} \cdot (W / 100))} \cdot 100$$

IC: ice content (% of total water content), W: water content (g/100 g), ΔH_{sample}: latent heat of melting (J/g), ΔH_{water}: latent heat of melting of water: 330 kJ/kg

1.5. Rheological measurements

Oscillation rheological tests were performed using a Physica MCR 51 rheometer (Anton-Paar Hungary Ltd.), with a plate and plate measurement system consisting of P-PTD200 plate and PP50/S (plate 50 mm in diameter) measuring bob using 2 mm gap size. Temperature sweep method was performed increasing temperature from -15°C to 0°C by a heating rate of 1°C/min at constant strain (0.005%) and at constant angular frequency (10 rad/s). All samples were measured five times in the same way.

Storage modulus (G', Pa) and loss modulus (G'', Pa) were recorded as a function of temperature. Results were recorded and analyzed using Rheoplus software ver 3.2. (Anton-Paar Hungary Ltd.) Complex viscosity at -10°C, and temperature at crossover point were determined based on the temperature sweep rheogram.

1.6. Sensory analysis

The organoleptic properties were evaluated by a sensory panel consisting of 15 trained panelists using a scoring system. As for maximum twenty points could be given for the texture while ten points for the colour, taste, texture, total impression, creaminess, homogeneity, scoopability, and susceptibility to melting.

1.7. Statistics

Effect of HNF AWh was evaluated by comparing certain properties of each sample to the control sample by Student t- test at 95% confidence level using Microsoft Excel software.

2. Results and discussion

DSC curves of HNF AWh and ice-cream samples (Fig.1.) showed similar tendencies. The first section of the curves from -40°C to -35°C provides information about T_g (glass transition) properties of the samples (SCHENZ, 1995). Beyond that an endothermic peak could be observed between -5 and 0°C which was reported to be the melting region of ice content (ALVAREZ et al. 2005). Between $+5$ and $+15^{\circ}\text{C}$ a small endothermic peak could be observed which indicated the melting of milk fat (MACNAUGHTAN & FARHAT, 2008). It was at 12°C in H0 sample and HNF AWh. In samples H20 and H40 in which milk content was higher than whey content, this peak shifted to the lower temperature region (appr. 7°C), when whey content was higher than milk content, the peak shifted to the higher region (appr. 16°C).

According to the literature the liquid fraction of milk fat (olein) has a melting range from $+20$ to 30°C (THANASUKARN et al. 2006), but it decreases in presence of emulsifiers (LOPEZ et al., 2006). As reported by KAYLEGIAN et al. (1993) there are some milk fat fractions which melt below 20°C . Considering the composition of milk-based ice-cream, in this case the second

peak indicated the melting of a milk fat fraction. Shifting of peak might be caused by interactions between milk fat and certain compounds of whey (ie. lactose, proteins).

Fig. 1.

Thermophysical properties: the cryoscopic and glass transition temperature, onset point, and heat are shown in Table 1.

Table 1.

Cryoscopic temperatures varied between -3.34 and -2.64°C, which is the temperature range of ice-cream mixes for freezing (BAER et al., 1999, CLARKE, 2012).

Compared to the first ice-cream samples, which did not contain acid whey, cryoscopic temperature of ice-cream made with 40% AWh significantly decreased due to the colligative properties (ie. decrease of onset point depending on the quantity of sugars, mineral salts, fats and proteins (BAER & CZMOWSKI, 1985)). In this case high lactose and mineral content had a reducing effect on onset point which has proportionately decreased as the quantity of AWh has increased ($R^2=0.91$).

Glass transition temperatures of ice-cream samples did not show significant differences in function of AWh concentration. However, melting range, onset point and heat varied significantly as the quantity of AWh was above 60% in the samples (SCHENZ, 1995).

Freezable water content at -50, -10 and 18°C, which is one of the most important quality properties of the ice-creams, is shown in Table 1. Freezable water content was changed significantly as AWh was added at all temperature levels investigated.

FW% (-50°C) refers to the total freezable water content of the samples and it decreased above 60% AWh content. FW% (-18°C) and FW% (-10°C) decreased even when 20% AWh was added. Decrease of FW in presence of AWh can be explained by the fact that lactose, mineral

salts, whey proteins can adsorb more water molecules on their surface than sucrose (BERLIN et al., 1973). Lower FW% might result in reduced melting which can be influenced by the fat content (ROSSA et al., 2012), and amount or size of the ice-crystals (MUSE & HARTEL, 2004).

Temperature sweep rheograms of ice-creams prepared using HNF AWh are shown in Fig. 2. Complex viscosity at -10°C and crossover point of samples are listed in Table 2.

Fig. 2.

Table 2

Rheograms of samples made with varying quantities of acid whey showed similar shape, but G' and G'' values were different. G' and G'' values have not changed until a given temperature, then started to decrease gradually, and finally reached the crossover point. (See Fig.2.)

Rheological behavior at low temperature mainly depends on the quantity of the ice and the microstructure of the ice-cream (e.g.: number and size of the air bubbles, fat repartition, size of ice-crystals etc.). The temperature at which the whole amount of free water is frozen (-50°C), storage modulus is on its maximum and ice-cream behaves like a solid matter. In low temperature range (from -50 to -15°C) the storage modulus doesn't change, but value of loss modulus increase slightly with increasing temperature due to decrease in quantity of ice (WILDMOSER et al., 2004). However, at higher temperatures (between -10 and 0°C) G' and G'' decreased because of the melting ice and finally the whole ice-cream was liquid.

The quantity of ice (quantity of frozen water) in ice-cream prepared using AWh diminishes from 70% of total quantity of water to about 50% between -18 and -10°C, (see Table 1.)

G' and G'' curves started to decrease at -10°C in control and at -12°C in sample H100 suggesting that melting of ice-cream increases with increasing quantity of AWh rich in organic macromolecules and mineral salts (BERLIN et al., 1973).

Slope of decreasing section of temperature sweep rheograms gradually diminished as the AWh content increased. It means that by using AWh gel ($G':G'' = 10:1$) temperature range of ice-cream (MEZGER 2006) became wider, and the samples were easier to portion, to scoop or to swallow.

As AWh content exceeded 60 % complex viscosities of ice-creams measured at -10°C (consumption temperature) were significantly lower than those of control indicating that ice-cream samples at -10°C became softer and easier to consume. The crossover means the temperature at which the sample changes from viscoelastic solid state to viscoelastic liquid state (MEZGER 2006). Above 60% AWh content crossover temperature increased suggesting that gel-like behavior of samples was more resistant to temperature increase, which was perceived by the consumer as a more stable ice-cream product.

Based on the results of rheological tests, the use of AWh produced more smoothly melting and more creamy milk-based ice-creams.

Results of sensory analysis scoring test and organoleptic texture profile analysis are shown in Table 3.

Table 3.

There were no significant differences between color and consistency of the samples made with various AWh contents. However, taste and overall impression scores gradually decreased when proportion of AWh exceeded 20% in the samples. Using more acid whey in

the ice-cream, characteristic aftertaste was perceptible, thus sensory properties degraded. Overall impression was mainly influenced by the taste.

Quality of the ice-cream depends on characteristics such as creaminess, melting, homogeneity and scoopability (PRINDIVILLE et al., 2000, DOOLEY et al., 2010, INOUE et al., 2009). However, scores given during sensory test showed that there were no significant differences between the ice-cream samples, scores of creaminess, homogeneity and scoopability decreased gradually with increasing quantity of AWh.

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Table 1. Thermo-physical properties and freezable water content of ice-creams prepared with HNF AWh

Sample	CT	Tg	To	ΔH	IC% (-50°C)	IC% (-18°C)	IC% (-10°C)
	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD
H0	-2.64 $\pm$ 0.08 ^a	-34.14 $\pm$ 0.97 ^a	-5.55 $\pm$ 0.57 ^a	189.3 $\pm$ 5.07 ^a	85 $\pm$ 2.27 ^a	77 $\pm$ 0.40 ^a	58 $\pm$ 1.59 ^a
H20	-2.69 $\pm$ 0.13 ^a	-35.21 $\pm$ 0.75 ^b	-6.33 $\pm$ 0.36 ^a	178.3 $\pm$ 17.4 ^a	79 $\pm$ 7.34 ^a	71 $\pm$ 1.78 ^b	51 $\pm$ 4.72 ^b
H40	-2.99 $\pm$ 0.13 ^b	-35.63 $\pm$ 0.35 ^b	-6.51 $\pm$ 0.91 ^a	172.6 $\pm$ 11.0 ^b	79 $\pm$ 5.01 ^a	71 $\pm$ 0.68 ^b	55 $\pm$ 1.13 ^b
H60	-3.04 $\pm$ 0.14 ^b	-35.67 $\pm$ 0.46 ^b	-6.72 $\pm$ 0.21 ^b	169.1 $\pm$ 22.8 ^b	80 $\pm$ 3.78 ^a	73 $\pm$ 0.20 ^b	55 $\pm$ 0.70 ^b
H80	-3.09 $\pm$ 0.01 ^b	-35.78 $\pm$ 0.68 ^b	-7.01 $\pm$ 0.27 ^b	184.6 $\pm$ 11.3 ^a	78 $\pm$ 4.75 ^b	70 $\pm$ 0.87 ^b	52 $\pm$ 1.71 ^b
H100	-3.34 $\pm$ 0.08 ^b	-36.53 $\pm$ 0.45 ^b	-7.96 $\pm$ 0.97 ^b	183.5 $\pm$ 2.66 ^a	81 $\pm$ 1.46 ^b	71 $\pm$ 0.12 ^b	50 $\pm$ 0.07 ^b

X: mean,

SD: standard deviation

^aThere is no significant difference compared to the sample (0)

^bThere is a significant difference compared to the sample (0)

CT: cryoscopic temperature (°C)

Tg: glass transition temperature (°C)

To: onset melting temperature (°C)

ΔH : heat (J/g)

IC% (-50°C): ice content (% of total water content) at -50°C,

IC% (-18°C): ice content (% of total water content) at -18°C

IC% (-10°C): ice content (% of total water content) at -10°C

Table 2. Rheological properties of ice-creams prepared with HNF AWh

Sample	CV	CrP
	X±SD	X±SD
H0	188.6±21.1 ^a	-0.05±0.017 ^a
H20	189.6±13.3 ^a	-0.04±0.004 ^a
H40	165.2±67.3 ^a	-0.03±0.017 ^a
H60	120.6±25.5 ^b	-0.02±0.002 ^b
H80	99.7±18.1 ^b	-0.01±0.005 ^b
H100	102.3±6.6 ^b	-0.02±0.006 ^b

X: mean,

SD: standard deviation

^aThere is no significant difference compared to the sample (0)

^bThere is a significant difference compared to the sample (0)

CV: Complex viscosity at -10°C (kPa.s)

CrP: Crossover point (°C)

Table 3. Results of sensory analysis and organoleptic texture profile analysis of ice-creams prepared with HNF AWh

Sample	COL	TAS	TEX	TIM	CRM	HOM	SCO	MES
	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD	X $\pm$ SD
H0	8.75 $\pm$ 1.60 ^a	8.83 $\pm$ 1.19 ^a	15.9 $\pm$ 2.91 ^a	7.73 $\pm$ 1.90 ^a	8.75 $\pm$ 1.14	9.33 $\pm$ 0.98	9.08 $\pm$ 1.00	7.58 $\pm$ 1.68
H20	8.58 $\pm$ 1.83 ^a	8.58 $\pm$ 1.44 ^a	14.9 $\pm$ 3.39 ^a	8.17 $\pm$ 1.80 ^a	8.50 $\pm$ 1.57 ^a	9.50 $\pm$ 1.00 ^a	8.92 $\pm$ 1.62 ^a	8.42 $\pm$ 1.38 ^a
H40	8.08 $\pm$ 1.56 ^a	6.50 $\pm$ 1.30 ^b	15.4 $\pm$ 3.61 ^a	6.09 $\pm$ 1.48 ^b	8.33 $\pm$ 1.40 ^a	8.83 $\pm$ 1.44 ^a	8.67 $\pm$ 1.45 ^a	7.58 $\pm$ 1.60 ^a
H60	8.33 $\pm$ 1.67 ^a	4.42 $\pm$ 1.62 ^b	14.9 $\pm$ 3.73 ^a	5.00 $\pm$ 1.26 ^b	8.08 $\pm$ 1.38 ^a	9.08 $\pm$ 1.16 ^a	8.33 $\pm$ 1.61 ^a	7.67 $\pm$ 1.61 ^a
H80	8.42 $\pm$ 1.56 ^a	4.33 $\pm$ 1.30 ^b	15.2 $\pm$ 1.81 ^a	5.00 $\pm$ 1.26 ^b	8.17 $\pm$ 1.40 ^a	8.92 $\pm$ 1.44 ^a	8.50 $\pm$ 1.45 ^a	7.75 $\pm$ 1.60 ^a
H100	8.50 $\pm$ 1.62 ^a	3.00 $\pm$ 1.21 ^b	15.0 $\pm$ 3.93 ^a	4.27 $\pm$ 1.19 ^b	7.83 $\pm$ 1.19 ^b	8.42 $\pm$ 1.56 ^a	8.42 $\pm$ 1.83 ^a	7.83 $\pm$ 1.19 ^a

X: mean,

SD: standard deviation

^aThere is no significant difference compared to the sample (0)

^bThere is a significant difference compared to the sample (0)

COL: colour

TAS: taste

TEX: texture

TIM: total impression

CRM: creaminess

HOM: homogeneity

SCO: scoopability

MES: melting susceptibility

Fig. 1. Differential scanning calorimetry melting curves for ice-cream samples

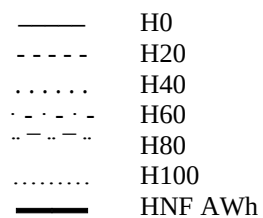
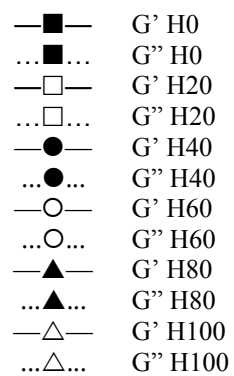


Fig. 2. Temperature sweep rheograms of ice-creams prepared using HNF AWh



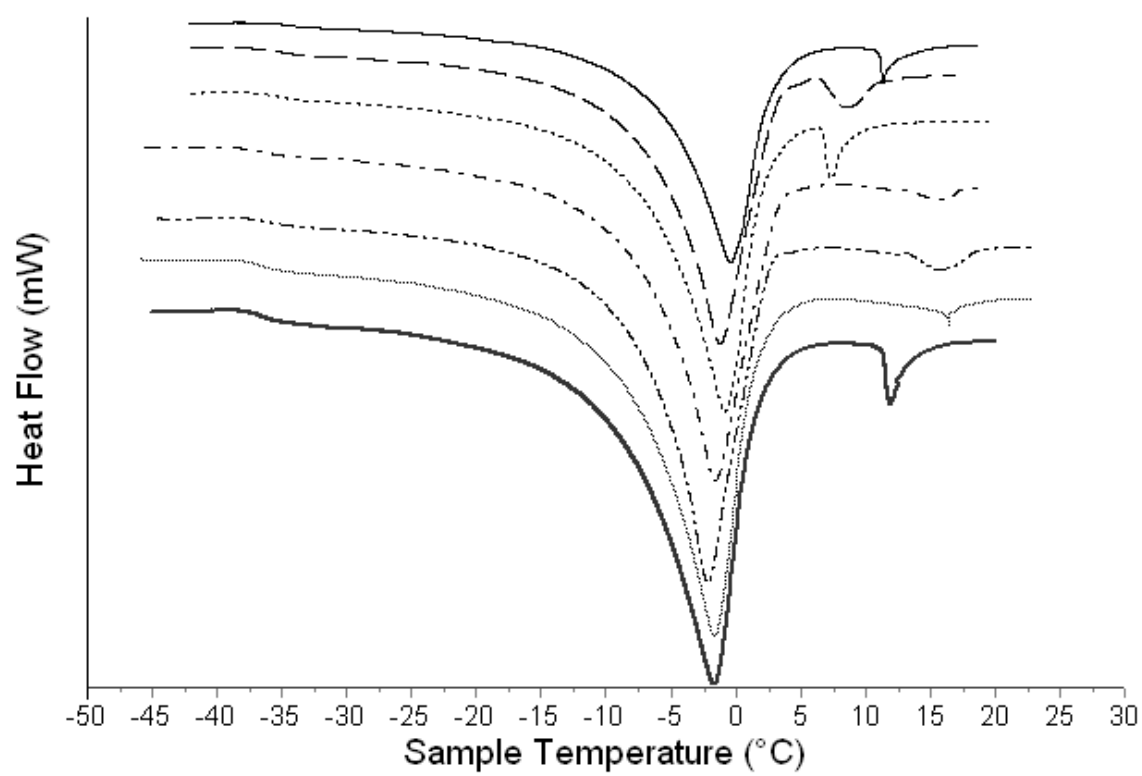


Fig. 1.

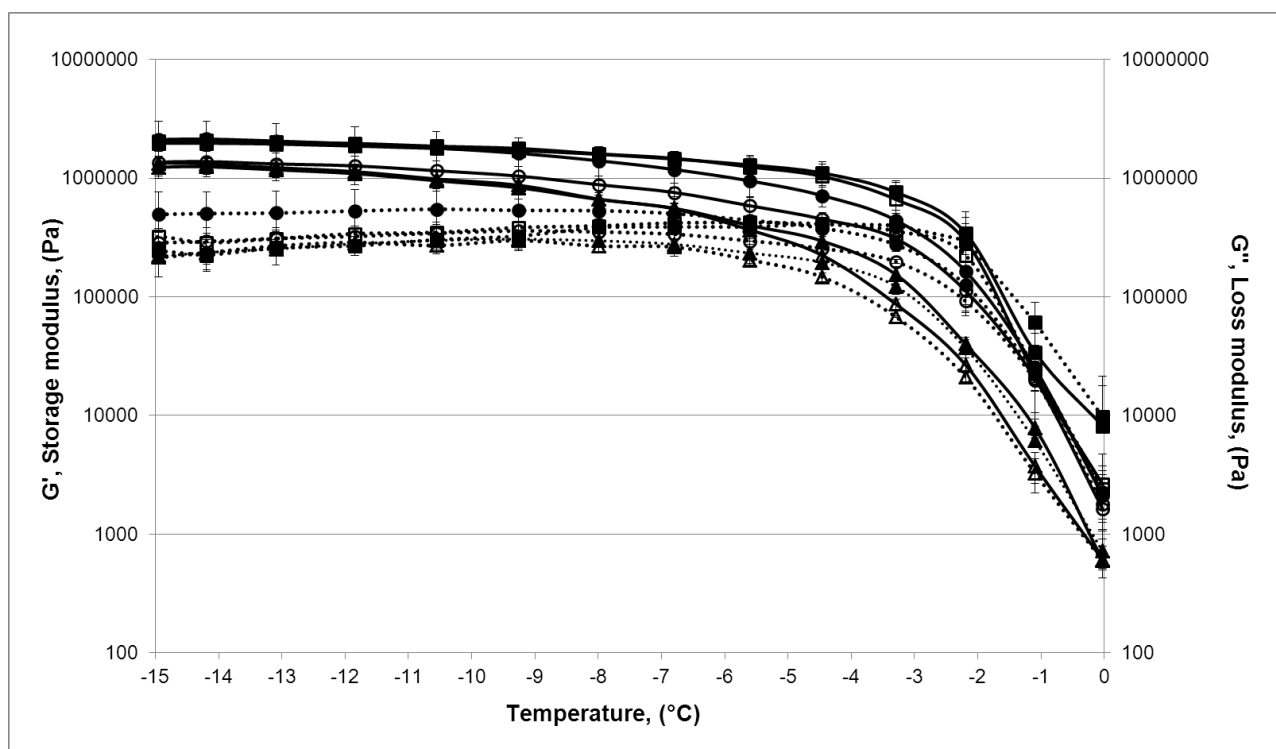


Fig. 2.