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SCIENTIFIC PAPER

MELTING AND CRYSTALLIZATION DSC PROFILES OF DIFFERENT TYPES OF MEAT

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Abstract

The aim of this study was to test the influence of scanning rate and meat type, on the thermo-physical properties of meat and content of the freezable water in frozen meat, using the differential scanning calorimetry (DSC) method. In this study three types of meat were investigated: beef (*M. Longissimus dorsi*), pork (*M. Longissimus dorsi*) and chicken meat (*Pectoralis major*). The cooling rate affected the onset ($T_c$on), peak (T_c) and end ($T_c$end) temperatures of crystallization process of beef meat ($p<0.05$). Decreasing cooling rate from 20 °C/min to 2 °C/min resulted in significant ($p<0.05$) change of the crystallization enthalpy (ΔH_c) of beef meat, from -220.17 J/g to -168.20 J/g, respectively. Reduction of the heating rate caused significant ($p<0.05$) decrease in enthalpy of melting (ΔH_m) for beef meat, from 228.87 J/g to 161.13 J/g. The heating rate affected the peak (T_m) and end temperatures ($T_m$end) of melting process of beef meat ($p<0.05$). The type of meat did not have effect on ΔH_c and ΔH_m as well as temperature of crystallization ($T_c$on, T_c, $T_c$end) and temperature of melting (T_m, $T_m$end) in meat. Significant ($p<0.05$) change in freezable water content were recorded between heating rate 20 °C/min and other heating rates, for all three meat types.

Key words: DSC, meat, crystallization, melting

Highlights

- Freezing rate influences the thermo-physical properties of frozen meat.
- Types of meat influence the thermal-physical properties of frozen meat.
- The temperatures of crystallization and melting of meat depend on freezing rate.
- The temperature of crystallization is different from the temperature of melting of some type of meat.
- Freezing rate affects the ratio of freezable/unfreezable water in the frozen meat.

Introduction

Differential scanning calorimetry (DSC) is a thermoanalytical technique used to study the thermal behaviors of food and pharmaceutical systems [1-6]. This technique has some advantages over other classical detection methods, as it is rapid and does not require additional sample preparation or solvent utilization and therefore, it is an environmental friendly technique. A DSC generated thermogram, in which the rate of heat transfer is plotted versus temperature for a small amount of sample, provides information on the energy released or absorbed in the form of latent heat during phase transition of that sample [2,7,8].

The most commonly occurring phase transitions in food are crystallization of water during freezing and melting of ice during thawing of food [3,9-12]. These processes are extremely important, because physico-chemical and sensory characteristics of foods may be altered after freezing and thawing [13-17].

Freezing and thawing processes play an important role in food processing [9,18]. Freezing is the most widely used technique for food preservation and especially if large quantities of meats are frozen before use [6,14,19]. To optimize these processes and the quality of the final product, it is very useful to analyze the phase transitions which occur in the product [2,20]. However, mathematical description of phase transition is difficult because of the heterogeneous nature of food [2,21-23]. Therefore, it is important to determine analytical techniques for monitoring melting and crystallization processes in food. These techniques are based on the measurements of thermo-physical properties of food, which change during a phase transition [2,23,24].

Thermo-physical properties of food depend to a great extent on food composition and previous treatment, but attention must be paid to the use of different measuring techniques because they can also influence the experimental results [23]. Calorimetry is one of the most powerful tools to study thermo-physical properties of food and phase transition phenomena [25] and it can be used in the analysis and characterization of oils and fats, crystallization and melting profiles, enthalpy of transitions and polymorphic forms [1,5,8,26,27]. The procedure of determining enthalpy in foods, the freezing and melting temperature and the content of unfreezable water by differential scanning calorimetry (DSC) is fast and allows systematic data acquisition and processing to be obtained by utilizing specific software [23,24]. DSC analysis can be performed with different scanning rates, however, there are little studies in the literature which compare DSC thermograms in case of different cooling and heating rates of food.

The aim of this study was to test the influence of selected factors, such as scanning rate and meat type, on the thermo-physical properties of meat and content of the freezable water in frozen meat.

Materials and methods

Standard procedure

In this study, three types of meat were investigated: beef (*M. Longissimusdorsi*), pork (*M. Longissimus dorsi*) and chicken meat (*Pectoralis major*). In all experiments, we used fresh post-rigor meat. Meat was purchased at the slaughter house.

Differential scanning calorimetry thermograms were obtained using a differential scanning calorimeter DSC (204 F1 Phoenix, NETZSCH–Gerätebau GmbH, Germany). Samples (14±2 mg) were weighed into 25 µl capacity aluminum pans. After that, pans were hermetically sealed. An identical empty pan was used as a reference sample during the experiments. The calibration of the cell was made following the DSC manufacturers' recommendation. Flow rate of purge nitrogen atmosphere was 20 ml/min.

The temperature of onset crystallization ($T_c$on), peak crystallization (T_c), end crystallization ($T_c$end) and enthalpy of crystallization ΔH_c (J/g) were measured from the cooling curves and analyzed for the crystallization process. The following temperatures were analyzed for the melting process: temperature of onset melting ($T_m$on), peak melting (T_m) and end melting ($T_m$end). The melting temperature interval was computed as width of melting peak (ΔT_m=$T_m$end-$T_m$on) and the crystallization temperature interval was computed as width of crystallization peak (ΔT_c=$T_c$on -$T_c$end). Enthalpy of melting ΔH_m (J/g) was determined as the area, limited by the melting curve and base line. The Proteus software, version 6.1.0 (NETZSCH–Gerätebau GmbH, Germany) was used to analyze the DSC thermograms and evaluate the thermo-physical properties of meat.

Influence of scanning rate on thermo-physical properties of beef meat

Samples of beef meat were cooled and heated at five rates (2, 5, 10, 15, 20 °C/min). In each scan the sample was equilibrated at 20 °C for 5 min and then cooled to -40 °C at a preset rate; after an isothermal holding stage (-40 °C) of 5 min, the sample was heated at same rate to 20 °C. All the measurements were performed in triplicate.

Influence of meat type on thermo-physical properties of meat

Samples of beef, pork and chicken meat were cooled and heated at 10 °C/min. This scan rate was chosen because it is the most commonly used scanning rate for DSC analysis. In each scan the sample was equilibrated at 20 °C for 5 min and then cooled to -40 °C at a scan rate of 10 °C/min; after an isothermal holding stage (-40 °C) of 5 min, the sample was heated at same rate to 20 °C. All the measurements were performed in triplicate for each meat sample.

Determination of the freezable water

The percent of freezable water (FW) was calculated using the area of the integrated endothermic peak at the range of 0 °C. The endothermic peak arises from the phase transition of ice into water. Eq. (1) was used in order for the freezable water of the meat to be estimated [19].

$$\mathrm{FW}\ (\%) = \frac{Q \cdot 100}{H_f \cdot m_s} \tag{1}$$

Where, FW is the percent of freezable water, Q is the enthalpy of melting (J/g), H_f represents the heat of fusion ice–water equal with 333.50 J/g of ice/water and m_s is the mass of the sample. The percent of unfreezable water (UFW) was calculated by subtracting the percent of freezable water from the percent of total water [28].

The total water content of meat was determined by the method of drying at 105±2 °C to constant mass [29].

Statistical analysis

The results of this study were presented as the mean values accompanied with their standard deviations of three measurements. One factor analysis of variance (ANOVA) was performed using the IBM SPSS Statistics for Windows, version 22.0 (Armonk, NY, United States). Where significant differences ($p<0.05$) were detected, Tukey's multiple comparison was used to compare treatment means and create statistically homogeneous groups.

Results and discussion

The effect of scanning rate on the crystallization and melting processes in beef meat

DSC is a suitable method to characterize phase transitions that require the intake or release of thermal enthalpy, such as crystallization and melting [1]. Figure 1 presents crystallization curves of beef meat obtained using different cooling rates (2, 5, 10, 15 and 20 °C/min). The shape of the curves, as well as the width of crystallization peaks changed depending on the cooling rate (curves are presented in the same scale). As seen in Figure 1, the width of crystallization peaks decreased with decreasing cooling rate, which resulted in significant ($p<0.05$) change of the crystallization enthalpy (ΔH_c) of beef meat, from -220.17 J/g for the rate of 20 °C/min to -168.20 J/g for the rate of 2 °C/min (Table 1). Tomaszewska-Gras [8] also observed the decrease in the width of crystallization peaks of milk fat with a decreasing cooling rate.

Figure 1

Different transition temperatures of crystallization for beef meat were recorded with various cooling rates (2, 5, 10, 15 and 20 °C/min). The temperature at which begins the process of crystallization ($T_c$on) in beef meat significantly ($p<0.05$) changed with changing cooling rate (Table

1). Therefore, the mean values of $T_c$on of beef meat were: -14.93, -19.40 and -18.10 °C, for the rate of 2, 10 and 20 °C/min, respectively (Table 1). The mean values of $T_c$on did not show difference for scanning rate from 5 to 20 °C/min ($p>0.05$), and $T_c$on for 2 °C/min was only different from 10 °C/min ($p<0.05$).

Table 1.

According to the physics, the onset values for pure substances should be always the same, despite of the scanning rates. For the tested meat samples, various cooling rates caused different courses of crystallization, which was manifested in different shapes of peaks, their sizes, as well as different temperatures [8]. A food material is a complex biochemical system where multiple interactions occur during any type of process [25], while the different situation is with pure substances.

Marini et al. [30] reported that chemical composition, especially water content, presents a great influence in thermo-physical properties of meat products. The soluble components such as various sugars, ions and acids and soluble proteins, will contribute to the freezing point depression, while the insoluble components such as fat and the insoluble proteins will not. Furthermore, some components, such as protein and starch, have such high molecular masses that their contribution to the mole fraction of solutes is negligible; it is the small molecules such as sugars, ions and acids, which contribute appreciably [**31**].

Similarly, as in the case of onset crystallization temperature ($T_c$on), the mean values of peak temperatures (T_c) and temperature of end crystallization ($T_c$end) for beef meat significantly ($p<0.05$) changed with the increase in the cooling rate (Table 1). The mean values of T_c of beef meat were: -14.93, -19.63 and -18.60 °C for the rate of 2, 10 and 20 °C/min, respectively. The mean values of T_c did not show difference for scanning rate from 5 to 20 °C/min ($p>0.05$), and T_c for 2 °C/min was statistically different from 10°C/min ($p<0.05$). The mean value of $T_c$end of beef meat was -15.60 °C for the rate of 2 °C/min and -22.00 °C for the rate of 20 °C/min (Table 1). Moreover, it was observed that the cooling rate had significant ($p<0.05$) effect on the crystallization temperature interval (ΔT_c) (Table 1).

This study was also conducted on the process of melting at different scanning rates. Figure 2 presents the melting curves of beef meat obtained at different heating rates (2, 5, 10, 15 and 20 °C/min). As it is presented in Figure 2, different heating rates (from 2 to 20 °C/min) influenced changes in the melting process (of ice) in beef meat. Similarly, as in the process of crystallization, the shape of the curves, the position and the size of peaks changed with various heating rates.

Figure 2

The crystallization curves exhibited narrower peaks when compared to the curves of melting. This indicated that during thawing, phase change occurred over a wider temperature range and more gradually when compared to freezing, as was previously observed in the literature [2]. Analogously, as in the case of crystallization, a reduction of the heating rate caused significant ($p<0.05$) decrease in width of melting peak (ΔT_m) for beef meat. In this study, significant ($p>0.05$) differences were not found between the values of onset melting temperature ($T_m$on) in the range of heating rates from 2 to 20 °C/min. On the other hand, significant ($p<0.05$) differences between mean values of peak melting (T_m) and end melting temperature ($T_m$end) of beef meat were observed for all the heating rates. The values of enthalpy of melting did not differ ($p>0.05$) for heating rates from 2 to 15 °C/min, while significantly ($p<0.05$) different value was recorded for heating rate of 20 °C/min (Table 1).

Enthalpy is used for calculating the total heat to be removed and to determine the rate removal during refrigeration and freezing of food products [32]. Peak area represents the melting latent heat of ice in the tested frozen meat sample.

The influence of the cooling rate on the temperatures and enthalpy of crystallization for beef meat is presented in Figure 3, and the influence of the heating rate on the temperatures and enthalpy of melting for beef meat is demonstrated in Figure 4.

The obtained parameters which indicate the crystallization temperatures of the meat ($T_c$on, T_c, $T_c$end) showed that these temperatures are lower when meat undergo freezing at higher freezing rate. The width of the crystallization temperature interval (ΔT_c), in which the crystallization process of water in meat takes place, increased with increasing cooling rate. In addition, the enthalpy of crystallization of meat samples gradually decreased with increasing freezing rate from 2 to 15 °C/min, while the reduction was more pronounced at freezing rate of 20 °C/min (Figure 3). Similarly, the width of melting peak (ΔT_m), in which the melting process of water in meat takes place, increased with increasing heating rate. For faster thawing procedures, enthalpy of melting increased during the melting of meat samples (Figure 4).

Figure 3

Figure 4

Influence of meat type on thermo-physical properties of meat

The DSC method can be used to obtain the freezing point, heat of fusion (ΔH) and apparent specific heat [33]. Figure 5 and Figure 6 present the crystallization and melting DSC curves of beef, pork and chicken meat for the scanning rate of 10 °C/min. Obtained results were analyzed statistically, and no significant ($p>0.05$) effect of meat type on temperatures ($T_c$on, T_c, $T_c$end) and enthalpy of crystallization (ΔH_c) were observed (Table 2).

Figure 5

Figure 6

Table 2.

In the melting process, no significant ($p>0.05$) differences were found between meat type and temperature of peak melting (T_m), end melting ($T_m$end) and enthalpy of melting (ΔH_m) at the scanning rate of 10 °C/min. In this study, it was observed that the crystallization temperature interval (ΔT_c), melting temperature interval (ΔT_m) and temperature of onset melting ($T_m$on) of beef meat, were significantly ($p<0.05$) different from those of pork and chicken meat (Table 2).

The DSC method can be used to obtain the freezing point, heat of fusion (ΔH) and apparent specific heat [33]. The onset of melting was considered as the freezing point (Tf). Freezing is a safe and a commonly used preservation method for meat and fish products in general [30,33].

Initial freezing point is defined as the temperature, which the first ice crystals start to form. Marini et al. [30] reported that chicken sausages (frankfurter type), mortadela (bologna type) and mechanically deboned chicken meat (MDCM) had very different initial freezing temperature and end point of freezing. Initial freezing temperature and end point of freezing for mortadela (bologna) were -4.46 and -10.14 ºC, respectively. MDCM presented an initial freezing temperature of -0.43 ºC and end point of freezing of -4.46 ºC, and for frankfurter sausages these temperatures were -2.49 ºC and -9.71 ºC, respectively.

The effect of scanning rate on the content of freezable and unfreezable water in meat

Different type of food has different water content that may undergo the phase transition, and hence on the basis of sample mass only partial water–ice transformations can be expected [25]. Water present in food can be classified into two categories according to its reaction to freezing process: freezable and unfreezable water. During freezing, only freezable water crystallizes into ice, whereas unfreezable water undergoes no changes [34].

Unfreezable water is the amount of water unavailable for freezing in a food product at reference temperature of -40 °C [9,32,35]. Unfreezable water is bound water, where as freezable water fraction reflects the fraction of free water of the total water in a product.

The mean values of freezable water and unfreezable water content in three meat types are presented in Table 3. Analogously, as enthalpy of melting, the mean values of freezable water contents and unfreezable water contents did not differ ($p>0.05$) for heating rates from 2 to 15 °C/min. However, significant ($p<0.05$) increase in freezable water content and the decrease in unfreezable water content were recorded between heating rates 2 and 20 °C/min for all three meat types (Table 3).

Tolstorebrov et al. [3] were investigated phase transitions for Atlantic Salmon, Cod, Herring, Mackerel and Rainbow Trout. The amount of unfreezable water was calculated by the DSC melting endotherm integration for scaning rate 5 °C/min and was in the range between 5.1% and 8.6% for all investigated samples.

Table 3.

Conclusions

The shape of the DSC cooling and heating curves of meat depended on the scanning rate. Therefore, it was observed that crystallization and melting peaks changed with an increasing scanning rate. The cooling rate effected the onset ($T_c$on), peak (T_c) and end temperatures ($T_c$end) of crystallization process for beef meat. Enthalpy of crystallization (ΔH_c) for beef meat changed with a changing cooling rate. The effect of heating rate on enthalpy of melting (ΔH_m), peak (T_m) and end temperatures ($T_m$end) of melting process for beef meat was reported. However, the type of meat did not influence temperatures of crystallization ($T_c$on, T_c, $T_c$end), enthalpy of crystallization (ΔH_c), temperatures of melting (T_m, $T_m$end) and enthalpy of melting (ΔH_m). In addition, the increase in freezable water content and the decrease in unfreezable water content were recorded between heating rates 2 and 20 °C/min for all three meat types. The information presented can be helpful for simple and rapid engineering calculations and for implementation in complex mathematical models of heat transfer.

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Figure captions

Figure 1. DSC crystallization curves of beef meat at different cooling rates (2, 5, 10, 15 and 20 °C/min)

Figure 2. DSC melting curves of beef meat at different heating rates (2, 5, 10, 15 and 20 °C/min)

Figure 3. Influence of cooling rate on the temperatures and enthalpy of beef meat crystallization ($T_c$on - temperature of onset crystallization; T_c - temperature of peak crystallization; $T_c$end - temperature of end crystallization; ΔT_c - width of crystallization peak; ΔH_c - enthalpy of crystallization)

Figure 4. Influence of heating rate on the temperature and enthalpy of beef meat ($T_m$on - temperature of onset melting; T_m - temperature of peak melting; $T_m$end - temperature of end melting; ΔT_m - width of melting peak; ΔH_m - enthalpy of melting)

Figure 5. DSC crystallization curves of three types of meat at cooling rate of 10 °C/min

Figure 6. DSC melting curves of three types of meat at heating rate of 10 °C/min

Table 1. Temperatures and enthalpies of crystallization and melting process for beef meat, in relation to diffe

Cooling/ heating rate (°C/min)	***Crystallization***					***Melting***		
	Temperature (°C)				Entalphy ΔH_c (J/g)	Temperature (°C)		
	$T_c$on	T_c	$T_c$end	ΔT_c		$T_m$on	T_m	$T_m$end
2	-14.93^{b} ±1.07	-14.93^{b} ± 1.07	-15.60^{c} ± 1.13	0.67^{a} ± 0.06	-168.20^{b} ± 6.30	-2.87^{a} ± 0.21	0.23^{a} ± 0.06	0.60^{a} ± 0.17
5	-16.23^{ab} ± 2.31	-16.40^{ab} ± 2.31	-17.77^{bc} ± 2.10	1.53^{b} ± 0.23	-171.00^{b} ± 4.10	-1.60^{a} ± 1.25	1.90^{b} ± 0.46	2.93^{b} ± 0.32
10	-19.40^{a} ± 1.00	-19.63^{a} ± 1.05	-21.93^{a} ± 0.85	2.53^{c} ± 0.15	-179.70^{b} ± 11.14	-1.43^{a} ± 0.06	3.73^{c} ± 0.06	5.27^{c} ± 0.15
15	-18.03^{ab} ± 0.75	-18.33^{ab} ± 0.75	-20.73^{ab} ± 0.55	2.70^{c} ± 0.20	-182.97^{b} ± 6.29	-2.63^{a} ± 0.06	5.10^{d} ± 0.30	7.13^{d} ± 0.25
20	-18.10^{ab} ± 1.30	-18.60^{ab} ± 1.20	-22.00^{a} ± 0.90	3.90^{d} ± 0.40	-220.17^{a} ± 2.55	-2.63^{a} ± 0.06	7.60^{e} ± 0.10	10.60^{e} ± 0.10

Data are expressed as mean ± standard deviation.

^{a,b}Mean values in the same column followed by different letters indicate significant difference ($p < 0.05$)

$T_c$on - temperature of onset crystallization; T_c - temperature of peak crystallization; $T_c$end - temperature of end crystalliz enthalpy of crystallization;

$T_m$on - temperature of onset melting; T_m - temperature of peak melting; $T_m$end - temperature of end melting; ΔT_m - width of

Table 2. Temperatures and enthalpies of crystallization and melting process in beef, pork and chicken mea

Sample	***Crystallization***					***Melt***		
	Temperature (°C)				Entalphy ΔH_c (J/g)	Temperature (°C		
	$T_c$on	T_c	$T_c$end	ΔT_c		$T_m$on	T_m	$T_m$en
Beef meat	-19.40^a ± 1.00	-19.63^a ± 1.05	-21.93^a ± 0.85	2.53^a ± 0.15	-179.70^a ± 11.14	-1.43^b ± 0.06	3.73^a ± 0.06	5.27 ± 0.1
Pork meat	-16.87^a ± 1.97	-17.23^a ± 1.86	-20.20^a ±1.95	3.33^b ± 0.31	-190.70^a ± 1.30	-3.07^a ± 0.35	4.27^a ± 0.55	5.90 ± 0.7
Chicken meat	-18.90^a ± 1.21	-19.27^a ±1.15	-22.00^a ±1.30	3.10^b ± 0.10	-183.33^a ± 6.53	-2.80^a ± 0.00	4.40^a ± 0.44	6.20 ± 0.3

Data are expressed as mean ± standard deviation.

^{a,b}Mean values in the same column followed by different letters indicate significant difference ($p<0.05$)

$T_c$on - temperature of onset crystallization; T_c - temperature of peak crystallization; $T_c$end - temperature of end crystallization; ΔT_c - crystallization;

$T_m$on - temperature of onset melting; T_m - temperature of peak melting; $T_m$end - temperature of end melting; ΔT_m - width of melting pea

Table 3. Content of freezable and unfreezable water in relation to different type of meat and different heating r

Heating rate (°C/min)	Beef meat		Pork meat		Chicken
	FW (%)	UFW (%)	FW (%)	UFW (%)	FW (%)
2	48.32[a] ± 2.71	26.52[b] ± 2.71	57.94[a] ±1.19	15.64[b] ± 1.19	52.07[a] ± 3.00
5	50.90[a] ± 4.98	23.94[b] ± 4.98	58.69[a] ± 2.61	14.89[b] ± 2.61	55.82[ab] ± 1.25
10	53.65[a] ± 3.20	21.19[b] ± 3.20	59.02[a] ± 0.02	14.56[b] ± 0.02	56.53[ab] ± 2.39
15	54.89[a] ± 1.89	19.95[b] ±1.89	59.19[a] ± 1.89	14.39[b] ± 1.89	57.52[ab] ± 1.28
20	68.63[b] ± 0.36	6.21[a] ± 0.36	64.05[b] ± 0.34	9.53[a] ± 0.34	61.53[b] ± 3.04

FW - freezable water

UFW - unfreezable water

[a,b] Data are expressed as mean ± standard deviation. Mean values in the same column followed by different l difference (p< 0.05).

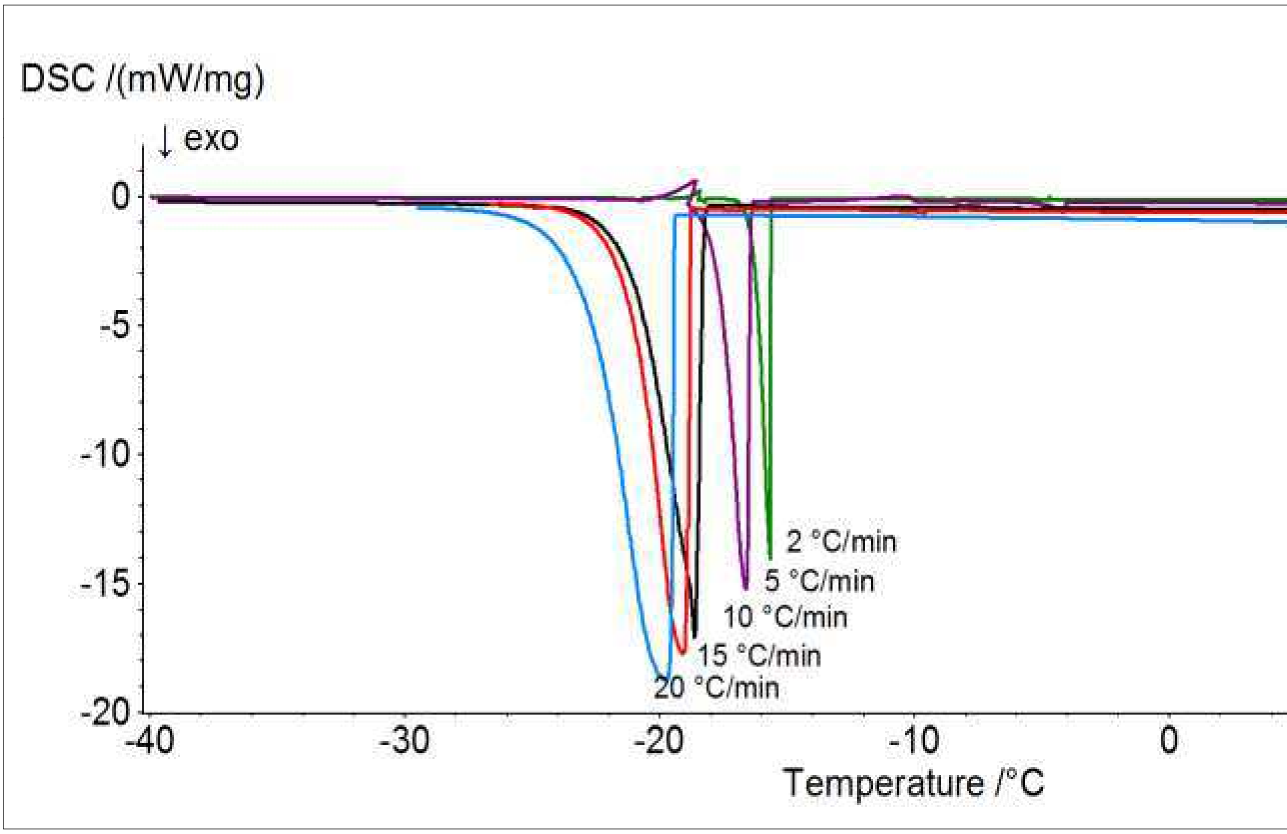
DSC /(mW/mg)
↓ exo
0
-5
-10
-15
-20
-40
-30
-20
-10
0
Temperature /°C
2 °C/min
5 °C/min
10 °C/min
15 °C/min
20 °C/min

Figure 1

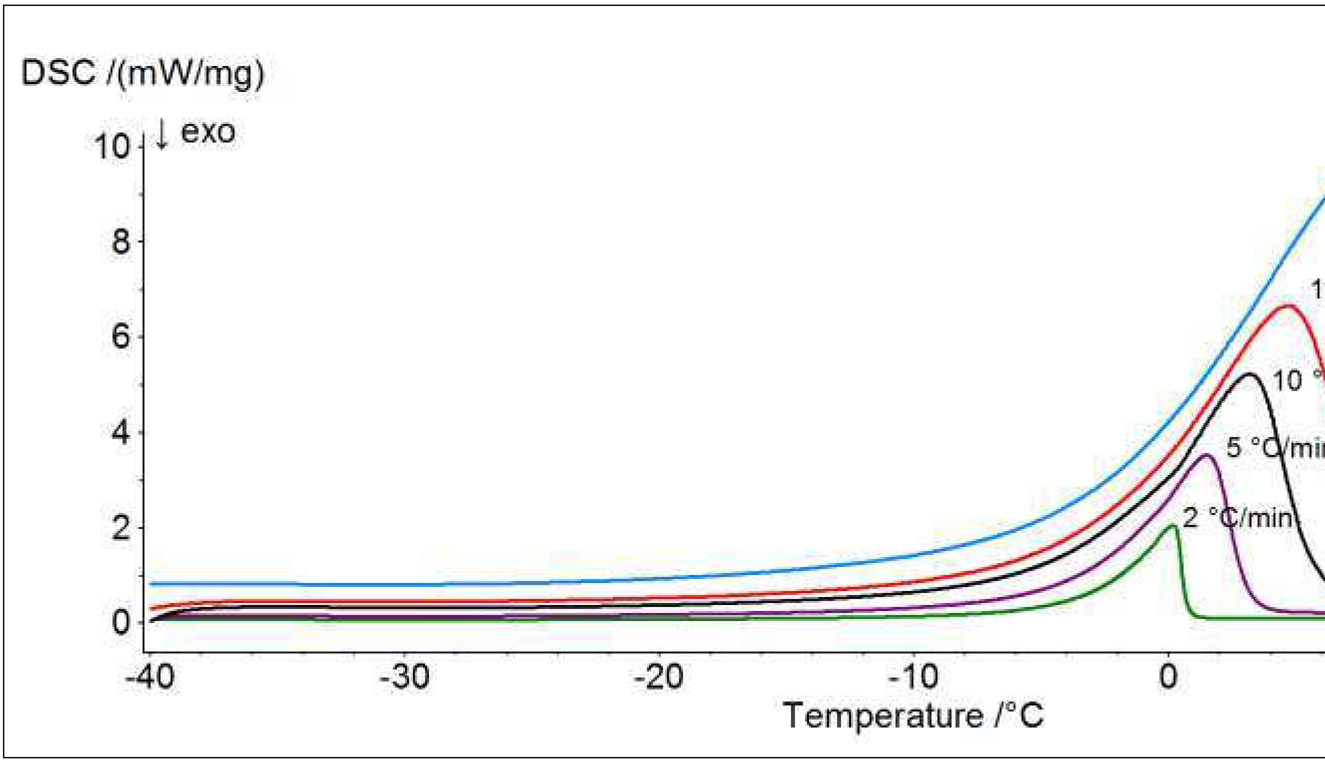
DSC /(mW/mg)
↓ exo
10
8
6
4
2
0
-40
-30
-20
-10
0
Temperature /°C
1
10 °
5 °C/mi
2 °C/min

Figure 2

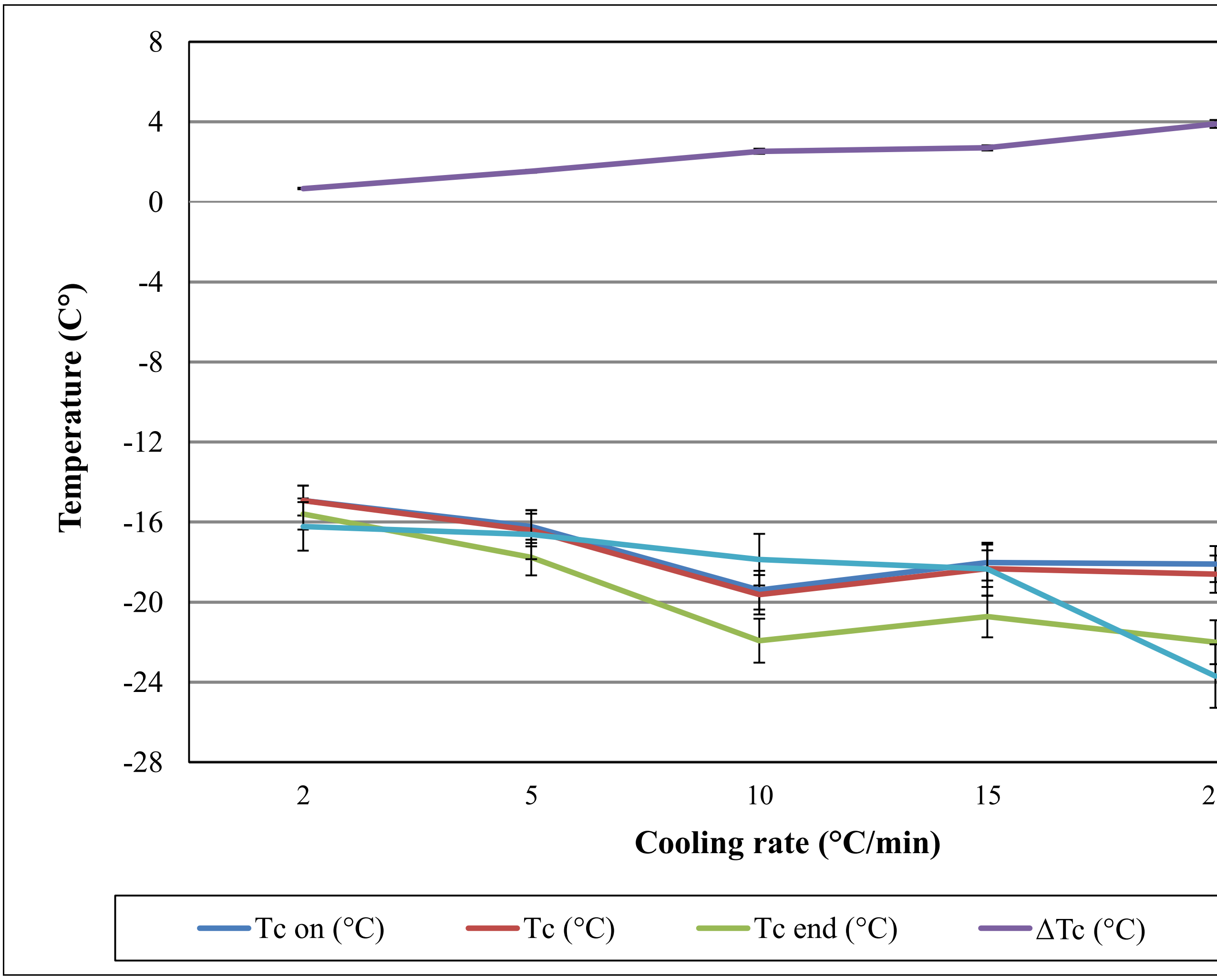
8
4
0
-4
-8
-12
-16
-20
-24
-28
Temperature (C°)
2
5
10
15
Cooling rate (°C/min)
Tc on (°C)
Tc (°C)
Tc end (°C)
ΔTc (°C)

Figure 3

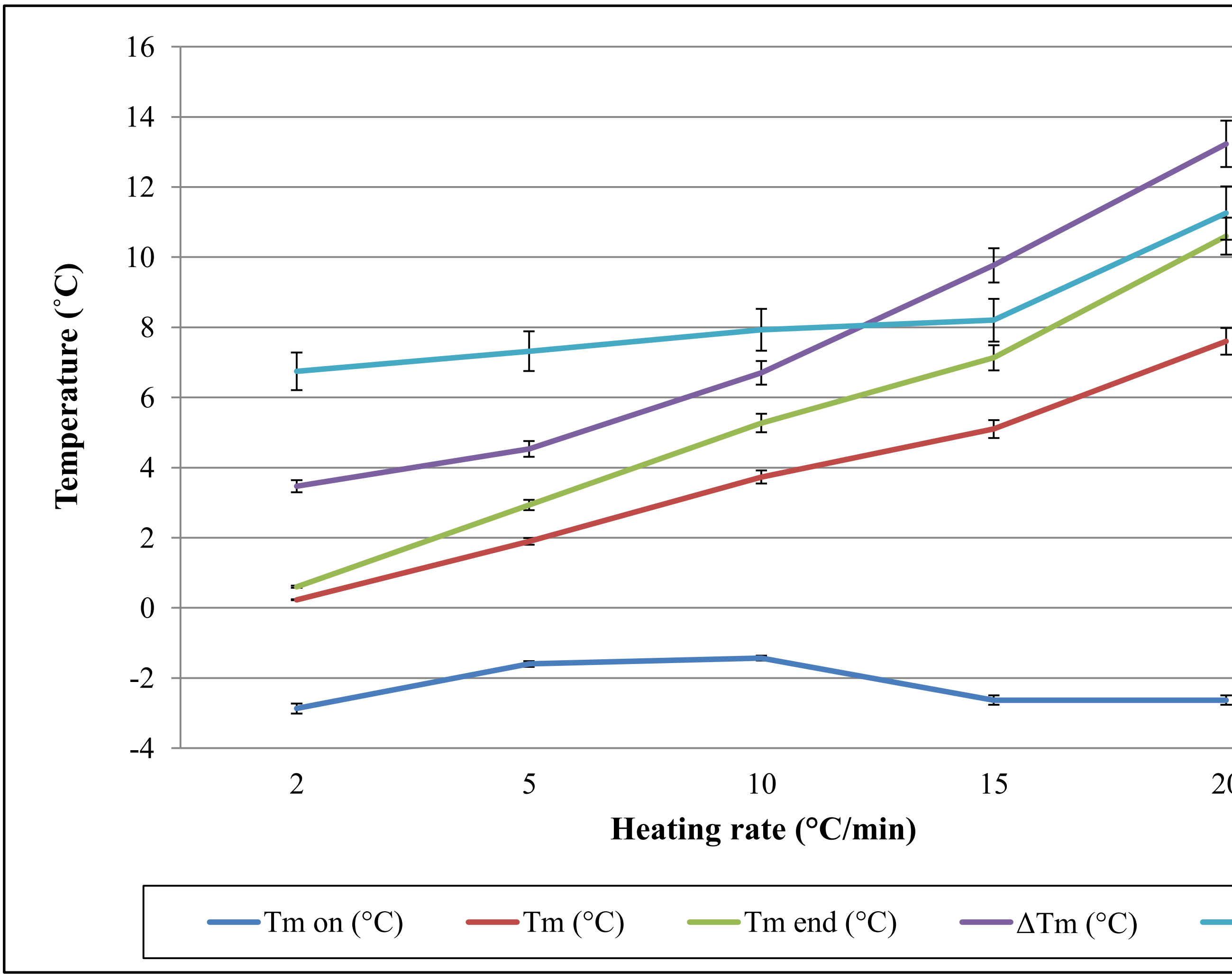
16
14
12
10
8
6
4
2
0
-2
-4
Temperature (°C)
2
5
10
15
Heating rate (°C/min)
Tm on (°C)
Tm (°C)
Tm end (°C)
ΔTm (°C)

Figure 4

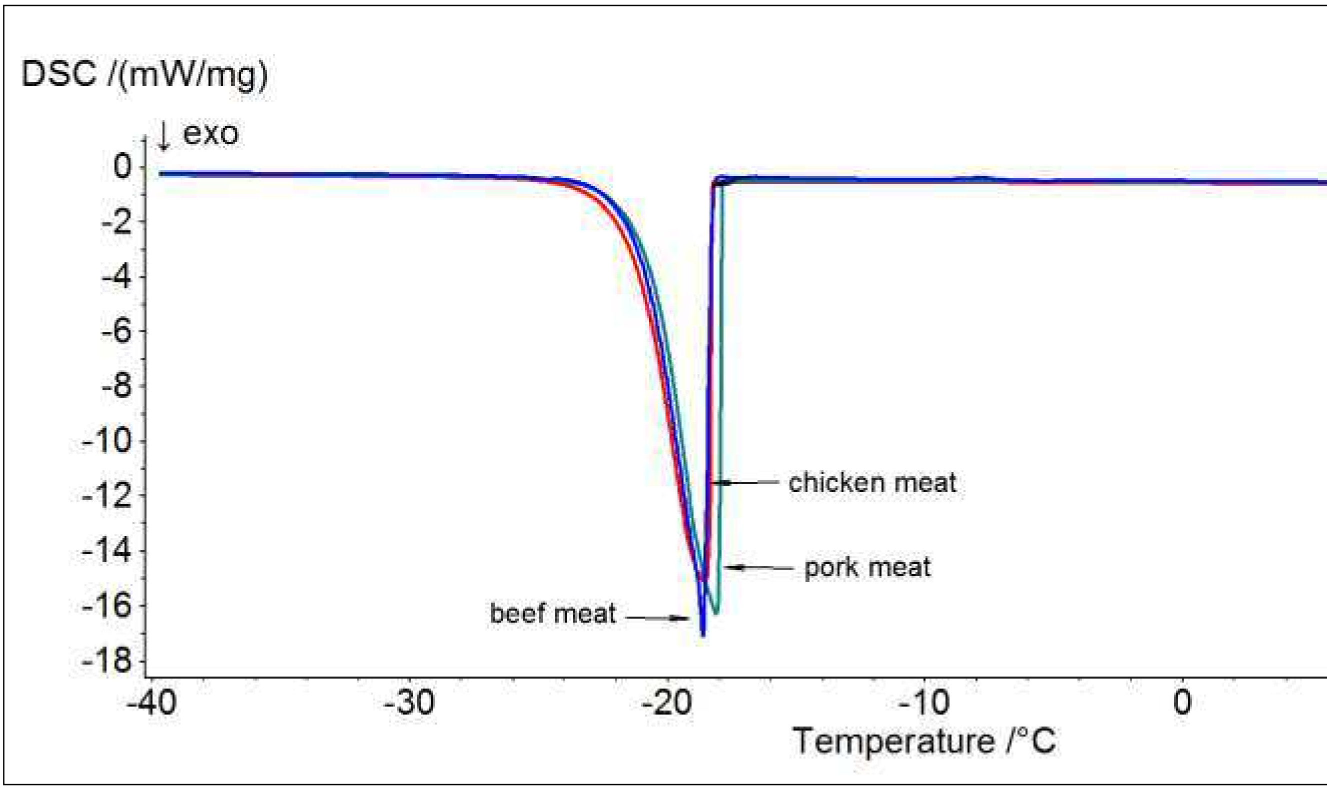
DSC /(mW/mg)
↓ exo
0
-2
-4
-6
-8
-10
-12
-14
-16
-18
-40
-30
-20
-10
0
Temperature /°C
chicken meat
pork meat
beef meat

Figure 5

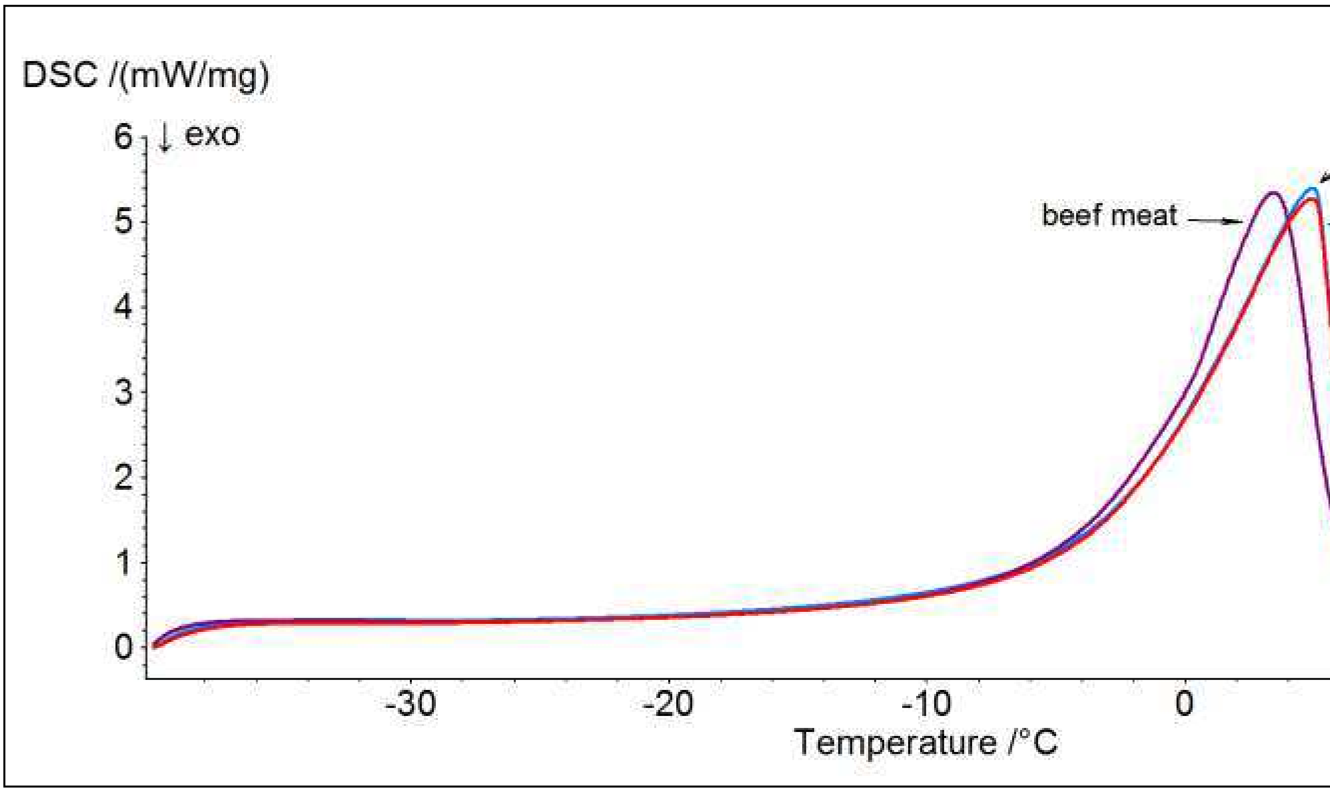

Figure 6