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Wheat grain cooking process as investigated by modulated temperature differential scanning calorimetry

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Abstract

The thermal properties of soft and hard wheat grains, cooked in a steam pressure cooker, as a function of cooking temperature and time were investigated by modulated temperature differential scanning calorimetry (MTDSC). Four cooking temperatures (110, 120, 130 and 140 °C) and six cooking times (20, 40, 60, 80, 100 and 120 min) for each temperature were studied. It was found that typical non-reversible heat flow thermograms of cooked and uncooked wheat grains consisted of two endothermic baseline shifts localised around 40–50 °C and then 60–70 °C. The second peaks of non-reversible heat flow thermograms (60–70 °C) were associated with starch gelatinisation. The degree of gelatinisation was quantified based on these peaks. In this study, starch was completely gelatinised within 60–80 min for cooking temperatures at 110–120 °C and within 20 min for cooking temperatures at 130–140 °C. MTDSC detected reversible endothermic baseline shifts in most samples, localised broadly around 48–67 °C with changes in heat capacity ranging from 0.02 to 0.06 J/g per °C. These reversible endothermic baseline shifts are related to the glass transition, which occurs during starch gelatinisation. Data on the specific heat capacity of the cooked wheat samples are provided.

Keywords: Wheat grain cooking; Modulated temperature differential scanning calorimetry; MTDSC

1. Introduction

The cooking of wheat grains using a batch process, with live steam, is the initial step for the production of some wheat-based cereal products. Though other cooking techniques are available, this traditional process is still used for some cereal products such as wheat flake biscuits, where the desired product characteristics cannot be obtained using other techniques (Caldwell et al., 2000). Most recent published works have concentrated on modern cooking techniques such as extrusion cooking and there has been little work on the batch type of cooking, though, there is still a need for a greater understanding of this older cooking system (Horrobin, Landman, & Ryder, 2003).

The cooking process develops the physical properties necessary for the development of texture, primarily by gelatinisation of the starchy grain fractions (Caldwell et al., 2000). Starch gelatinisation has been investigated by various techniques for many years. Thermal analysis techniques generally involving the use of differential scanning calorimetry (DSC) have been widely used to study gelatinisation (Lelièvre & Liu, 1994) in a variety of systems including the cooking of wheat grains (Jankowski & Rha, 1986; Stapley, Gladden, & Fryer, 1997). DSC has proved to be an effective tool for quantifying the rate/degree of gelatinisation (Ndife, Sumnu, & Baymdirli, 1998; Turhan & Gunasekaran, 2002). Much of the previous work has focused on starch gelatinisation as influenced by starch types, process conditions and effects of ingredients, e.g. sugar, NaCl, etc. in the starch and/or flour system. There has been a very limited discussion of starch gelatinisation in the whole grain system (without isolation). DSC has also been used in determining the glass transition of various cereal materials (Bindzus et al., 2002; Carvalho & Mitchell, 2001; Jang, Lee, Cho, & Pyun, 2001; Kalichevsky & Blanshard,

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1993; Kalichevsky, Jaroszkiewicz, Ablett, Blanshard, & Lillford, 1992; Kalichevsky, Jaroszkiewicz, & Blanshard, 1992; Laaksonen, Kuuva, Jouppila, & Roos, 2002; Laaksonen & Roos, 2000, 2001; Laaksonen, Roos, & Labuza, 2001; Noel, Parker, Ring, & Tatham, 1995; Zeleznak & Hoseney, 1987).

Recently, modulated temperature differential scanning calorimetry (MTDSC), which provides more capability than conventional DSC, has been used for thermal characterisation of food materials (Baik, Sablani, Marcotte, & Castaigne, 1999; Bell & Touma, 1996; Cuq & Icard-Verniere, 2001; Lai & Cheng-Yi-Lii, 1999). In MTDSC, the sample undergoes a sinusoidal temperature modulation (oscillation) overlaid on the conventional linear heating or cooling ramp. This provides the benefits of separating reversible and non-reversible thermal events, improving resolution of closely occurring or overlapping transitions and giving good precision in the measurement of heat capacity. The total, reversible and non-reversible heat flows can be quantified during the transition of the samples in one single scan. The reversible and non-reversible signals reveal the thermodynamic and kinetic characteristics, respectively, of transitions. Examples of events associated with non-reversible signals are the endothermic relaxation of amorphous materials, gelatinisation, recrystallisation and protein denaturation. Reversible events include glass transition and simultaneous crystallisation (Bell & Touma, 1996; Cuq & Icard-Verniere, 2001; Reading, Elliott, & Hill, 1993). This study aims to investigate wheat grain cooking process, as well as observe thermal events associated with starch gelatinisation, glass transition and specific heat capacity using the MTDSC.

2. Materials and methods

2.1. Wheat samples and preparation

Mixed varieties of soft and hard wheat grains (10.5 and 15.5% protein content, respectively) obtained from grain traders in Australia were used. Samples (100 g) of sorted whole-wheat grains with fairly uniform sizes were placed on a wire mesh and then cooked in a pressure cooker, based on a static autoclave (45 l volume, 413 kPa maximum pressure). The initial moisture content of wheat grains was adjusted to about 10% (dry basis), by vacuum drying at 40 °C until desired moisture content was reached, before cooking. The moisture content was determined using the standard air-oven method (AACC, 2000). Wheat samples were cooked immediately or kept in desiccator with a saturated salt solution at the desired relative humidity to maintain the moisture content (if needed). Four cooking temperatures (110, 120, 130 and 140 °C) and six cooking times (20, 40, 60, 80, 100 and 120 min) for each temperature were studied. At least three replicates were conducted. The nominal cooking time was defined as the time for which the desired temperature was reached and held constantly. The total cooking time was about 10 min longer due to the need to pressurize and vent to ensure saturated steam conditions (about 6 min) and to depressurize (about 4 min).

Once cooking conditions were achieved, cooked wheat grains were removed from the cooker and immediately kept at 0 °C to avoid any changes in remaining starch. The wheat kernels were ground, using a Warling blender, to a fine particle size and kept frozen in an airtight container until used for MTDSC experiment (thawing before use at room temperature).

2.2. MTDSC experiment

The moisture content of cooked wheat samples was determined using the standard air-oven method (AACC, 2000). It was found that the final moisture content of cooked wheat grains ranged from 25 to 60% (wet basis). The moisture content of all samples was adjusted to 70% (wet basis) for MTDSC analysis, which was carried out in a TA 2920 DSC (TA Instruments, New Castle, DE, USA). Moisture-adjusted samples were kept in a small airtight container overnight on a slow rotating roller at room temperature to ensure that samples were completely equilibrated. Cooling in the MTDSC was carried out using liquid nitrogen, and the purge gas was helium. The temperature, enthalpy and heat capacity of the instrument were calibrated using indium, zinc, tin, mercury and sapphire. The wheat samples (20 ± 5 mg) were weighed into aluminium DSC pans and hermetically sealed. An empty pan was used as the reference. In earlier studies in our laboratory on the influence of amplitude (0.5–2 °C), period (30–100 s) and heating rate (2.5–7.5 °C/min) on reversible and non-reversible changes in starch-containing systems (0–80% amylose), the following MTDSC conditions were found to be the optimum for measurable changes, and therefore used in this present study (Tan, Wee, Sopade, & Halley, 2004a): equilibrate at 25 °C, modulate at ±1 °C, period of 60 s, heating rate of 5 °C/min to 140 °C. The selected samples were scanned and rescanned for confirmation of the results. At least two replicates were conducted for all samples except cooked wheat grains at high temperatures (130 and 140 °C) where starch gelatinisation was found to be complete. Thermograms of cooked wheat grains were obtained and data were analysed for non-reversible heat flow, reversible heat flow and heat capacity using the TA Universal Analysis Software Version 2.6D. The percentage degree of gelatinisation (DG) was determined by comparing the enthalpy of cooked and uncooked samples obtained from the non-reversible heat flow thermograms (Eq. (1)). Uncooked wheat samples were ground and adjusted to 70% (wet basis) moisture content for analysis

$$\text{Degree of gelatinisation (\%)} = \left(1 - \frac{\Delta H_1}{\Delta H_2}\right)100 \qquad (1)$$

where ΔH_1 and ΔH_2 are the enthalpy of cooked and uncooked wheat samples, respectively.

3. Results and discussion

Typical non-reversible heat flow thermograms of cooked wheat grains determined using the MTDSC are shown in Fig. 1a. Generally, two endothermic peaks, which disappeared when samples were reheated (Fig. 1b), were obtained. The first endothermic peaks were obtained in most samples while the second peaks were found only in wheat samples which had been cooked for short times and/or at low temperatures (110–120 °C for not longer than 60 min). The peak temperatures of the first peaks were localised approximately between 40 and 50 °C and the second peaks between 60 and 70 °C. These temperatures slightly increased for wheat samples, which had been cooked for longer times. The temperatures (onset, peak and end temperatures) for the non-reversible heat flow thermograms are summarised in Table 1.

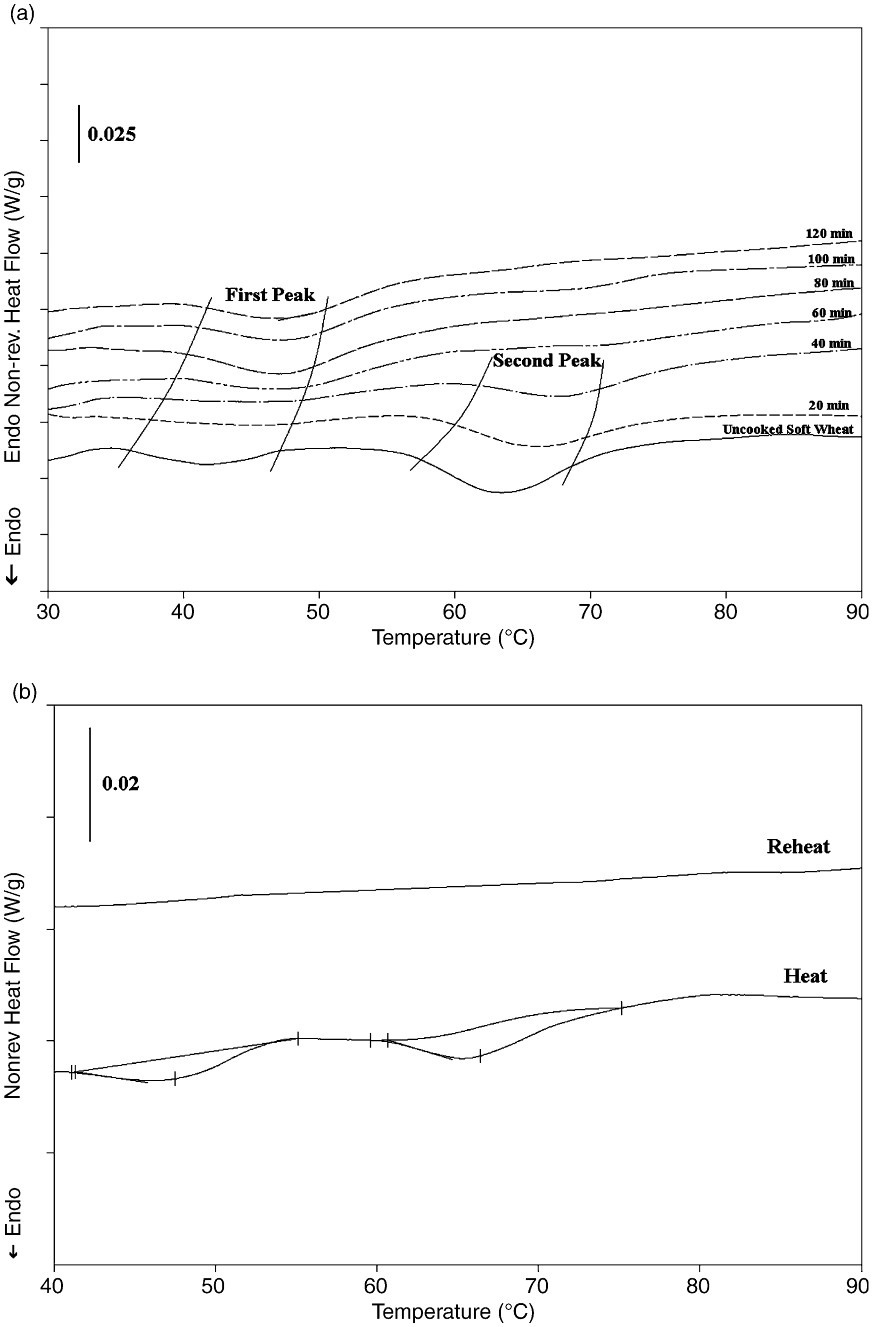

Fig. 1. (a) Non-reversible heat flow thermograms of cooked wheat samples (from soft wheat grains cooked at 110 °C for 20–120 min and uncooked sample) and (b) non-reversible heat flow thermograms of cooked wheat samples during heating and reheating (from hard wheat grains cooked at 110 °C for 20 min).

Table 1
Onset, peak and end temperatures (T_o, T_p and T_e) from non-reversible heat flow thermograms of cooked and uncooked wheat samples

Samples	First endothermic peaks			Second endothermic peaks		
	T_o (°C)	T_p (°C)	T_e (°C)	T_o (°C)	T_p (°C)	T_e (°C)
Uncooked soft wheat	35.4	40.0	45.8	56.2	63.9	76.6
Uncooked hard wheat	–	–	–	56.4	63.5	74.4
Soft 110 °C 20–120 min	41.3	48.9	57.9	62.4	69.6	79.1
Soft 120 °C 20–120 min	42.2	49.1	58.6	63.9	69.7	79.2
Soft 130 °C 20–120 min	41.4	46.7	55.1	–	–	–
Soft 140 °C 20–120 min	41.2	47.8	56.6	–	–	–
Hard 110 °C 20–120 min	41.3	48.2	57.1	61.5	68.8	78.6
Hard 120 °C 20–120 min	38.8	46.3	55.8	64.0	71.3	81.6
Hard 130 °C 20–120 min	41.6	47.6	55.9	–	–	–
Hard 140 °C 20–120 min	41.9	48.1	55.2	–	–	–

The temperatures are shown on an average basis of all cooking times for each cooking temperature.

Generally, when using conventional DSC for starch/water mixture, a single endothermic peak is observed in the mixture with excess water (the volume fraction of polysaccharide is less than about 0.5). However, with limited water, two endothermic peaks are apparent (Biliaderis, 1990; Donovan, 1979; Lelièvre & Liu, 1994). Some investigators found two endothermic peaks in excessive water system and three peaks in limited water system (Fukuoka, Ohta, & Watanabe, 2002; Münzing, 1991). In this paper, a cooked wheat system, which is more complex than simple starch/water system, was studied. The first peak was found in most samples and it was interpreted in terms of the endothermic relaxation of the protein fraction (Cuq & Icard-Verniere, 2001) and the second peak was judged to be associated with starch gelatinisation as it was not detected in wheat samples which had been cooked for longer times (100–120 min) or at high temperatures (130–140 °C). The enthalpy observed from the second peaks was used to quantify the DG (Eq. (1)).

In this study, when wheat grains were cooked as described in Section 2, starches were quickly gelatinised. Gelatinisation was complete within 60–80 min when cooking at 110–120 °C and within 20 min when cooking at 130–140 °C. The graphs of DG for soft and hard wheat grains cooked at 110–120 °C are shown in Fig. 2. Notably data for samples cooked at 110–120 °C for 100–120 min and at 130–140 °C for all cooking times are not shown in this figure as starch is completely gelatinised under these conditions.

Under the same cooking conditions, hard wheat provided a higher DG than soft wheat, due to the lower starch content and the influences from other components of the hard wheat. Protein and flour components (other than starch) have been found to delay the starch transition reaction. Eliasson (1983) studied a starch–gluten system and found that the gelatinisation peak temperature of the starch increased and the enthalpy decreased in the presence of gluten protein. Mohamed and Rayas-Duarte (2003) reported that higher amounts of protein in the wheat starch–protein/gluten blend increased the onset and peak temperatures and decreased enthalpy of the starch gelatinisation, as measured by DSC. In this study, similar patterns of enthalpy values were

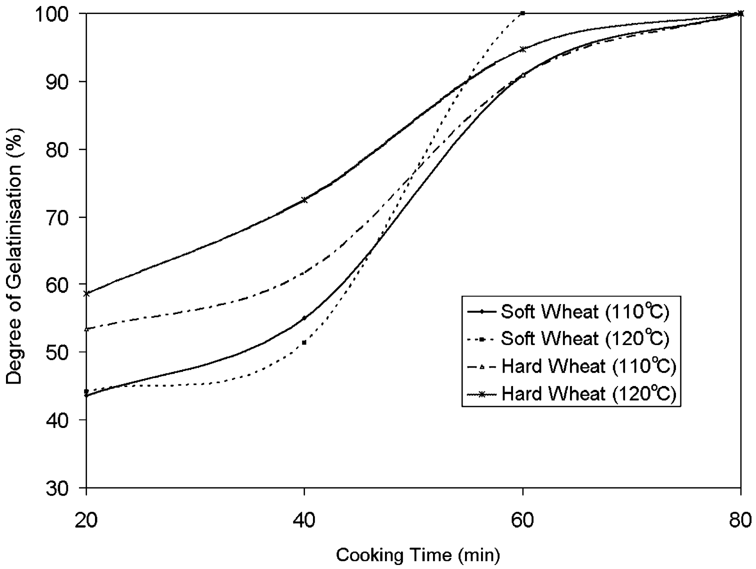

Fig. 2. Percentage degree of gelatinisation of soft and hard wheat grains cooked under pressure at 110 and 120 °C for 20, 40, 60 and 80 min.

observed. The lower enthalpy obtained from hard wheat samples resulted in the higher DG when compared to soft wheat samples, obtained from the same cooking conditions. However, the results of onset and peak temperatures did not clearly agree with the findings from Eliasson (1983) and Mohamed and Rayas-Duarte (2003) (see Table 1). Chevallier and Colonna (1999) stated that phase transitions in water–protein–starch mixtures (similar to whole-wheat system in this study) are varied and complex, therefore, DSC results need careful interpretation.

Also, it should be noted that this study involved the cooking of wheat grains on a laboratory scale and without any other ingredients. However, in an industrial environment, on a large scale with additive ingredients, e.g. malt extract, salt, fortification agents may be used. This could decrease the DG in the whole system. The presence of sugars and sodium chloride has been found to inhibit gelatinisation reaction as well as increase gelatinisation temperature. The inhibition depended on a concentration of sugars and salts together with starch/water mixture ratio

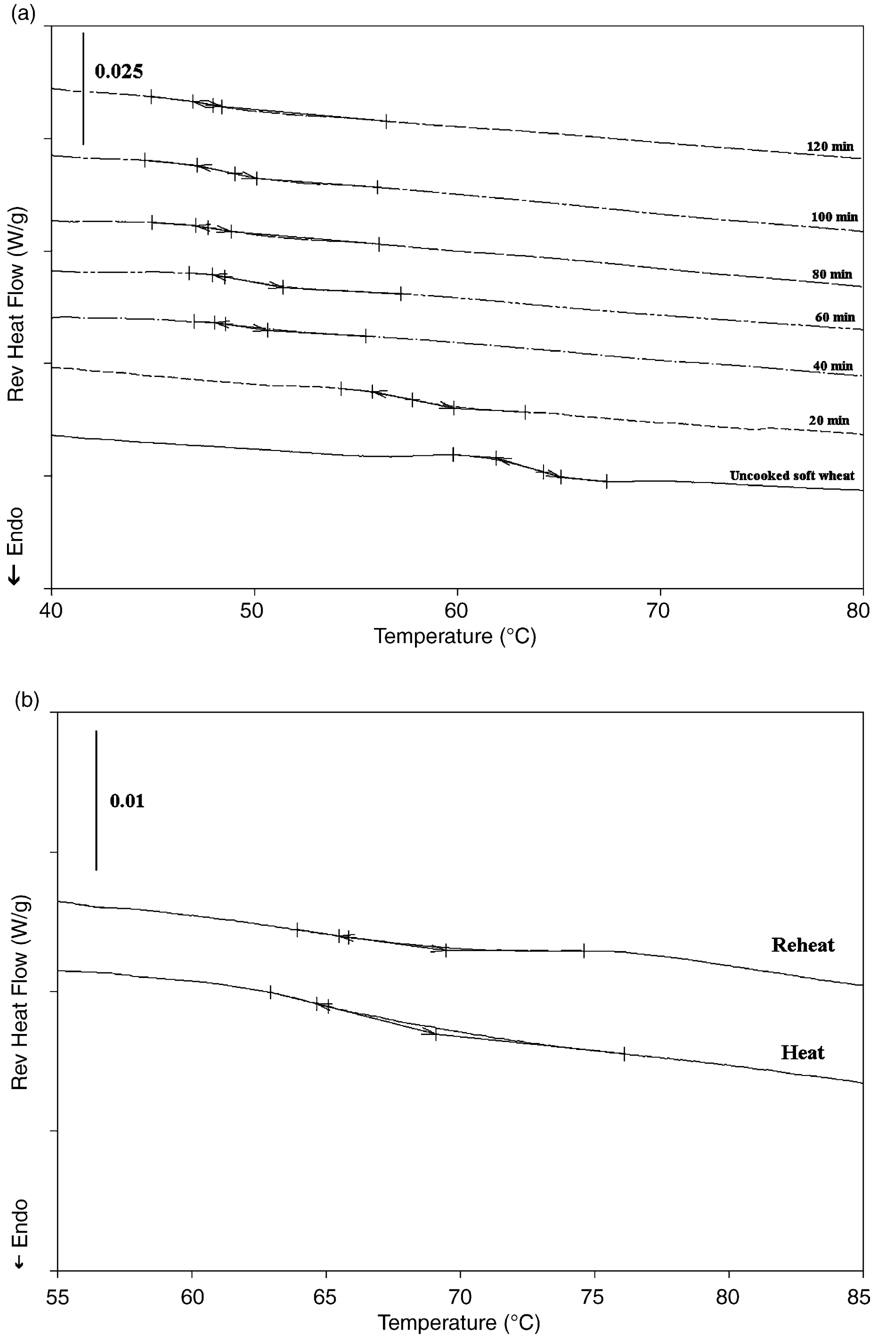

Fig. 3. (a) Reversible heat flow thermograms of cooked wheat samples (from soft wheat grains cooked at 120 °C for 20–120 min and uncooked sample) and (b) reversible heat flow thermograms of cooked wheat samples during heating and reheating (from hard wheat grains cooked at 110 °C for 20 min).

Table 2
Onset, inflection and end temperatures (T_o, T_i and T_e) including ΔC_p from reversible heat flow thermograms of cooked and uncooked wheat samples

Samples	T_o (°C)	T_i (°C)	T_e (°C)	ΔC_p (J/g per °C)
Uncooked soft wheat	62.0	64.2	65.9	0.065
Uncooked hard wheat	62.8	64.4	66.7	0.042
Soft 110 °C 20–120 min	64.6	65.7	68.1	0.029
Soft 120 °C 20–120 min	64.1	66.9	68.8	0.029
Soft 130 °C 20–120 min	59.1	62.5	63.7	0.030
Soft 140 °C 20–120 min	45.0	48.2	49.9	0.041
Hard 110 °C 20–120 min	62.2	63.4	65.3	0.025
Hard 120 °C 20–120 min	62.9	64.8	66.3	0.022
Hard 130 °C 20–120 min	59.7	62.1	63.8	0.024
Hard 140 °C 20–120 min	45.6	47.7	49.6	0.035

The temperatures and heat capacity changes are shown on an average basis of all cooking times for each cooking temperature.

which consequently gave different patterns of DSC thermograms (Buck & Walker, 1988; Chinachoti, Kim-Shin, Mari, & Lo, 1991; Jang et al., 2001; Lund, 1984; Maaurf, Che Man, Asbi, Junainah, & Kennedy, 2001; Perry & Donald, 2002; Wootton & Bamunuarachchi, 1980). For reversible heat flow, most samples showed a small endothermic baseline shift localised broadly around 48–67 °C. Typical thermograms are shown in Fig. 3a. The thermogram from a selected sample showing this reversible event is given in Fig. 3b. The shifts of endothermic baseline were characterised by the onset, inflection and end temperatures together with heat capacity changes (ΔC_p) and these are summarised in Table 2. The reversible endothermic baseline shifts found in this study relate to the glass transition of the cooked wheat samples. The glass transition temperature (T_g) is relatively in the same range as the starch gelatinisation temperature (second peak for non-reversible heat flow thermograms). Biliaderis, Page, Maurice, and Juliano (1986) and Slade and Levine (1988) believed that the glass transition occurred immediately prior to gelatinisation. However, Zeleznak & Hoseney, (1987) explained that heat capacity changes during gelatinisation were related to granule swelling and the initiation of crystallite melting and reported that T_g was lower than the gelatinisation temperature in native wheat starch. Interestingly, these studies were conducted using conventional DSC. Tan et al. (2004a) used MTDSC to study starch gelatinisation in four types of maize starch and consistently obtained T_g to be within the gelatinisation range. The determination of T_g for complex food systems such as cereal products is difficult. The use of DSC to observe the state transition of cereal products generally gives a broad signal with a small change in heat capacity (Laaksonen & Roos, 2003) and MTDSC is reported to be less sensitive for the T_g of pasta and corn flake at low moisture contents Bell & Touma, (1996). Laaksonen & Roos, (2003) mentioned that other techniques such as dynamic–mechanical (thermal) analysis (DMA/DMTA) or dielectric (thermal) analysis (DEA/DETA) are more sensitive than DSC for state transition determination. The specific heat capacity values of the uncooked and some selected cooked wheat samples are shown in Fig. 4. Specific heat capacity is the amount of heat required to raise the temperature of a unit mass of materials (cooked wheat grains in this study) by 1 K and this is useful for understanding the role of heat transfer in the cooking process. It was found that cooking process affected the specific heat capacity values of the samples. Cooked

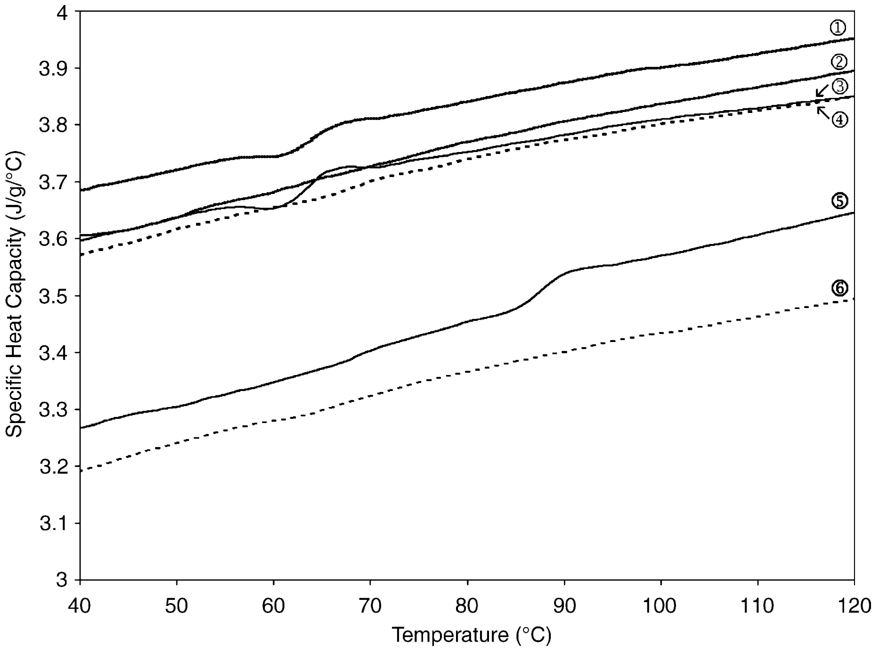

Fig. 4. Specific heat capacity values of some cooked and uncooked wheat samples as a function of temperature; (1) uncooked hard wheat, (2) soft wheat 120 °C 40 min, (3) uncooked soft wheat, (4) soft wheat 110 °C 40 min, (5) hard wheat 120 °C 40 min, (6) hard wheat 110 °C 40 min.

wheat grains from different cooking conditions gave different values of specific heat capacity. Specific heat capacity values of uncooked hard wheat are generally higher than those from uncooked soft wheat, in contrast to cooked samples, possibly due to the difference in their constituents and the ways that those constituents were affected by cooking process. Published studies of the specific heat capacity of complex food systems determined by experimental measurements are limited. Much of work focused on starch–water system (Noel & Ring, 1992; Pyda, 2001; Sopade & LeGrys, 1991) and/or starch–water system with plasticiser, e.g. glycerol (Tan, Wee, Sopade, & Halley, 2004b). There is a need for more studies in this area and MTDSC could be a useful tool.

4. Conclusion

MTDSC proved to be a useful tool for investigation of wheat grain cooking process. Typically, two endothermic baseline shifts for non-reversible heat flow thermograms were obtained for uncooked and cooked wheat samples. The first peak localised around 40–50 °C while the second peak localised around 60–70 °C and the latter was associated with starch gelatinisation. It was found that gelatinsation was complete within 60–80 min for cooking temperatures at 110–120 °C and within 20 min for cooking temperatures at 130–140 °C. The use of MTDSC in this study was also able to detect a possible glass transition which occurred during starch gelatinisation, even though there were relatively small changes in heat capacity (0.02–0.06 J/g per °C) and a broad range of T_g (48–67 °C). The specific heat capacity values of the samples were found to be affected by the cooking process and wheat types.

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