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Gelatinisation of sago starch in the presence of sucrose and sodium chloride as assessed by differential scanning calorimetry

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Abstract

The inhibitory effect of sucrose and sodium chloride on sago starch gelatinisation was investigated by differential scanning calorimetry (DSC). The temperature of gelatinisation of starch in the presence of low levels of water and high levels of sucrose was found to increase in the presence of sucrose, whereas the gelatinisation enthalpy was unaffected. The gelatinisation temperature range was not as broad in the presence of sucrose as without sucrose. Furthermore, the shape of the gelatinisation endotherm was changed by the addition of sucrose. The double endotherm obtained in limited water:starch systems was changed into a single endotherm, similar to the endotherm obtained in excess water:starch systems at a higher temperature. DSC was also used to examine the effects of water and sodium chloride content on the phase transitions of sago starch. Samples were adjusted to starch:water ratios of 2:3 and 3:2 in sodium chloride concentrations of 0.0, 1.0, 2.0, 3.0, 4.0, 5.0 M. The gelatinisation temperatures of sago starch increased and then decreased as the sodium chloride concentration increased. Sodium chloride created similar effects on the endotherms in excess water content and on the first endotherm with limited water content. In the presence of sucrose and sodium chloride, gelatinisation shifted to higher temperatures, and enthalpy associated with the endothermic process decreased. The extent of temperature shift and enthalpy change was dependent on the water to starch to solutes ratios. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Sago starch; Gelatinisation; Sucrose; Sodium chloride

1. Introduction

Studies have proposed that sugars increase the gelatinisation temperature (T_p) of starches by various means, such as their ability to compete for water against starch and to reduce the system's water activity (D'Appolonia, 1972; Derby, Miller, Miller & Trimbo, 1975), the sugar–starch interaction (Lelievre, 1976; Spies & Hoseney, 1982; Hansen, Paukstelis, & Setser, 1987; Hansen, Setser & Paukstelis, 1989) and the increase in free volume, resulting in less plasticizing effect of sucrose–water solvent (Levine & Slade, 1988; Levine & Slade, 1989).

Starch gelatinisation in the presence of small sugars has been described as a mechanical relaxation process affected by the mobility of aqueous sugar solutions. The retardation effect of concentrated sugar solution on, for example, the glass transition temperature (T_g) governing starch gelatinisation has been suggested to result from 'antiplasticization' by sugar water co-solvent, relative to the extent of plasticization by water alone (Slade & Levine, 1988). This retardation effect has also been attributed to the capability of sugar to limit water availability to the starch granule. It has been known for decades that various sugars, including sucrose, fructose, and glucose, raise the temperature of starch gelatinisation and delay the increase in viscosity (pasting) and that the effect on the gelatinisation phenomenon increases with increasing sugar concentration (Chungcharoen & Lund, 1987; Paredez-Lopez & Hemandez-Lopez, 1991; Eliasson, 1992).

The differential scanning calorimetry (DSC) technique shows that the gelatinisation temperature increases, and that the swelling of starch granules decreases in the presence of sugars. Different sugars and salts have been reported to exert this effect to different extents when compared on a molar basis (Ahmed & Williams, 1999a and b). Sucrose is commonly reported to have a greater effect than other disaccharides (Savage & Osman, 1978). Generally, monosaccharides delay gelatinisation less than disaccharides,

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Table 1
Effect of sucrose on the gelatinisation parameters of sago starch differing in water:starch ratio. All samples are in triplicate ± standard deviation. T_o — Onset temperature, T_p — Peak temperature, T_m — Melting/Conclusion temperature. $\Delta T = T_m - T_o$, ΔH = Enthalpy

g sucro/g starch	Water:starch ratio	T_o (°C)	T_p (°C)	T_m (°C)	ΔT (°C)	ΔH (J/g starch)
0.0	3:1	64.4 ± 0.4	70.4 ± 0.2	80.4 ± 0.3	15.9	5.37 ± 0.27
0.2		66.5 ± 1.0	72.8 ± 1.0	82.1 ± 0.9	15.6	4.99 ± 0.94
0.4		68.6 ± 0.3	73.6 ± 0.1	82.3 ± 0.1	13.6	5.13 ± 0.44
0.5		69.9 ± 0.9	74.3 ± 0.2	82.4 ± 0.1	12.4	5.09 ± 0.03
0.6		71.2 ± 0.1	77.0 ± 0.8	87.3 ± 0.3	16.1	5.19 ± 0.14
1.0		74.4 ± 0.1	79.2 ± 0.1	87.9 ± 1.0	14.1	4.82 ± 0.21
1.7		78.0 ± 0.2	85.2 ± 0.1	91.7 ± 0.4	13.6	4.30 ± 0.12
0.0	1:1	65.7 ± 1.0	69.9 ± 0.8	90.6 ± 0.1	24.9	3.43 ± 0.27
0.2		69.1 ± 0.2	76.2 ± 1.0	92.3 ± 1.3	23.2	3.32 ± 0.24
0.4		73.1 ± 0.5	80.9 ± 0.6	95.3 ± 1.3	22.1	2.70 ± 0.19
0.5		75.6 ± 0.1	83.3 ± 0.2	97.1 ± 0.2	21.4	2.77 ± 0.21
0.6		78.8 ± 0.4	86.5 ± 0.6	98.7 ± 0.1	19.8	3.50 ± 0.47
1.0		92.3 ± 0.7	101.4 ± 1.1	109.6 ± 0.5	17.3	2.52 ± 0.34
1.7		105.1 ± 0.1	112.7 ± 0.5	118.7 ± 0.4	13.6	2.04 ± 0.05

except maltose, which acts like monosaccharides. Many workers have observed that the addition of sucrose, which dissolves in the water, has the functional effect of increasing solvent volume (Spies & Hoseney, 1982; Ghiasi, Hiseney & Varriano-Marston, 1983).

At water:starch ratios in the range of 10:1 to 2:1 the effect on the DSC endotherm of adding sucrose is an increase of the gelatinisation temperature. Most workers reported an increase in the onset (T_o), peak maximum (T_p) and melting (T_m) temperatures (Evans & Haisman, 1982; Kim, Hansen & Setser, 1986; Chungcharoen & Lund, 1987; Buck & Walker, 1988) whereas Wootton and Bamunuarachchi (1980) only noticed an effect on T_p. The enthalpy of gelatinisation (ΔH), as reported using DSC, has been found to be

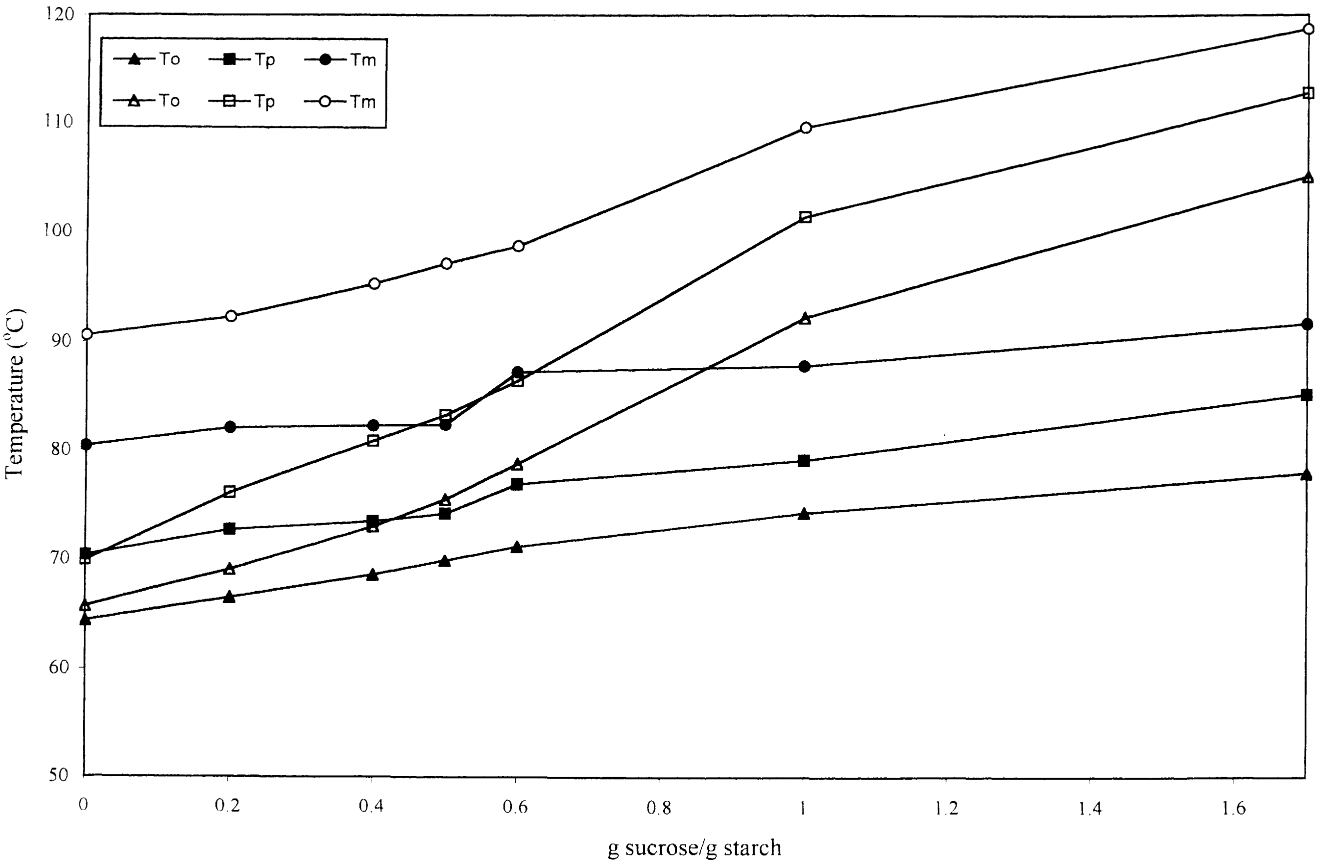


Fig. 1. The influence of sucrose on the gelatinisation temperature interval of sago starch. -▲- -■- -●- water-to-starch ratio 1:1; -△- -□- ○- water to starch ratio 3:1. (T_o — Onset, T_p — Peak and T_m — Melting Temperatures).

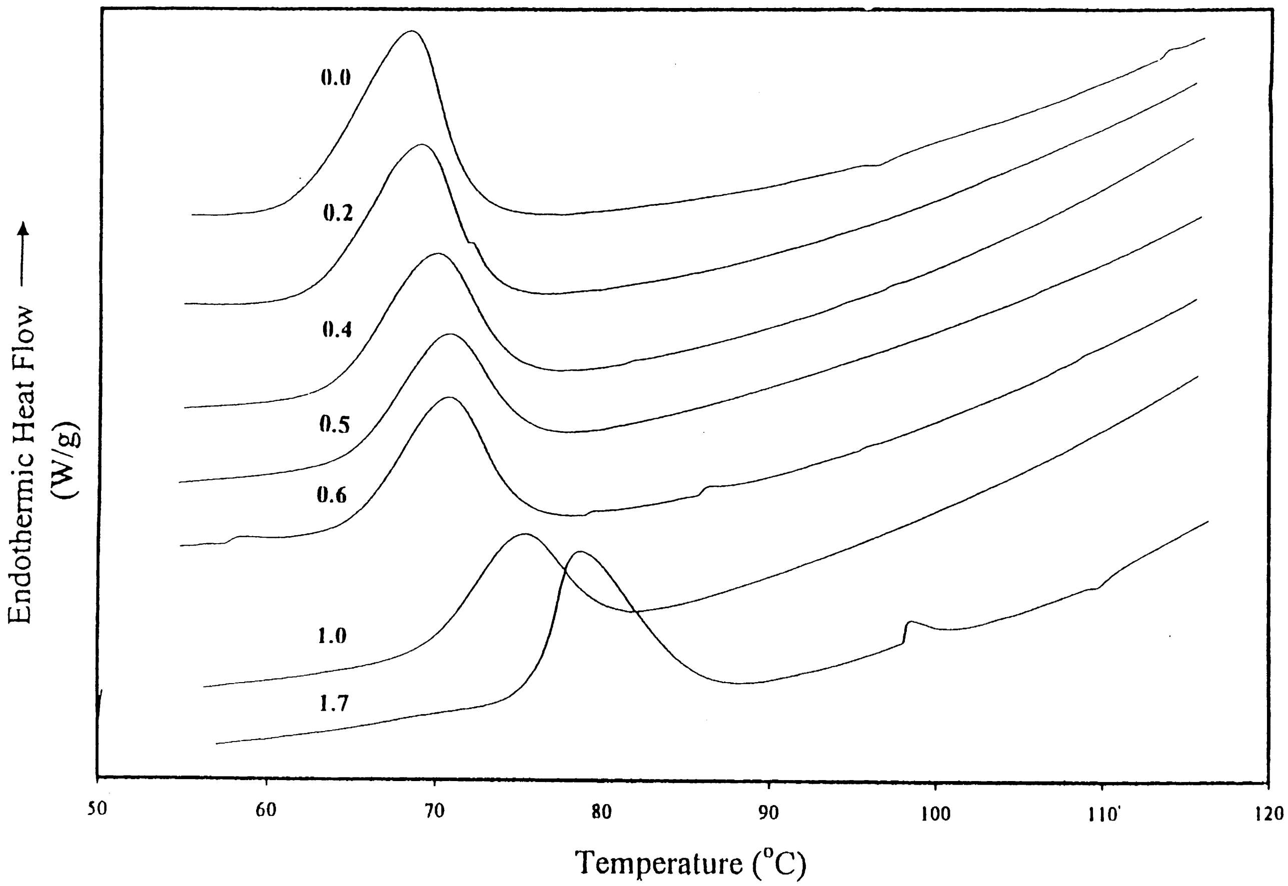


Fig. 2. The DSC thermograms of sago starch at different levels of sucrose (numbers indicate sucrose to starch ratios). The water to starch ratio was 3:1.

unaffected by the addition of sugars (Eliasson, 1992). In these experiments the water levels used have been fairly high, as indicated above, whereas the sugar levels have not been very high. For example, Chungcharoen and Lund (1987) used a sucrose to starch ratio of 0.4:1 and Buck and Walker (1988) used a sucrose to starch ratio of 1:1.

Sodium chloride (NaCl) had a different effect on starch gelatinisation. When the sodium chloride concentration increased, the enthalpy value decreased (Bello-Perez & Paredes-Lopez, 1995). Chungcharoen and Lund (1987) reported that sodium chloride was found to increase the gelatinisation temperature and decrease the enthalpy of transition in all rice flour and isolated rice starch samples. Wootton and Bamunuarachchi (1980) reported that sodium chloride appeared to exhibit a maximum inhibitory effect on starch gelatinisation at a concentration of 6–9%. They explained that the decrease in enthalpy when salt was added could arise from the influence of sodium and chloride ions on water, starch and their interactions. When they examined samples under polarised light, the loss of birefringence in the samples was delayed as a result of addition of sodium chloride.

According to Oosten (1982), when sodium chloride was added to a starch of flour suspension, some alcoholic groups in the starch granules were converted to sodium alcoholate groups. These compounds were better dissociated, thus causing a rise in the Donnan potential, which more effectively excluded the chloride ions from the granules. However, the absorption of sodium ions was rather limited, hence, the increase in gelatinisation began (at a higher temperature) with chloride ions assisting gelatinisation by breaking hydrogen bonds between starch chains.

Evans and Haisman (1982) found that sodium chloride and calcium chloride at low concentrations increased slightly the gelatinisation temperatures, but when the concentrations increased further the gelatinisation temperatures decreased. Salt has also been observed to reduce the degree of gelatinisation as well as raise the gelatinisation temperature (Wootton & Bamunuarachchi, 1980). Oosten (1982) suggested that starch acts as a weak acid ion-exchanger and that cations tend to protect and stabilise the granule structure. On the other hand, anions act as gelatinising agents by rupturing hydrogen bonds. Jane (1993) proposed that starch gelatinisation in such salts seems to be controlled by two effects: water structure and electrostatic interaction between starch and ions. These two effects conflict with one another and result in complex patterns depending on the concentration of the salts.

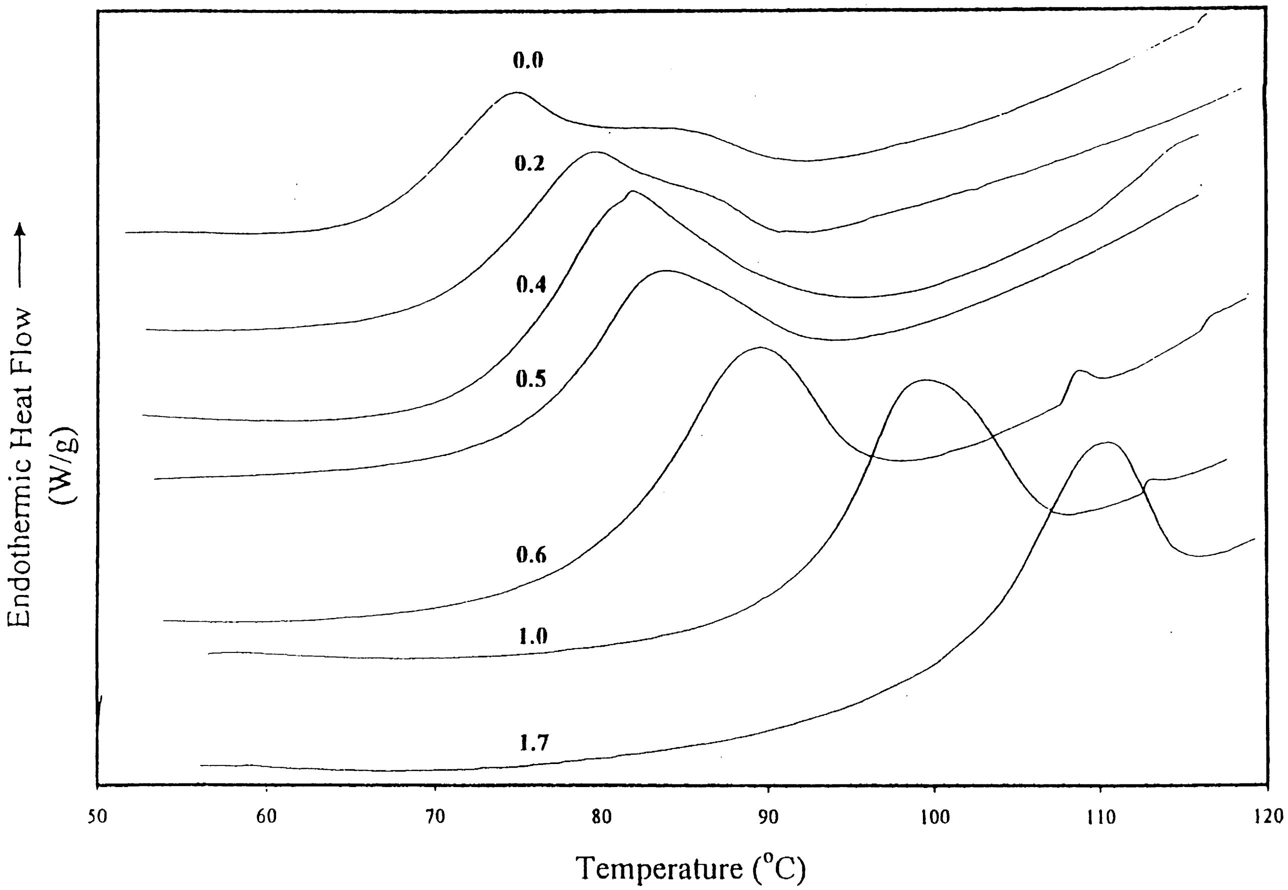


Fig. 3. The DSC thermograms of sago starch at different levels of sucrose (numbers indicate sucrose to starch ratios). The water to starch ratio was 1:1.

The aim of the present investigation was to study sago starch gelatinisation at low water contents and high sugar and sodium chloride levels. The changes in the DSC endotherm from the double endotherm into the narrow single endotherm were also examined in some detail.

2. Materials and methods

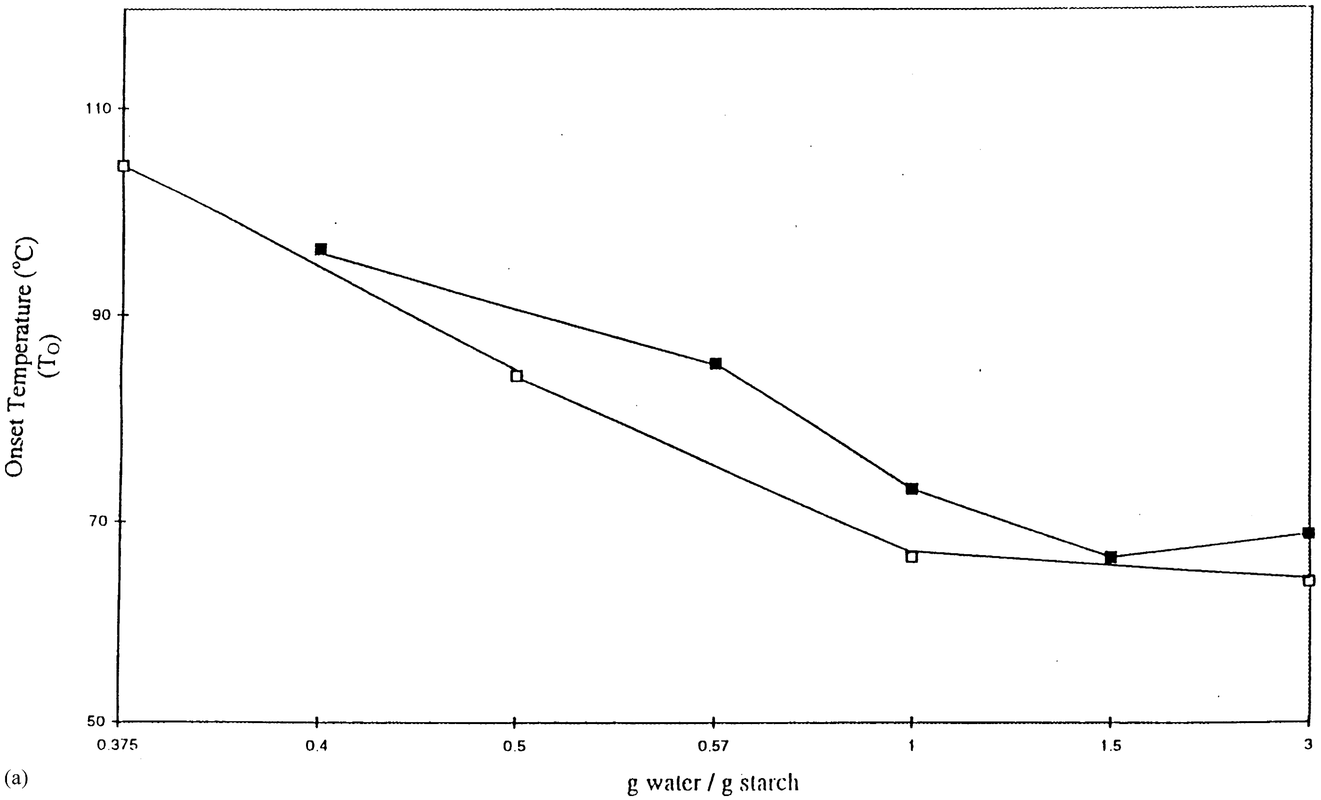
2.1. Materials

Sago starches (*Metroxylon sagu*) used were obtained from PPES Sago Industries (Mukah, Sarawak, Malaysia). Sucrose and sodium chloride (NaCl) were AR grade reagents and deionised distilled water was used.

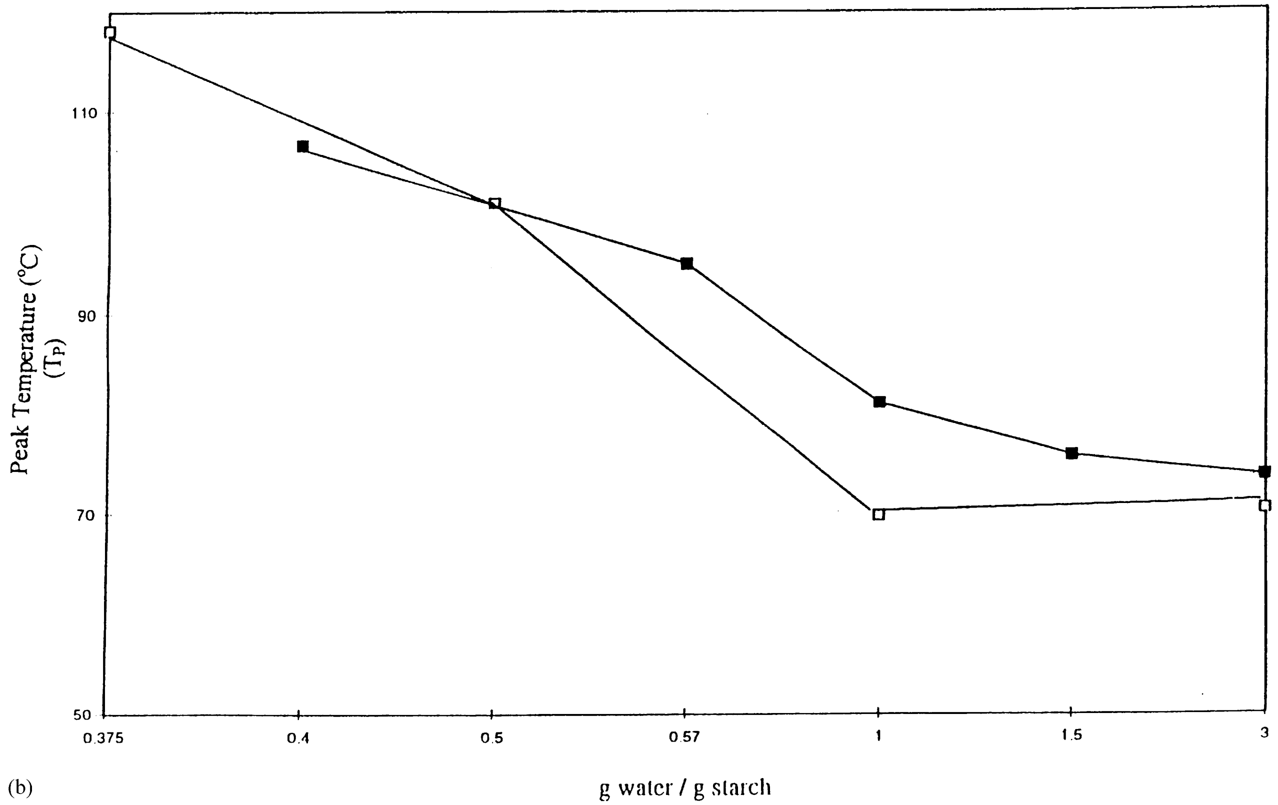
2.2. Methods

Sago starch was weighed into a tarred, aluminium DSC sample pan and sugar solution and/or water was added using a microsyringe to give a precalculated water:starch ratio (3:1 or 1:1) and sugar:starch ratio until the granules were wetted and uniformly distributed across the bottom of the pan. Sugar solutions of the highest concentration possible, and not crystalline sugar, were used in order to obtain DSC thermograms without peaks caused by dissolution of the sugar. In the case of sucrose, it was possible to study sugar levels up to 1.7 g sucrose/g starch. For sodium chloride, samples were adjusted to sago starch:water ratios of 2:3 and 3:2, and concentrations of sodium chloride were prepared at 1.0, 2.0, 3.0, 4.0 and 5.0 M in the aqueous phases of starch suspension.

The final weighed samples (9.0–11.0 mg) were immediately hermetically sealed using a volatile sample sealer accessory. All samples were analysed within 1 h of sample preparation. The DSC measurements were carried out in a Perkin Elmer DSC-7 with a heating rate of 10°C/min. Heating rates in the region 8–15°C are considered normal (Mackenzie, 1970). Melting points and enthalpies for indium (m.p. 156.6°C, ΔH_m 28.5 J/g) and *n*-dodecane (m.p. 96.5°C, ΔH_m 218.73 J/g) were used for temperature and heat quality capacity calibration. An empty pan was used as a reference to balance the heat capacity of the sample pan. The analysis was started at 20°C and conducted up to 130°C. This upper end temperature was above the gelatinisation peak. The characteristic of the onset (T_o), peak (T_p), and melting (T_m) temperatures were determined. The gelatinisation enthalpy was expressed in J/g starch. The results given are the mean of three runs (triplicate).



(a)



(b)

Fig. 4. The influence of water content on the gelatinisation temperature interval (a) (T_o — Onset Temperature) of sago starch. -■- 0.4 g sucrose/g starch, -□- No sucrose added. (b)(T_p — Peak Temperature) of sago starch. -■- 0.4 g sucrose/g starch, -□- No sucrose added. (c) (T_m — Melting Temperature) of sago starch. -■- 0.4 g sucrose/g starch, -□- No sucrose added.

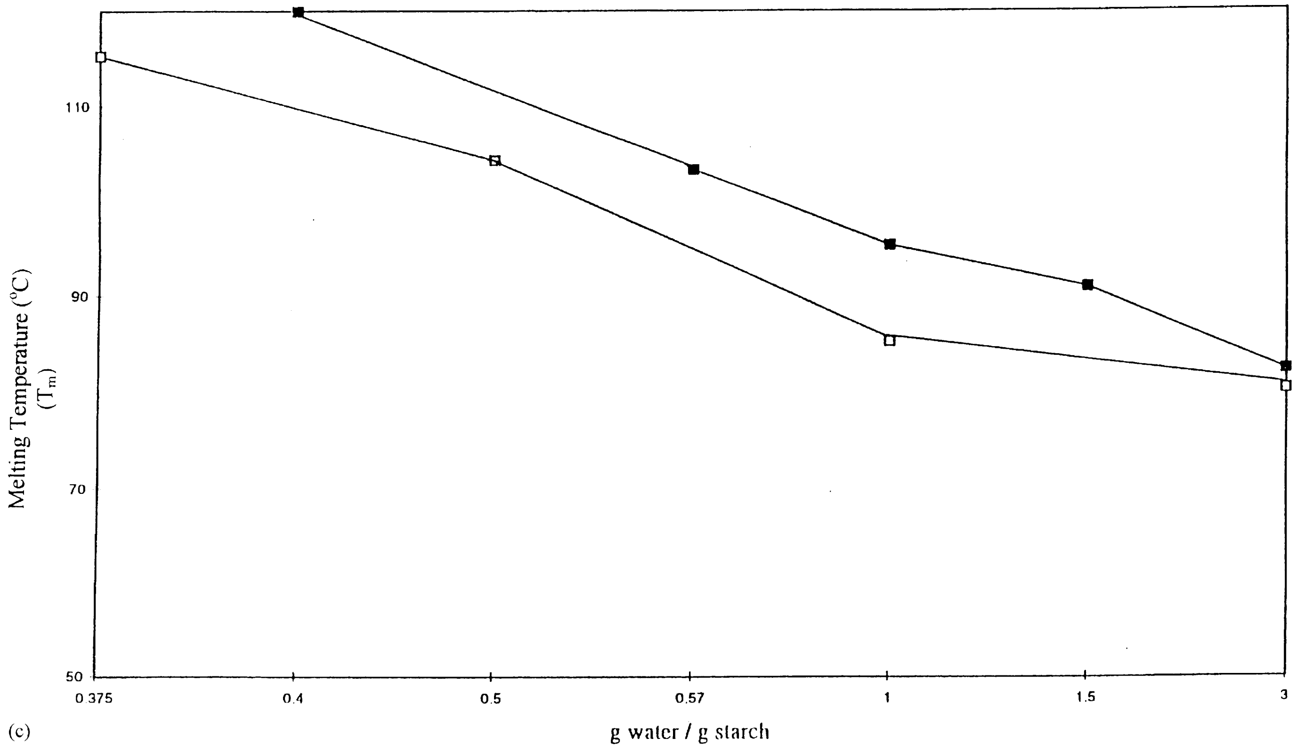


Fig. 4. (*continued*)

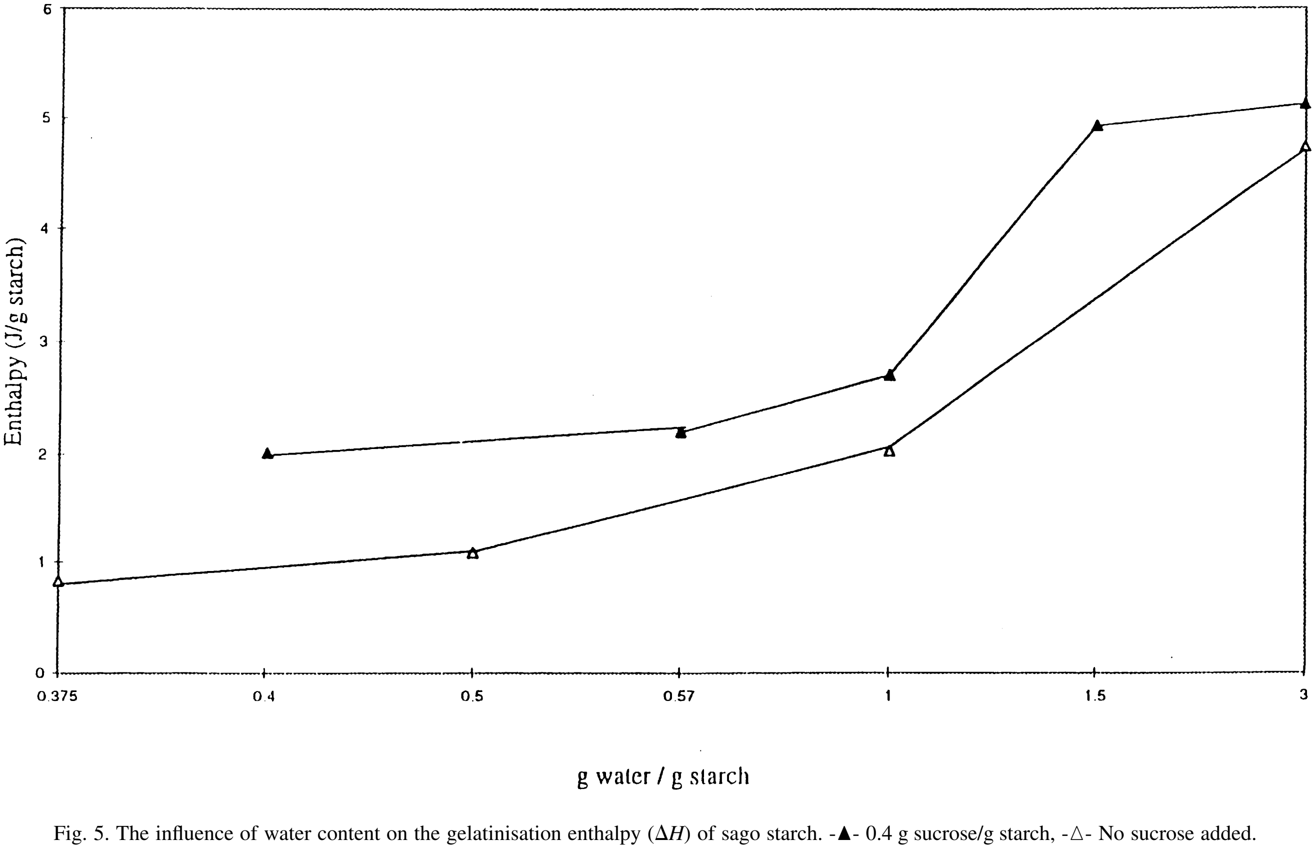


Fig. 5. The influence of water content on the gelatinisation enthalpy (ΔH) of sago starch. -▲- 0.4 g sucrose/g starch, -△- No sucrose added.

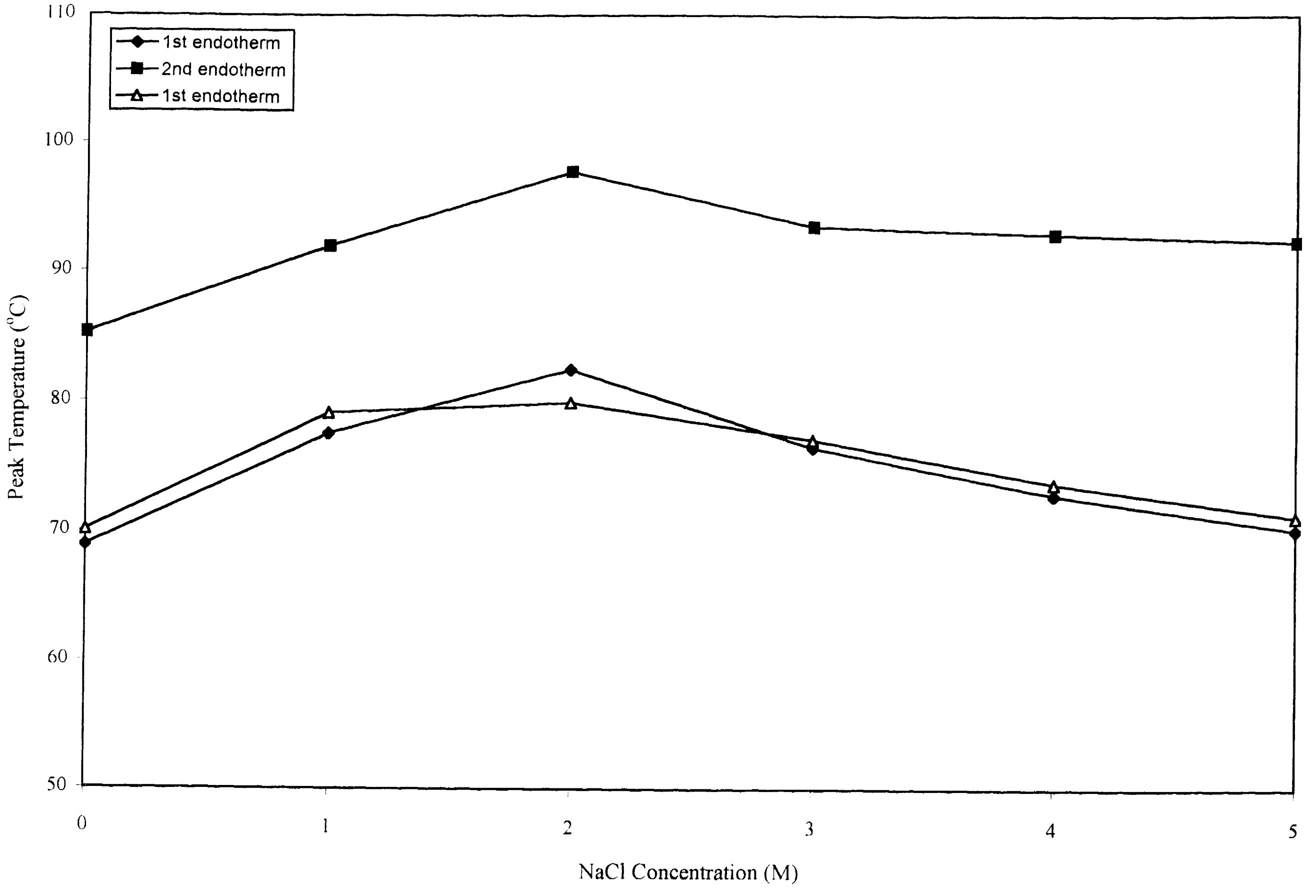


Fig. 6. Effect of salt on peak temperature (T_p) of phase transition. -◇- -■- Starch/water ratio 3:2, -△- starch/water ratio 2:3.

3. Results and discussion

3.1. Effect of sucrose on starch gelatinisation

Sucrose affects the DSC endotherm in different ways, depending on the starch–water level. The type of sugar and concentration are known to affect gelatinisation (Figs. 2 and 3). The principal effect of sugar on starch is to raise its gelatinisation temperature. In systems with excess water (i.e. a single, narrow endotherm in the starch–water systems) sucrose causes an increase in T_o, T_p and T_m while $\Delta T((T_m - T_o)$ is more or less unaffected (Fig. 2).

When increasing levels of sucrose were added to sago starch at a water:starch ratio of 3:1, T_o, T_p and T_m were all increased. ΔH for samples of ratios from 0.0 to 0.6 g sucrose/g starch remained unchanged. T_o, T_p and T_m showed a decrease for samples with ratios of g sucrose/g starch from 1.0–1.7. However, ΔH were not very much affected (Table 1). The enthalpy of gelatinisation (ΔH) has been reported to be unaffected by the addition of sugars (Evans & Haisman, 1982) but increased (Kohyama & Nishinari, 1991) as well as decreased values (Wootton & Bamunuarachchi, 1980; Kim et al., 1986; Chungcharoen & Lund, 1987; Buck & Walker, 1988) have also been reported.

Sucrose delays the start of the gelatinisation process. For example, from 0.0–0.2 g sucrose/g starch the delay of gelatinisation temperature ranged between 2.1 and 3.4°C at water:starch ratio 3:1 and 1:1. T_o, T_p and T_m as a function of the sucrose to starch ratio are shown in Fig. 1. ΔH was 5.37 ± 0.27 J/g starch without added sucrose and 4.30 ± 0.12 J/g starch at the sucrose to starch ratio 1.7:1. The shape of the endotherms did not change with the increase in sucrose level (Fig. 2). These results are in agreement with the results reported by Eliasson (1992). In their investigation, T_o increased from about 58.3°C for potato starch at a water to starch ratio of 3:1 to about 72.1°C at a sucrose to starch ratio of 1.7:1. In the present investigation of sago starch (Fig. 1), T_o increased from 64.4°C at a water to starch ratio of 3:1 to 78.0°C at a sucrose to starch ratio of 1.7:1.

The effect was even more pronounced at the water to starch ratio of 1:1 (Fig. 1). The delaying effect of sucrose was, thus, greater at lower water:starch ratios. Chungcharoen and Lund (1987) also observed this when sucrose was added to rice starches. In limited water systems (i.e. a double endotherm is obtained for the starch water mixture) T_o, T_p and T_m increase when sucrose was added but ΔT decreases. This means that the double endotherm was changed into the type of endotherm obtained in excess water systems. ΔH seems not to be affected by sucrose (Table 1).

The change in T_o, T_p and T_m with increasing sucrose levels

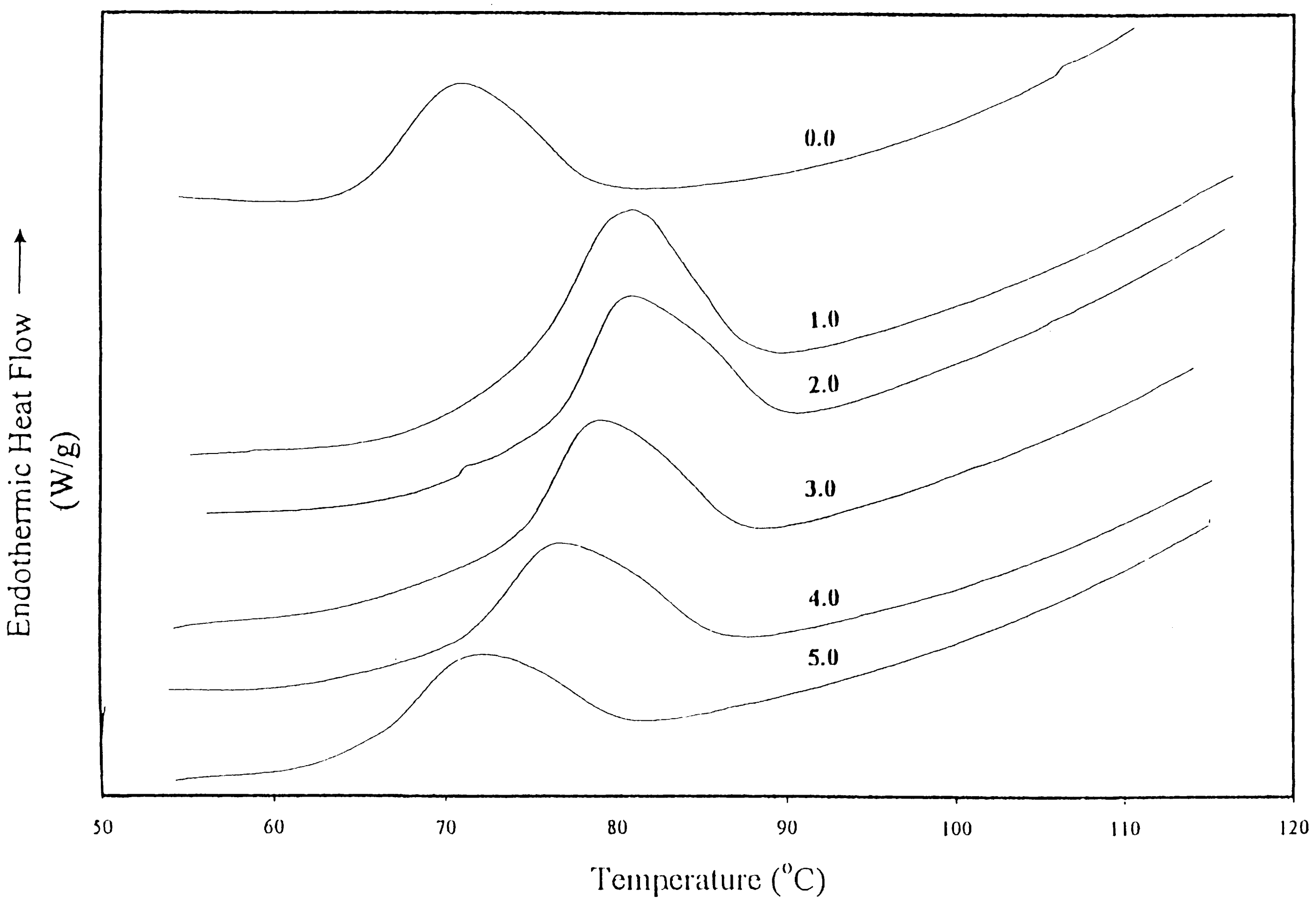


Fig. 7. DSC thermograms of sago starch at a 2:3 starch water ratio. Numbers indicate the molarity of sodium chloride in the aqueous phase of the starch suspension.

at the water:starch ratio 1:1 (Fig. 1) show that the addition of sucrose caused not only a delay in the gelatinisation but also narrowed the temperature range. This is also seen in Fig. 3 where the corresponding DSC thermograms are shown; such thermograms confirm the report by Eliasson (1992). When the sucrose concentration was increased, the shoulder on the high temperature side of the endotherm disappeared. The sucrose addition, thus, had the same effect as increasing the water content. The amount of sucrose necessary to bring about the change from a double endotherm into a single endotherm depends on the type of starch. The reason for the double endotherm has been attributed to the destabilisation of the crystalline domains when the amorphous parts are hydrated, an effect that would be different at low and high water contents (Donovan, 1979). It has been assumed that the first and second peaks (double endotherm) were the melting peaks of starch crystals. This may have been because less dissolved sugar solution was available to produce a single, sharp starch gelatinisation endotherm. Thus a typical gelatinisation endotherm would not be observed. It has also been suggested that the DSC endotherm is composed of a change in heat capacity caused by the glass transition in the amorphous region followed by the melting of crystallites (amylopectin branches) (Slade & Levine, 1988).

In the case of sago starch (water:starch ratio of 1:1) the single endotherm was obtained at the sucrose to starch ratio 0.4:1 without any sign of a shoulder. The disappearance of the shoulder on the high temperature side of the endotherm is thus related to the amount of amylopectin in the starch. Proximate analysis by Roslin and Asbi (1995) indicated that the sago starch contained 75.56–77.26% amylopectin. As amylopectin builds up the crystalline domains in the starch granule (French, 1984), the effect of sucrose seems to be related to the degree of crystallinity of the starch granule. It can be seen from Fig. 3 that at the low sucrose levels (below about 0.6 g sucrose/g starch) T_m is not affected as much as T_o and T_p. ΔH was 3.43 ± 0.27 J/g starch for the sample without added sucrose and at the sucrose to starch ratio 1.7:1 ΔH was 2.04 ± 0.05 J/g starch. In the case of sago starch, ΔH did not depend on the amount of sucrose present (Fig. 5).

The influence of the water content was also investigated, both with (0.4 g sucrose/g starch) and without added sucrose. The gelatinisation temperatures are compared in Fig. 4a–c and the corresponding ΔH values in Fig. 5. The results in Fig. 4a and b show that T_o and T_p do not depend on the water content above a water:starch ratio of 1:1. However, when sucrose was added, T_o and T_p both depend

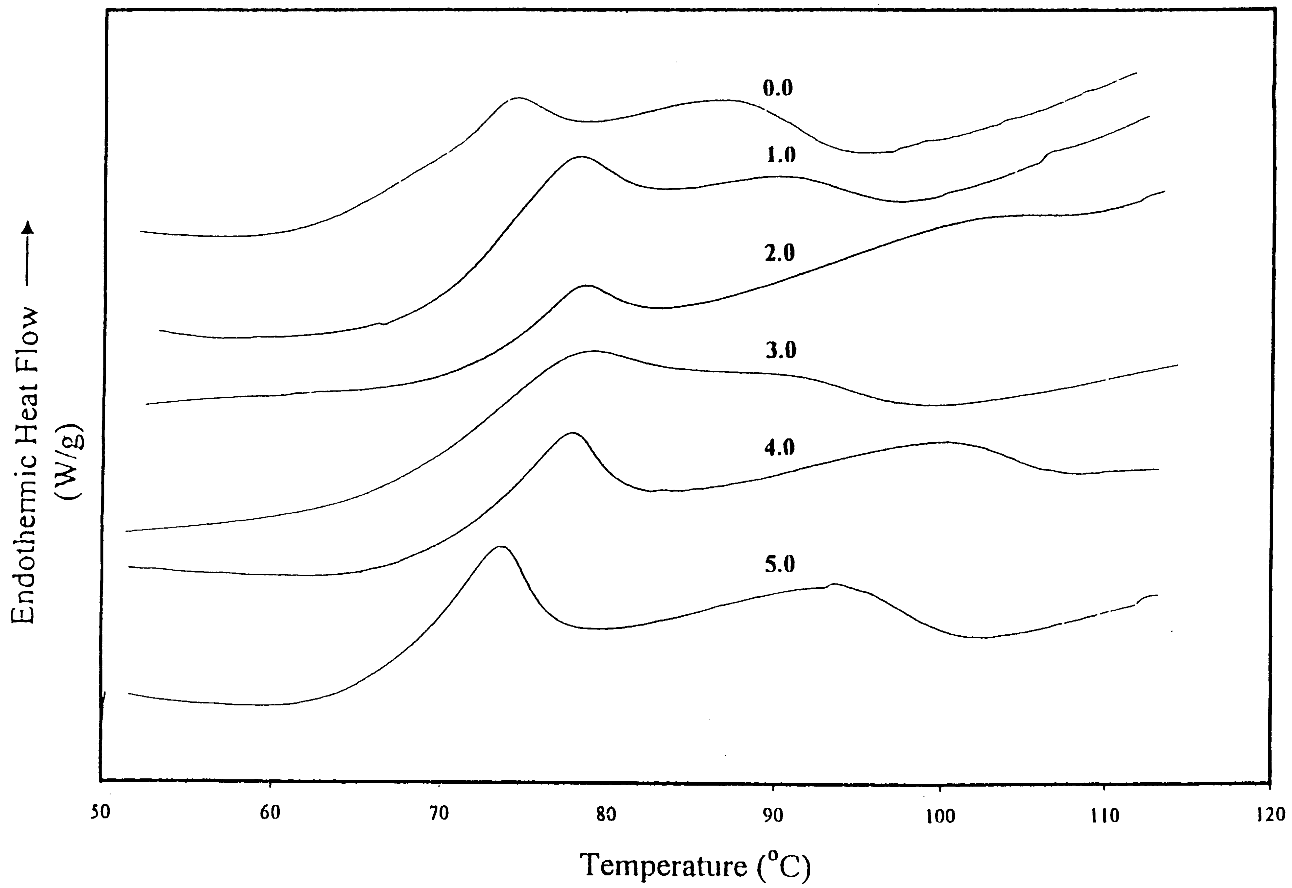


Fig. 8. DSC thermograms of sago starch at a 3:2 starch water ratio. Numbers indicate the molarity of sodium chloride in the aqueous phase of the starch suspension.

on the water content at all water:starch ratios. T_o and T_p were higher in the presence of sucrose at all water to starch ratios. On the other hand, T_m was similar for samples with and without sucrose at high levels of water, but when the water:starch ratio decreased to below 1:1 T_m was higher in the presence of sucrose (Fig. 4c).

In the case of sago starch, it has been argued that sucrose 'binds' water, thus making less water available for starch hydration. Results obtained by DSC at different water:starch ratios have shown that this explanation cannot be correct. This result is similar to those reported by Eliasson (1992) on potato, waxy maize and high amylose maize starches. If the water:starch ratio decreases, T_o remains the same, as does T_p (Eliasson, 1992). At water:starch ratios below 2:1 a shoulder at the high temperature side of the endotherm emerges, and T_m increases. At very low water levels an endotherm at a temperature above the typical gelatinisation temperature range might be observed (Burt and Russell, 1983). If sucrose (or any other sugar) is added, T_o T_p and T_m increase, which should not be the case if the only function sucrose had was to decrease the amount of water available to the starch. The observation that the double endotherm might change into a single endotherm under certain conditions suggests that more water is available to the starch (Ghiasi et al., 1983).

Slade and Levine (1987) suggested that sugar solution acts as an antiplasticizing co-solvent compared to the action of water as a diluent. Thus, greater energy is required to overcome the antiplasticizing effect and melt the crystals. Spies and Hoseney (1982) suggested that the delay in gelatinisation was caused by the decreased water activity of the sugar solution compared with water, and by the molecular size of the sugar. They speculated that starch–sugar interactions played a role. Sugar is supposed to stabilise the starch granule structure (Kim et al., 1986). Evans and Haisman (1982) explained the effect on the gelatinisation temperature by a combination of water activity and volume fraction of water inside the granules.

ΔH decreased with decreasing water content below 1 g water/g starch (Fig. 5) as expected (Donovan, 1979). If the standard deviation (±5%) is taken into account, there seemed to be no difference in the ΔH for samples with and without sucrose at a given water content.

3.2. Effect of sodium chloride on starch gelatinisation

Generally, as the concentration of sodium chloride increased, the gelatinisation temperature increased to a

Table 2
Effect of sodium chloride (NaCl) on the differential scanning calorimetry characteristics of sago starch as a function of starch:water ratio. All samples are in triplicate ± standard deviation. T_o — Onset temperature, T_{p1} — Peak temperature (first endotherm), T_{p2} — Peak temperature (second endotherm), T_m — Melting/Conclusion temperature, ΔH = Enthalpy

NaCl concentrations (M)	Starch:water ratio	T_o (°C)	T_p (°C)	T_m (°C)	ΔT (°C)	ΔH (J/g)
0.0	3:2	64.7 ± 1.0	68.9 ± 0.9	85.3 ± 0.2	94.8 ± 0.1	5.25 ± 0.05
1.0		67.9 ± 0.2	77.5 ± 0.1	91.9 ± 0.2	101.4 ± 0.9	5.03 ± 0.08
2.0		73.9 ± 0.9	82.5 ± 0.1	97.8 ± 0.4	107.4 ± 0.8	4.86 ± 0.47
3.0		68.9 ± 1.2	76.5 ± 0.6	93.5 ± 0.1	104.6 ± 0.3	4.14 ± 0.34
4.0		64.4 ± 1.8	72.8 ± 0.2	92.9 ± 0.4	104.4 ± 0.1	3.65 ± 0.05
5.0		62.4 ± 1.3	70.1 ± 1.0	92.3 ± 0.1	104.0 ± 0.8	2.76 ± 0.27
0.0	2:3	63.8 ± 0.3	70.1 ± 0.2	–	84.2 ± 1.3	5.48 ± 1.00
1.0		71.1 ± 0.9	79.1 ± 0.3	–	91.1 ± 0.6	5.31 ± 0.44
2.0		72.9 ± 0.1	79.9 ± 0.1	–	92.8 ± 0.2	4.42 ± 0.23
3.0		72.0 ± 0.7	77.1 ± 0.3	–	90.9 ± 0.1	4.22 ± 0.61
4.0		70.7 ± 0.1	73.7 ± 0.7	–	90.6 ± 0.6	3.70 ± 0.50
5.0		61.7 ± 0.2	71.1 ± 0.9		84.4 ± 0.1	3.67 ± 0.40

certain level and then decreased as the concentration increased (Fig. 6). When a swelling-inhibitor (e.g. sodium chloride) is added to a starch suspension, some protons of alcohol groups in the starch granule become exchanged by sodium ions. These alcoholates were better dissociated, thus causing a rise in the Donnan potential. However, the exchange capacity of starch for the sodium ion was limited, so the increase of the gelatinisation temperature was also restricted. When the concentration of sodium chloride was raised beyond a certain level, the influence of the anion becomes dominant. This would contribute to a decrease in the gelatinisation temperature of starch (Oosten, 1979, 1982, 1983, 1990.

Sago starch (starch to water ratio 2:3) showed the highest peak temperatures of the endotherms at 1.0–2.0 M concentrations of sodium chloride (Fig. 7). The peak temperatures were raised from 70.2 to 79.9°C as the salt concentration was increased from 0.0 to 2.0 M. At concentrations of 2.0–5.0 M, the peak temperatures of endotherms decreased with an increase of salt concentration, but were still higher than those of their respective starches without added salt.

The profiles of the DSC thermal curves of sago starch of low water content (starch to water ratio of 3:2) showed two endothermic transitions for all sodium chloride concentrations (Fig. 8). The DSC thermogram of sago starch is similar to the result on potato starch reported by Lii and Lee (1993) in the presence of limited amounts of water. The effects of sodium chloride on the peak temperatures of the first endotherms with limited water content (starch to water ratio of 3:2) (Table 2) were similar to those of the first endotherms with excess water (starch to water ratio of 2:3). The first endothermic peak could reflect an entropy-driven swelling process (Donovan, 1979; Blanshard, 1987), implying that changes in water content would not affect the influence of sodium chloride on the swelling of granules during starch gelatinisation.

At a starch:water ratio of 3:2, the second endotherms in sago starch were highest at the concentrations of 2.0 M (97.8°C) and decreased when sodium chloride concentrations were increased (3.0–5.0 M). Sodium chloride was found to decrease the enthalpy of transition in sago starch samples (Table 2). Wootton and Bamunuarachchi (1980) suggested that the decrease in enthalpy when salt was added to the system could arise from the influence of sodium and chloride ions on water, starch and their interactions. Chungcharoen and Lund (1987) found that 2.5% sodium chloride addition to rice flour and starch shifted gelatinisation to higher temperatures, but the enthalpy associated with the endothermic process decreased with salt addition. One probable reason for the above observation would be the inhibition of the opening of crystalline regions in the starch granules by sodium chloride (Ganz, 1965).

In the presence of sucrose or sodium chloride, gelatinisation temperatures of sago starch shifted to higher temperatures and enthalpy decreased. The effect of the presence of solutes in the system was more pronounced in a limited water system. In the addition of sucrose the double endotherm obtained in limited water: starch systems was changed into a single endotherm, similar to the endotherm obtained in excess water: starch systems at a higher temperature. Sodium chloride created similar effects on the endotherms in excess water content and on the first endotherm with limited water content.

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