Rapid communication

Effects of Na_2CO_3 and NaOH on DSC thermal profiles of selected native cereal starches

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Abstract

The effects of Na_2CO_3 and NaOH (at a concentration of 1 g alkalising agent/100 g dry starch) on the gelatinisation properties of 1:4 and 2:3 starch/water mixtures were studied using differential scanning calorimetry (DSC). The starches studied included wheat, rice, waxy rice, corn and waxy corn. The addition of Na_2CO_3 or NaOH resulted in significant increases in transition temperatures (onset, peak and conclusion), associated with starch gelatinisation, suggesting a stabilisation of the starch granules, probably through electrostatic interactions between Na^+ ions and starch hydroxyl groups. The presence of Na_2CO_3 generally caused a significant reduction in gelatinisation enthalpy (ΔH_G) of the higher moisture (1:4) systems, but had no effect in the less hydrated (2:3) systems. NaOH generally had no effect on ΔH_G of 1:4 systems, but gave rise to inconsistent effects in the 2:3 systems. Heating of alkali-containing samples above the gelatinisation temperature range gave rise to an intriguing exothermic peak, the origin of which was attributed to repolymerisation of the products of alkaline hydrolysis of starch. A separate experiment, conducted to investigate the influence of increasing Na_2CO_3 concentration (up to 3 g/100 g starch) on thermal properties of 1:4 waxy rice starch/water systems, showed a gradual increase in gelatinisation transition temperatures, a general decline in ΔH_G, and a marginal decrease in peak temperature of the repolymerisation exotherm. © 2002 Published by Elsevier Science Ltd.

Keywords: Differential scanning calorimetry; Gelatinisation; Starch; *Kansui*; Thermal properties; Repolymerisation

1. Introduction

Alkalising agents are important in the preparation of many traditional starch-based food products. Examples include the use of lime in the production of tortillas and *kansui* (a mixture of sodium and potassium carbonate) in the preparation of Chinese wheat noodles and waxy rice dumplings. The use of *kansui* is considered essential for the development of desirable product quality characteristics such as colour and texture. In Malaysia, sodium carbonate has been found to be the predominant salt used in the formulation of *kansui* (Lim, 1991). In some applications, *kansui* has been replaced by sodium hydroxide (NaOH). Besides being an important additive in the production of the aforementioned products, NaOH and other alkalis are generally employed as catalysts in certain chemical modification processes

of starch. NaOH is also frequently used as a deproteinising agent during starch extraction.

Despite the age-old usage of alkalising agents, there is still much which is not known concerning the effects of these reagents on starch properties. Although the ability of aqueous alkali in inducing starch swelling is common knowledge, most of the work has been centred on NaOH in systems containing relatively low starch concentrations ($<25\%$, w/w) (Leach, Schoch, & Chessman, 1961; Maher, 1983a, 1983b; Ragheb, El-Thalouth, & Tawfik, 1995; Wootton & Ho, 1989). Alkali-induced gelatinisation of starch, followed in these experiments mainly by rheological measurements and/or microscopic examination, was generally found to be dependent on the type of starch, type and concentration of alkali, treatment temperature, and duration of treatment. In the case of lime [$Ca(OH)_2$], which is used in the preparation of tortillas, Gomez, McDonough, Rooney, and Waniska (1989) observed decreased starch crystallinity and birefringence in corn cooked with lime. More recently, Bryant and Hamaker (1997) studied the effect

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of $Ca(OH)_2$ on gelatinisation of corn flour and starch, using a variety of techniques, including differential scanning calorimetry (DSC). Gelatinisation temperatures were found to increase with increasing lime concentration from 0 to 1% (w/v). This effect was attributed to ionisation of starch hydroxyl groups (at the high pH of the system) which promoted formation of Ca-starch crosslinks, thereby making the starch granules stronger and more rigid.

As far as the authors could ascertain, the influence of alkalising agents (especially *kansui*) on the thermal properties of starches has yet to be studied in any great detail. The main objectives of the present study were to observe the effects of sodium carbonate and sodium hydroxide on the thermal profiles associated with gelatinisation of selected cereal starches (wheat, corn, waxy corn, rice and waxy rice) at high and intermediate water contents, as determined by DSC.

2. Materials and methods

2.1. Materials

Wheat starch (WS), rice starch (RS) and corn starch (CS) were obtained from Sigma Chemical Company (St. Louis, MO, USA). Waxy corn starch (WCS; "Amioca") was kindly donated by National Starch and Chemical. Waxy rice starch (WRS) was isolated from commercial waxy rice flour ("Erawan" brand, Cho Heng Rice Vermicelli Factory Co. Ltd., Thailand) using the alkali steeping method described by Juliano (1984). Analytical grade anhydrous sodium carbonate (Na_2CO_3) and NaOH, obtained from R&M Chemicals (Essex, UK), were used directly, without further purification.

2.2. Determination of moisture content of starch samples

Moisture content was determined by drying triplicate 5-g samples to constant weight in an air oven at 105 °C. The pH of all starch slurries prepared was determined, in duplicate, using an Orion pH Meter (Model 410A) which was calibrated at pH 7 and 10.

2.3. Preparation of starch slurries

Starch slurries were prepared at two dry starch/water ratios, i.e. 1:4 or 2:3 by weight. Na_2CO_3 or NaOH was added at a concentration of 1 g/100 g dry starch, such a level being commonly encountered in the preparation of traditional starch-based products. All samples were thoroughly mixed by hand. Control, as well as alkali-containing samples, were prepared in duplicate. Two sub-samples were taken from each slurried sample for DSC scanning. In a separate experiment, 1:4 WRS/

water systems containing different levels of Na_2CO_3 were prepared and scanned.

2.4. Differential scanning calorimetry

Slurried samples of about 10 mg (weighed to 0.01 mg), with or without added alkalising reagent, were filled into preweighed aluminium sample pans, which were then hermetically sealed using a Du Pont encapsulation press and reweighed. All sealed samples were equilibrated at 25 °C for 1–1.5 h before being heated at 10 °C min^{-1} from 20 to 130 °C in a Du Pont 2910 differential scanning calorimeter, equipped with a standard DSC cell and a Thermal Analyst 2000 Controller, in order to obtain the gelatinisation thermal profiles. An empty aluminium reference pan was used to balance the heat capacity of the sample pan. Samples were then immediately quench-cooled to 20 °C and then rescanned.

Heat flow and temperature were calibrated using pure indium. Onset (T_o), peak (T_p) and conclusion (T_c) temperatures, as well as overall enthalpy (ΔH_G, expressed as J g^{-1} dry starch) associated with starch gelatinisation, were determined as described by Seow and Teo (1993) using a Du Pont DSC Standard Data Analysis Programme Software (Version 4.0B).

3. Results and discussion

3.1. Gelatinisation thermal profiles

Figs. 1–5 show the effects of Na_2CO_3 and NaOH on DSC thermal profiles on heating 1:4 and 2:3 WS/water, CS/water, WCS/water, RS/water, and WRS/water systems, respectively. In the absence of alkali, the 1:4 systems exhibited a single gelatinisation endotherm transition while the more concentrated systems gave rise, in some cases, to two conjoined endotherms over a broad range of temperature ($\sim$58–85 °C). Biphasic gelatinisation endotherms are commonly observed when starch/water systems at intermediate moisture contents are heated, although the structural origin of such phase transformations remains very much a subject of great interest and debate (Biliaderis, 1998; Waigh, Gidley, Komanshek, & Donald, 2000). Minor endothermic transitions (attributed to "melting" of amylose–lipid complexes) were also observed to occur at temperatures slightly above the gelatinisation endotherms in the case of the non-waxy starches.

The effects of Na_2CO_3 or NaOH on the DSC thermal profiles of the different types of starches studied were qualitatively similar, although there were some notable differences. The most obvious effects arising from the presence of the alkalising agents were: (1) a shift in transition temperatures of the gelatinisation endotherm to slightly but significantly ($P < 0.05$ or better) higher

temperatures, (2) a shift in baseline in the opposite (i.e. exothermic) direction to that normally observed upon completion of the gelatinisation endotherm (particularly in the presence of Na_2CO_3), which suggests a substantial difference in heat capacity between a gelatinised starch/water system and a gelatinised starch/ water/alkali system, and (3) the emergence of an intriguing exothermic thermal event, which was large enough to eclipse the small endothermic transitions generally ascribed to "melting" of amylose–lipid complexes observed in the thermograms of the non-waxy starches studied, when heating was extended beyond the
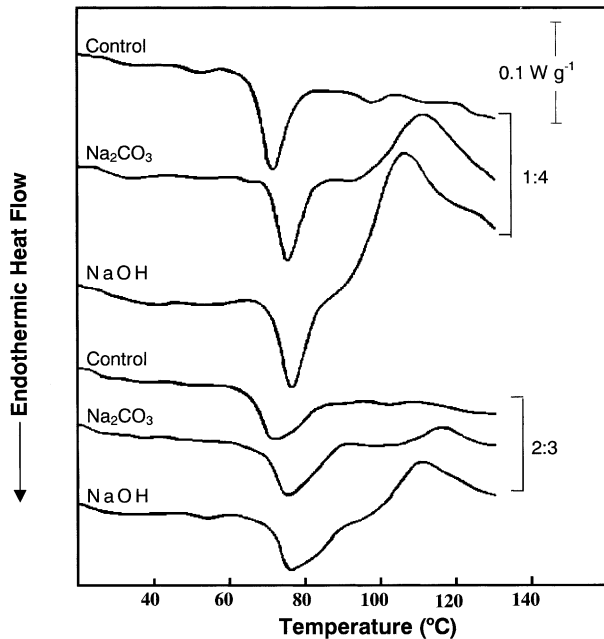

Fig. 1. DSC thermograms of 1:4 and 2:3 wheat starch/water systems with or without alkalising agents at a level of 1 g/100 g starch. (All thermal curves are normalised to 1 g of dry starch.)
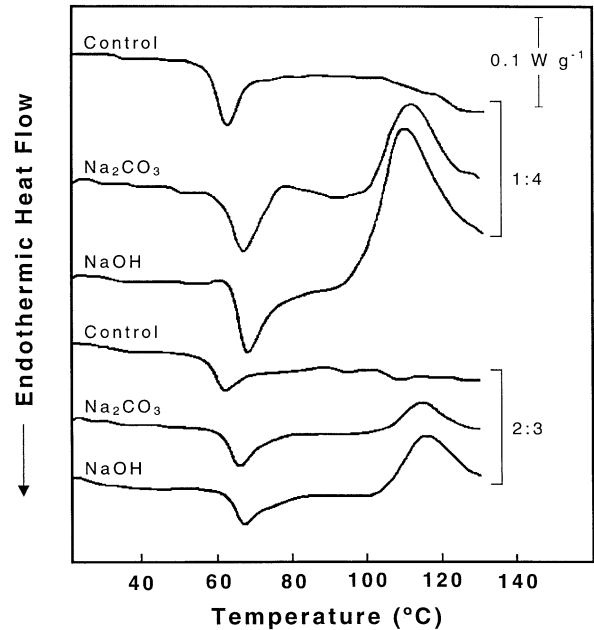

Fig. 3. DSC thermograms of 1:4 and 2:3 waxy corn starch/water systems with or without alkalising agents at a level of 1 g/100 g starch. (All thermal curves are normalised to 1 g of dry starch.)
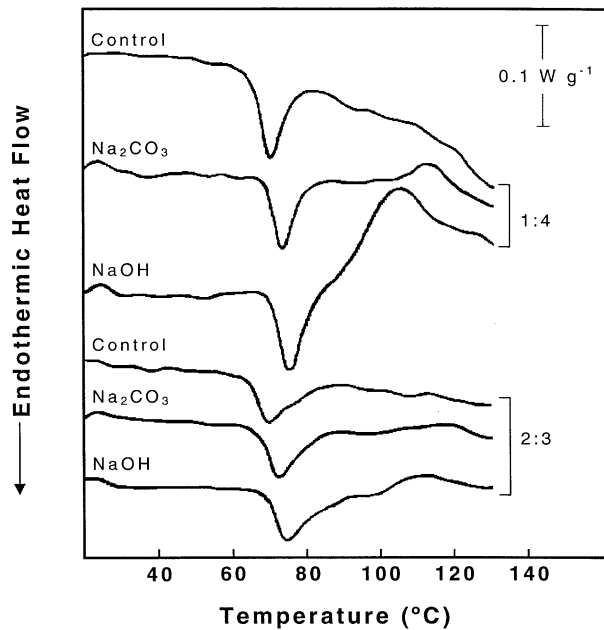

Fig. 2. DSC thermograms of 1:4 and 2:3 corn starch/water systems with or without alkalising agents at a level of 1 g/100 g starch. (All thermal curves are normalised to 1 g of dry starch.)
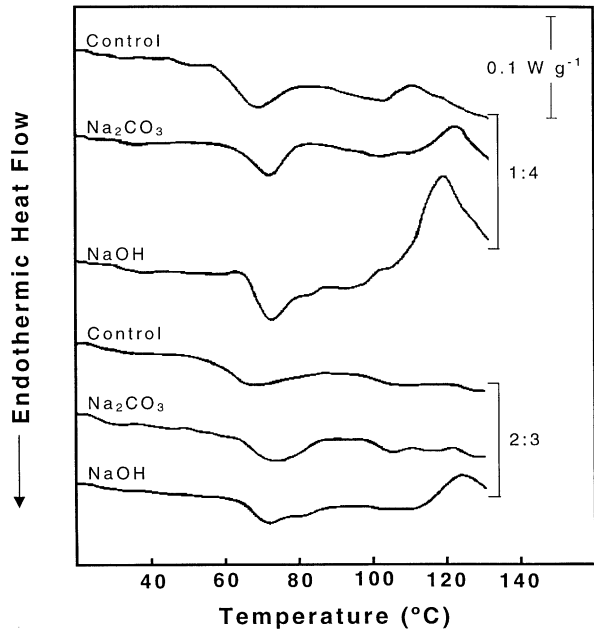

Fig. 4. DSC thermograms of 1:4 and 2:3 rice starch/water systems with or without alkalising agents at a level of 1 g/100 g starch. (All thermal curves are normalised to 1 g of dry starch.)

gelatinisation range. Both the gelatinisation endotherm and the exothermic transition were found to be thermally irreversible on immediate cooling and rescanning (DSC curves not shown).
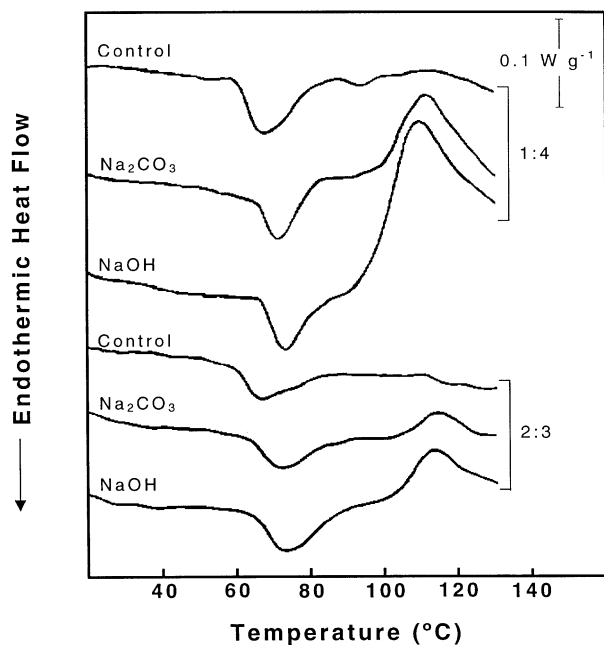

Fig. 5. DSC thermograms of 1:4 and 2:3 waxy rice starch/water systems with or without alkalising agents at a level of 1 g/100 g starch. (All thermal curves are normalised to 1 g of dry starch.)

The significant increases in gelatinisation transition temperatures in the presence of Na_2CO_3 or NaOH (Tables 1 and 2) suggest an enhancement of starch granule stability, probably through electrostatic interactions between Na^+ ions and hydroxyl groups of starch. Starch may be viewed as a weak acidic ion-exchanger which, in the presence of water, exhibits a Donnan-potential (Oosten, 1990). The negative electrical charges of the starch particles tend to attract cations and repel anions, thereby promoting penetration of sodium ions into the amorphous regions of the starch granules. Under high pH conditions, starch hydroxyl groups would have a greater propensity to ionise and create even more binding sites for cations. While anions may tend to destabilise the starch granules by rupturing hydrogen bonds, as hypothesised by Oosten (1990), such destabilising effects may be far weaker than the stabilising effects of cations. Several researchers have reported similar stabilising effects of NaCl (and other sodium salts in some cases), especially at lower concentrations (Abd Ghani, Che Man, Mi, & Mat Hashim, 1999; Evans & Haisman, 1982; Ghiasi, Hoseney, & Varriano-Marston, 1982; Jane, 1993; Lii & Lee, 1993; Wootton & Bamunuarachchi, 1980). Oosten (1979) had also noted the elevation of the gelatinisation temperature of starch in the presence of hydroxide. Bryant and Hamaker (1997) reported that DSC transition temperatures associated with gelatinisation of isolated corn starch increased with increasing lime [$Ca(OH)_2$] concentration up to 1% (w/v), although enthalpy values

Table 1
Effects of Na_2CO_3 and NaOH (added at a level of 1 g/100 g starch) on transition temperatures and enthalpy associated with gelatinisation of 1:4 starch/water systems

Starch	Treatment	Transition temperature (°C)[a]			ΔH_G (J g^{-1} starch)[a]
		T_o	T_p	T_c	
Wheat	Control	56.5a±0.2	61.6a±0.3	68.9a±0.2	14.7a±2.2
	Na_2CO_3	60.7b±0.4	66.7b±0.2	75.2b±0.9	14.6a±2.7
	NaOH	62.0c±0.4	67.4c±0.5	75.2b±1.0	14.7a±2.6
Corn	Control	64.6a±0.3	69.4a±0.2	76.3a±0.8	20.7a±3.9
	Na_2CO_3	68.2b±0.3	73.2c±0.3	79.8b±0.6	16.6b±1.8
	NaOH	68.8c±0.3	74.3c±0.2	81.4c±0.5	21.8a±1.5
Waxy corn	Control	65.6a±0.4	71.1a±0.2	78.7a±1.0	21.5a±1.1
	Na_2CO_3	70.3b±0.2	75.7b±0.1	82.4b±0.5	17.3b±2.4
	NaOH	70.7c±0.3	76.3c±0.3	83.5c±0.8	21.8a±2.6
Rice	Control	56.5a±1.0	66.5a±0.4	78.0a±1.3	12.4a±1.2
	Na_2CO_3	59.9b±1.0	69.8b–0.2	77.4a±1.0	10.9b±1.1
	NaOH	63.4c±0.9	71.4c±0.5	82.3b±1.7	13.5c±1.2
Waxy rice	Control	59.5a±0.1	66.7a±0.4	81.3a±1.5	19.0a±1.8
	Na_2CO_3	63.4b±0.8	70.8b±0.3	86.1b±1.2	17.2a±0.2
	NaOH	66.0c±0.3	72.8c±0.2	84.0ab±2.5	15.0b±1.2

[a] Values are means±S.D. ($n = 4$). Means within a column related to a particular starch with the same letter are not significantly different at the 5% level of probability. (Analysis of variance, followed by LSD, was carried out using Minitab 10 for Windows, Release 10.1, State College, PA.)

were not affected. In the present study, effects, if any, of Na_2CO_3 or NaOH on overall gelatinisation enthalpy (ΔH_G) were somewhat inconclusive, due in part to the lack of accuracy in determining ΔH_G (Table 1). In higher moisture (1:4) systems, the presence of Na_2CO_3 appeared to significantly ($P < 0.05$ or better) decrease ΔH_G of all of the starches studied (with the exception of wheat and waxy rice starches, which showed no significant change in ΔH_G), while NaOH had no effect on ΔH_G of most of the starches studied. However, in the less hydrated (2:3) systems, Na_2CO_3 did not significantly affect ΔH_G except, again, in the case of wheat starch where a slight but significant decrease in ΔH_G was detected, while the effect of NaOH on ΔH_G was inconsistent.

The magnitude and temperature location of the exothermic transition, characteristic of alkali-containing systems, appeared to vary with the type of starch, the alkalising agent added, and the water content of the system. This exotherm was considerably larger and occurred at lower temperatures in systems of higher moisture content and those with added NaOH. The origin of this exothermic transition remains unclear. Liu, Ramsden, and Corke (1997) and Liu, Corke, and Ramsden (2000) obtained DSC gelatinisation thermograms for some samples of maize starches that apparently also exhibited similar exotherms. They attributed the existence of such exothermic peaks to amylose–lipid complexation despite the fact that such transitions also occurred in waxy maize samples containing little amylose. Such a complexation reaction would also be unlikely to occur over the same range of temperature reported for the "melting" of amylose-lipid complexes. We offer what is probably a more plausible explanation. NaOH and other alkalis can depolymerise starch, especially on heating (Jackson, Choto-Owen, Waniska, & Rooney, 1988; Ragheb et al., 1995; Wootton & Ho, 1989). In fact, NaOH has been reported to be capable of hydrolysing starch into shorter chain molecules at a concentration as low as 0.001 M (0.01%) in the aqueous phase, with amylopectin being more affected than amylose (Jackson et al., 1988). Alkaline hydrolysis or rupture of glucosidic linkages may be followed by repolymerisation of oligosaccharides (or even glucose monomers) into larger molecules on heating to high temperatures, thereby giving rise to an exotherm in the DSC thermograms of alkali-containing samples. This may also explain the visual observation of colour development in such samples. Based on such a supposition, it is clear that, in any particular starch/water system and at the same alkali concentration of 1 g/100 g starch, the presence of NaOH induced greater repolymerisation than Na_2CO_3. This may be related to the higher pH of NaOH-containing systems (pH≈11) compared with Na_2CO_3-containing samples (pH≈10).

3.2. Effects of Na_2CO_3 concentration

The effects of increasing Na_2CO_3 concentration were studied in a separate experiment using 1:4 WRS/water

Table 2
Effects of Na_2CO_3 and NaOH (added at a level of 1 g/100 g starch) on transition temperatures and enthalpy associated with gelatinisation of 2:3 starch/water systems

Starch	Treatment	Transition temperature (°C)[a]			ΔH (J g^{-1} starch)[a]
		T_o	T_p	T_c	
Wheat	Control	56.3a±0.1	61.2a±0.3	83.8a±1.4	12.0a±0.8
	Na_2CO_3	59.9b±0.3	65.2b±0.4	81.1b±2.0	11.2b±0.6
	NaOH	61.6c±0.2	66.5c±0.3	84.1a±1.9	9.9c±0.8
Corn	Control	63.7a±0.3	69.1a±0.2	81.7a±2.1	15.7a±0.5
	Na_2CO_3	66.8b±0.1	72.1b±0.1	85.5b±1.9	15.3a±0.9
	NaOH	68.0c±0.2	73.6c±0.2	87.7b±1.9	17.3b±0.6
Waxy corn	Control	64.9a±0.2	71.0a±0.3	87.0a±1.3	19.8a±0.8
	Na_2CO_3	68.3b±0.2	74.7b±0.2	87.1a±0.9	19.1a±0.9
	NaOH	69.8c±0.3	75.8c±0.3	90.0b±1.1	19.1a±1.2
Rice	Control	57.4a±0.6	67.5a±0.4	83.6a±1.0	11.9a±0.9
	Na_2CO_3	60.0b±0.7	70.1b±0.5	85.0b±0.8	11.5a±0.4
	NaOH	62.9c±0.5	71.2c±0.4	88.3c±0.5	10.5b±0.6
Waxy rice	Control	57.4a±0.4	65.3a±0.7	82.4a±0.6	14.2a±0.8
	Na_2CO_3	62.5b±0.4	71.1b±0.7	86.0b±1.7	13.8a±0.6
	NaOH	64.3c±0.5	72.2c±0.4	85.4b±1.4	15.6b±1.3

[a] Values are means±S.D. ($n = 4$). Means within a column (with reference to a particular starch) with the same letter are not significantly different at the 5% level of probability.

systems. Fig. 6 shows the change in pH of WRS–water mixtures with increasing Na_2CO_3 concentration. Addition of Na_2CO_3 to a concentration of 0.25 g Na_2CO_3/100 g dry starch (equivalent to ~0.06 g Na_2CO_3/100 g water) increased the pH of the starch slurry sharply from ~6.3 to ~9.5. However, a further increase in Na_2CO_3 concentration to 3 g Na_2CO_3/100 starch (equivalent to 0.75 g Na_2CO_3/100 g water) increased the pH by only ~1 unit.
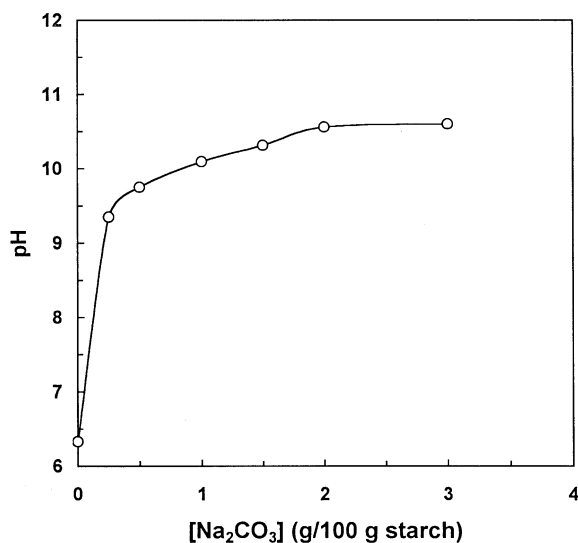

Fig. 6. Effect of Na_2CO_3 concentration on pH profile of 1:4 waxy rice starch/water mixtures.

Fig. 7 shows the effects of increasing Na_2CO_3 concentration on DSC thermal profiles on heating 1:4 WRS/water systems. The marked shift in baseline of the endothermic transition associated with starch gelatinisation, and the appearance of the exotherm attributed to repolymerisation of starch hydrolysis products in the presence of Na_2CO_3, were again noteworthy. Increasing Na_2CO_3 concentration gave rise to contrasting effects on the gelatinisation endotherm and the repolymerisation exotherm, in that the former was shifted to progressively higher temperatures and the latter to slightly lower temperatures.

The effects of increasing Na_2CO_3 concentration on transition temperatures are shown in greater detail in Fig. 8. Gelatinisation transition temperatures were enhanced relatively more sharply up to a concentration of 1 g Na_2CO_3/100 g starch before levelling off on further addition of Na_2CO_3. The gelatinisation range (i.e. T_c–T_o) did not appear to be substantially affected by differences in Na_2CO_3 concentration. Peak temperature (T_r) of the repolymerisation exotherm appeared to decrease marginally with increasing Na_2CO_3 concentration.

Concomitant with the increase in gelatinisation transition temperatures was a decline in ΔH_G (which was approximately rectilinear) as Na_2CO_3 concentration was increased (Fig. 9). Decreases in ΔH_G have been similarly observed when starches were gelatinised in excess water in the presence of NaCl (Abd Ghani et al., 1999; Chungchareon & Lund, 1987; Wootton &
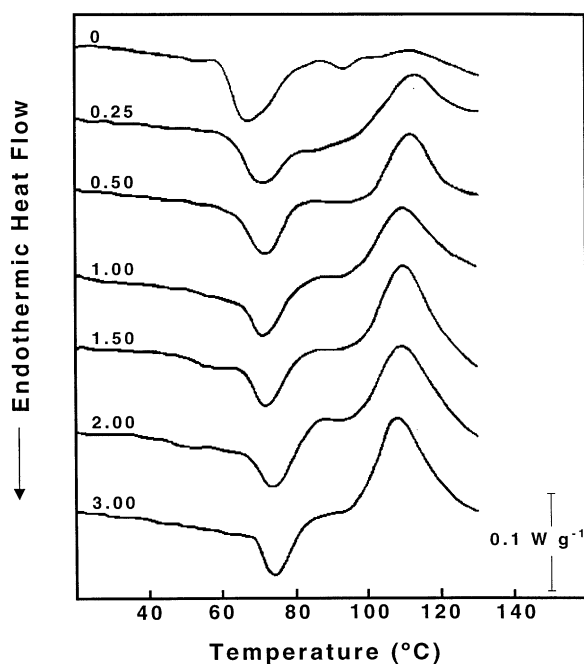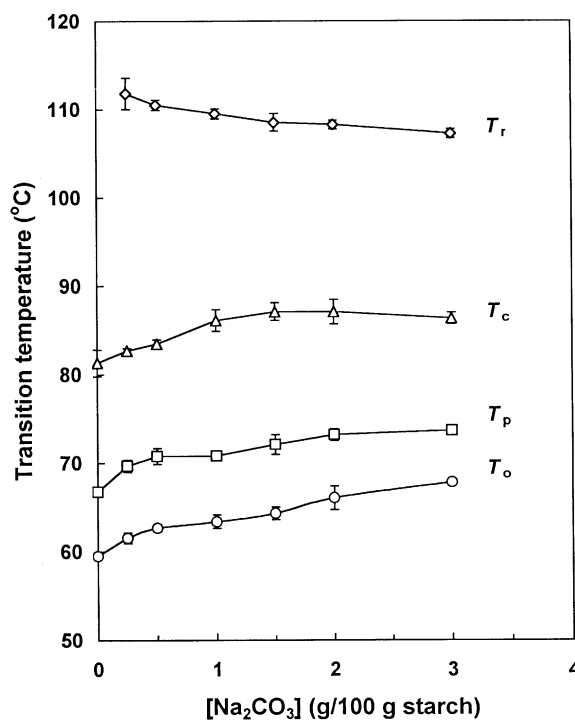

Fig. 7. Effect of Na_2CO_3 concentration on DSC thermal profiles of 1:4 waxy rice starch/water mixtures. (All thermal curves are normalised to 1 g of dry starch.)

Fig. 8. Effect of Na_2CO_3 concentration on gelatinisation transition temperatures and repolymerisation peak temperature in 1:4 waxy rice starch/water mixtures. (Error bar denotes ±1 S.D. from the mean.)
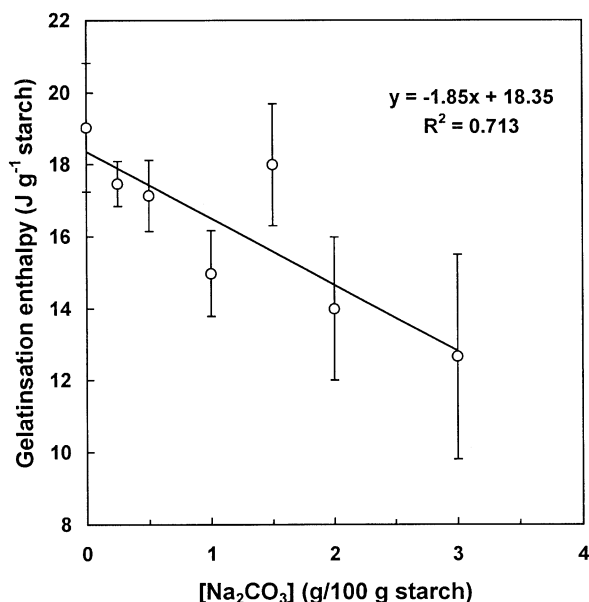
Fig. 9. Effect of Na_2CO_3 concentration on enthalpy associated with gelatinisation of 1:4 waxy rice starch/water mixtures. (Error bar denotes ±1 S.D. from the mean.).

Bamunuarachchi, 1980). Chinachoti, Steinberg, and Vilota (1990) assumed that such a decrease in ΔH_G reflected a decrease in the degree of starch gelatinisation. Thus, in the present system, if the gelatinisation enthalpy of the control sample was equated with 100% gelatinisation, then the degree of gelatinisation in the presence of 3% Na_2CO_3 was only ∼67%. While there appears to be some similarity between the effects of NaCl and Na_2CO_3, it is noteworthy that no exothermic transition has ever been reported for systems containing NaCl (a neutral salt).

4. Conclusion

The results of the present study show that Na_2CO_3 and NaOH, at a concentration normally applied in the preparation of traditional Asian foods, influence the gelatinisation behaviour of cereal starches through their ability to stabilise starch granules, probably via electrostatic interactions between Na^+ ions and starch hydroxyl groups. Heating of alkali-containing samples to temperatures slightly above the gelatinisation range appeared to lead to repolymerisation of lower molecular mass products derived from alkali-catalysed depolymerisation of starch. Thermal gelatinisation of starch in the presence of alkali, using different temperature regimes, is, therefore, likely to result in wide variations in properties of the gelatinised starch (such as colour, paste viscosity, gelation and gel properties, and retrogradation tendency). Since NaOH is a common reagent used in the extraction or modification of starches, significant variations in starch properties might also arise from the presence of different levels of residual alkali in the isolated or modified starches.

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