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Effect of sodium phosphate on the pasting, thermal, and rheological properties of potato and chickpea starches

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RESEARCH ARTICLE

Abstract

Differential scanning calorimetry (DSC) was used to determine the thermal properties of starch in controlled environment. Rapid visco analyser (RVA), Brookfield viscometer, and texture analyser were used to determine the effect of sodium phosphate (0.5, 1.0, and 1.5 M at pH 5, 7, and 9) on the cooking parameters, viscosity properties, and gel texture of potato (PS) and chickpea starch (CPS), respectively. Unlike chickpea starch at 0.5 and 1.0 M salt concentrations, the peak viscosity of potato starch at all salt concentrations decreased by about 50% as compared to control sample, especially at pH 5. CPS exhibited much higher setback values compared to PS. Gelatinisation temperatures of PS and CPS increased significantly ($P \leq 0.05$) as compared to control samples. Power law model confirmed pseudoplasticity of both starch gels (n<1). The DSC profile showed higher peak temperature at higher salt concentration, but lower enthalpy at higher salt concentration. Arrhenius equation showed the temperature dependency where the average activation energy (Ea = 18,629 K/J/mol) of CPS across salt concentration was higher compared to potato starch (6,094 K/J/mol). Gel hardness of the starches cooked in sodium phosphate generally increased with higher pH, except for CPS at 1.5 M sodium phosphate.

Keywords: chickpea, DSC, kinetics, pasting, potato, RVA, starch

1. Introduction

Commonly, salts co-exist with other ingredients in many food products. The effect of salts on the flowing properties of starches has been studied by other researchers. Reports showed that the effect of salts on starch retrogradation (Chang and Liu, 1991; Katsuta, 1998), granule swelling (Zhu *et al.*, 2009), and rheological properties (Ahmad and Williams, 1999; Katsuta, 1998) is significant. Unlike high NaCl concentration, at low NaCl concentration the gelatinisation temperature of starches with A-, B-, or C-types increases. However, the effect of salts on starch retrogradation varies due to the structural difference in X-ray diffraction patterns of starches (Lii and Lee, 1993). Differential scanning calorimetry (DSC) of rice starch showed that salts significantly increased starch gelatinisation temperatures, whereas the enthalpy (ΔH) was marginally affected. Gel structures formed after rice starch gelatinisation were significantly enhanced by salts as shown by dynamic rheology (Samutsri and Suphantharika, 2012). Waxy or common corn starches exhibited lower pasting viscosity and ΔH at higher sodium chloride (Baik *et al.*, 2010; Bello-Perze and Paredes-Lopez, 1995; Chungcharoen and Lund, 1987). The peak viscosity, trough, final viscosities, and pasting temperatures of Baizhi starches increased as NaCl concentration increased from 0 to 3.0% (Zhou *et al.*, 2011). In addition, the peak viscosity of the same starch had increased at 0.2% Na_2CO_3 concentration, but decreased at higher concentrations. In the presence of 0.1% NaOH, the peak viscosity of the starch increased, but decreased at 0.2%NaOH. Calcium chloride and sodium sulphite decreased the peak viscosity of cassava starch, whereas sodium sulphite significantly increased swelling and breakdown, unlike sodium chloride (Jyothi *et al.*, 2005). Reports indicated that sodium chloride increased the gelatinisation temperature of sago starch at lower salt concentration, but not at higher salt concentration (Maaurf *et al.*, 2001).

Salts can either raise or reduce the gelatinisation temperature as determined by DSC, according to their structure and concentration. Salts listed on the upper end of the Hofmeister lyotropic table, such as potassium citrate (cation) and sodium citrate (anion), Na_2SO_4, sodium and potassium acetates, increase the gelatinisation temperature and consequently are indicated as gelatinisation inhibitors (Villwock and BeMiller, 2005). Prevalent variation was observed in the pasting and swelling properties of cassava starch in the presence of cations, anions, acids (acetic acid) and oxidizing agents (sodium metabisulphite), where lower concentrations (1%) of acids and sodium metabisulphite enhanced the peak viscosity. However, sodium chloride was reported to reduce granule swelling which is unlike sodium metabisulphite (Jyothi *et al.*, 2005). Amongst other salts, NaCl was reported to increase peak temperature only at lower concentration (Ahmad and Williams, 1999; Chinachoti *et al.*, 1991; Jane 1993; Lii and Lee, 1993; Maaurf et *al*., 2001; Wootton and Bamunuarachchi, 1980). Acetic acid improved cassava gel clarity (Jyothi *et al.*, 2005). Zhu *et al.* (2009) indicated that monovalent salts used in the study had a similar protective effect at 0.1 M concentration. At higher concentrations, a markedly different effect was observed for chlorides, as compared to nitrites. Phosphate monoesters in potato starch are negatively charged groups. The repulsion due to these ionic groups prevents granule-granule association and increases the granule water-binding capacity and starch swelling power. So, the presence of salts can affect such repulsion and may cause physicochemical properties on potato starch (Kaur *et al.*, 2007; Singh *et al.*, 2004a). Zhou *et al.* (2011) reported that F^-, K^+, and SO_4^{2-} decrease starch swelling power, while Br^-, NO^{3-}, I^-, SCN^-, Na^+, and Li^+ enhance swelling power.

Because sodium phosphate is commonly used in the food industry and proven to interact with food ingredients in a way that changes the functional properties of some of these ingredients, it has been selected for this study. In addition, phosphates are commonly used in starch chemical modification to produce specialty starch products. In general, it is used in food product as a buffering agent, emulsifier, texturizer, and as nutrient. In starchy food, such as baked products, it reacts with bicarbonates to produce carbon dioxide in self-rising flour (Lampila, 2013). Phosphates are known to reduce cooking time of cereals and aid in the extrusion flow. The pasting properties of instant noodle powder were improved when 33% of modified potato starch was replaced by 0.030% monosodium phosphate or 0.300% disodium phosphate (Wang *et al.*, 2011). With respect to wheat flour, the addition of phosphates increased the gelatinisation temperature and ΔH of melting of starch in whole wheat flour. In addition, rapid visco analyser (RVA) analysis showed that phosphates significantly increased whole wheat flour peak viscosity and final viscosity (Niu *et al.*, 2014). Potato starch was selected for this project because potato processing typically starts by cleaning potato and pulping before directing to the final use such as snack foods. Therefore, phosphates can play the role of pH buffer or emulsifier. Since potato pulp is almost 100% starch, it is beneficial to report the effect of phosphate on potato starch. Chickpea meal as raw material for some products is widely used in many countries, but its application is limited. Starch is about 70% chickpea meal and due to its relatively high amylose content (29-32%) it is highly recommended for hot viscosity product application (Singh *et al.*, 2004b). Therefore, the performance of chickpea starch in sodium phosphate will add more information for possible new application. The choice of the pH range in this project was to cover probable pH range used in the food industry (pH 5 or 7). Testing starch properties at high molarity and pH (pH 9) is also valuable for starch modification because most chemical starch modification is done at high salt content and pH, such as crosslinking and acetylation.

In the present study, the effects of sodium phosphate on the thermal, pasting and rheological properties of potato starch (a tuber) and chickpea starch (legume) were studied with different concentrations and at different pHs.

2. Materials and methods

Materials

Potato starch was provided by Winlab Laboratory Chemicals (Market Harborough, UK) Chickpea (CP) (*Cicerarietinum* var. *surutato*) starch was isolated using whole grains purchased from a local market.

Methods

Isolation of chickpea starch

Chickpea grains were crushed using Brabender rotary mill (Brabender®, Duisburg, Germany) to obtain whole meal. The whole meal was suspended in distilled water (50/50; w/w) and mixed in heavy duty blender for 5 min. The slurry passed through 200 mesh sieve was centrifuged at 2,000 g for 15 min. After centrifugation, the dark layer on top of the pellet was removed and the white material at the bottom of the bottle was re-suspended in distilled water (50/50; w/w) and centrifuged using the same conditions mentioned above. This procedure was repeated five times. After washing with distilled water, pure white starch was mixed with acetone and air-dried. The dry starch was ground (very mild low speed blending for less than 1 min to avoid starch damage) in a coffee grinder, and stored in air-tight container at 4 °C for further use.

Pasting properties

Both starches were suspended in 0.5, 1 and 1.5 M sodium phosphate (NaH_2PO_4) solutions at pH of 5, 7 and 9. The pH was adjusted by using 1 N HCl or 5 N NaOH. The pH of the control sample was 6.1. The different suspensions were cooked using rapid visco analyser. The cooked samples were then used for testing by Brookfield viscometer and the texture analyser. The control sample was cooked in distilled water.

Starch pasting properties (peak viscosity, breakdown, setback and final viscosity) were determined using a Rapid Visco Analyser (Newport Scientific, Warriwood, Australia). The gel generated by RVA testing is used for rheological properties testing. Since, potato starch viscosity is naturally higher than most native starches, lower concentration was used so as to stay within the range of the rheometer used for this project. Chickpea starch (3 g on 14% moisture basis) or potato starch (1.5 g on 14% moisture basis) were directly weighed into aluminium canisters. The total amount was completed to 28 g with 0.5, 1.0 or 1.5 M NaH_2PO_4 solutions at pH 5, 7 or 9. The samples prepared with distilled water were used as control. The temperature profile included a temperature holding step (50 °C for 30 s), a linear temperature increase to 95 °C at 10.23 °C/min, a holding step (4 min at 95 °C), a linear temperature decrease to 50 °C at 22.5 °C/min, and a final isothermal step at 50 °C for 2 min. The mixing paddle speed was 960 rpm for the first 10 s and then 160 rpm for the remainder of the experiment. Pasting parameters, peak viscosity, breakdown, setback, and final viscosity, were calculated using Thermocline® for Windows software provided by the RVA manufacturer (Newport Scientific).

Thermal properties

The thermal properties of the starches were determined by DSC (MicroDSC III Evo; Setaram Instruments, Caluire, France). After the samples (240 mg) were weighed into Standard Hastelloy cell, 400 µl 0.5, 1 or 1.5 M sodium phosphate solutions at 5, 7 or 9 pH were added into the samples. The samples tested with distilled water were used as controls. There was the same amount of distilled water with the sample in the reference cell. After equilibration for 1 h, sample was heated from 20 to 110 °C at a heating rate of 2 °C/min. Gelatinisation parameters (onset, peak temperatures, and ΔH J/g) were calculated using Calisto Processing software for DSC (Setaram Instruments). Also, these parameters were calculated for the amylose lipid complex observed at higher temperature than starch gelatinisation peak.

Rheological measurements

Starch samples (1.5 g for potato starch (PS) and 3.0 g for chickpea starch (CPS) in a total of 28 g) were cooked in RVA using the same protocol as in the previous section. Dynamic viscoelastic and steady flow properties of PS and CPS gels prepared with distilled water (as a control) or salt solutions, obtained from RVA, were determined using a Brookfield viscometer (Brookfield DV-III; Brookfield Engineering Laboratories, Inc., Middleboro, MA, USA) equipped with a standardised LV3 spindle (diameter of 0.7 cm). The internal radius of the cylinder used for measurements was 1.15 cm. The shear rate constant (SRC) and spindle multiplier constant (SMC) were 0.33 and 128, respectively. Viscosity and shear stress measurements were done at 25 different rpm's starting from 2 to 26 rpm in increments of 2 rpm and from 26 to 2 rpm in decrements of 2 rpm. The shear rate was maintained between 0.66 s^{-1} (2 rpm) and 8.58 s^{-1} (26 rpm). Data was collected at 50, 30 and 20 °C for apparent viscosity (mPa.s) and shear stress (N/m^2) in triplicates.

The spindle constants (SMC and SRC) were determined as follows:

$$SMC = \frac{RI \times RPM}{TK \times 10{,}000} \quad (1)$$

Where SMC = spindle multiplier constant, which was used to calculate cP values; RI = full scale viscosity range of the rheometer (cP); TK = DV-III torque constant given by the manufacturer = 1; RI = 100 × n/Y; n = viscosity in cP of the Newtonian fluid; and Y = torque % reading at the selected RPM (100 rpm).

$$SRC = \frac{2W\,Rb^2RC^2}{X^2\,[RC^2 - Rb^2]} \quad (2)$$

Where SRC = shear rate constant (1/s), which was used for calculating shear rate and shear stress; Rc = radius of container (cm); Rb = radius of spindle (cm); X = radius at which the shear rate is to be calculated (normally the same as Rb in cm); and W = angular velocity of spindle (Rad/s):

$$W = \frac{2\pi}{60 \times N} \quad (3)$$

Where N = spindle speed in rpm.

Temperature dependency (Arrhenius Equation)

The consistency index (K) of the power law was used as a marker of the viscous character of the starch gel for the temperature dependency. It was calculated from the Arrhenius equation model fitted to the experimental data.

Gel texture

The gels of PS and CPS prepared with distilled water (as controls) or salt solutions, obtained from pasting in the RVA as described in section 'Rheological measurements' were used for determination of gel texture parameters. The gels (35 mm in height) were transferred into beakers 30 mm diameter and stored overnight at room temperature. Gel compression was done using Brookfield CT3 Texture Analyser (Brookfield Engineering Laboratories, Inc.) in two penetration cycles at a speed of 0.5 mm/s to a distance of 10 mm into the gel via a 12.7 mm wide and 35 mm high cylindrical probe. Gel hardness, springiness, cohesiveness and adhesiveness were determined. Textural parameters were automatically calculated by the instrument as specified by the manufacturer, except for gumminess which is considered the product of hardness and cohesiveness, and chewiness as the product of gumminess and springiness.

Statistical analysis

Measurements were done in triplicate. One way analysis of variance (ANOVA) technique was used to study the effect of NaH_2PO_4 of a specific molarity (0.5, 1 or 1.5 M) at different pH (5, 7 or 9) on potato and chickpea starch. Duncan's multiple range test at $P \leq 0.05$ was used to compare means using PASW® Statistics 18 software (SPSS Inc., Chicago, IL, USA).

3. Results and discussion

The peak viscosity (PV) of PS dropped by about 44-50% in sodium phosphate regardless of molarity compared to the control (Table 1). The PV of PS cooked in the same salt concentration increased with increasing pH. Overall, sodium phosphate reduced the peak viscosity of PS starch at all concentrations and pH values. The highest PV value (1,132 cP) for samples prepared in sodium phosphate was obtained for the 1.0 M at pH 9, followed by 1.5 M (1,089 cP) (Table 1 and Figure 1). Lower PV could indicate low swelling power of starch granules whereas the high PV of PS at higher pH shows higher swelling. The repulsion between the naturally present negatively charged phosphate groups in potato starch is reported to increase water binding and swelling of PS (Lim and Seib, 1993). The lower swelling of PS in presence of monovalent metals which leads to lower PV could be attributed to the disruption of repulsion between the negatively charged phosphate groups by the Na^+. This was apparent in the PV values of PS starch shown in Table 1. Conversely, reports in the literature indicated that sodium ions (Na^+) increase swelling power of cereal starches (Zhu *et al.*, 2009). The data in Table 1 showed that the inhibitory action of sodium phosphate was less at high pH, which could be caused by increased repulsion at higher pH. One can say that sodium phosphate effect on PS is pH dependent rather than molarity dependent, because the change in PV is more significant across pH compared to

Table 1. General effect of sodium phosphate (0.5, 1.0, 1.5 M) and pH (5, 7, 9) on the rapid visco analyser properties of potato (1.5 g) and chickpea (3.0 g) starches. Means ± standard deviations are given.[1,2]

	Potato starch				Chickpea starch			
	DW	pH=5	pH=7	pH=9	DW	pH=5	pH=7	pH=9
0.5 M[3]								
PV	2,034.5±20.5a	694.5±12.0d	788.5±24.7c	993.0±9.9b	2,828.00±91.9d	3,918.50±12.02c	4,555.50±3.5b	5,283.50±54.4a
FV	1,521.0±42.4a	833.0±32.5c	805.0±31.1c	1,256.0±7.0b	4,066.50±194.4c	5,684.00±36.77b	5,816.50±65.7b	7,273.00±31.1a
Setback	349.0±8.4b	243.5±13.4c	222.5±7.7c	433.5±24.7a	2,471.50±106.7d	3,017.50±67.18c	3,540.00±131.5b	4,045.00±36.7a
PT	67.9±0.3c	74.4±0.4a	74.02±0.0a	70.7±0.9b	69.20±0.4d	78.88±0.3a	77.58±0.4b	75.98±0.04c
1.0 M								
PV	2,034.5±20.5a	776.0±0.0d	924.5±7.7c	1,132.5±33.2b	2,828.0±91.9d	3,711.0±73.5c	4,339.0±93.3b	6,046.0±22.6a
FV	1,521.0±42.4a	894.5±10.6d	1,020.0±14.1c	1,340.5±31.8b	4,066.5±19.4d	5,542.0±10.8c	6,866.0±65.0b	8,115.0±10.9a
Setback	349.0±8.4b	157.0±4.2d	215.0±4.2c	486.0±21.2a	2,471.5±10.7c	2,614.0±10.0c	5,126.0±24.0a	4,777.5±67.1b
PT	67.9±0.3d	86.6±1.4a	81.1±1.0b	74.6±0.0c	69.2±0.4d	86.6±0.3a	84.4±0.8b	77.5±0.4c
1.5 M								
PV	2,034.5±20.5a	484.5±7.7d	698.5±40.3c	1,089.5±3.5b	2,828.0±91.9b	2,394.0±46.6c	1,267.5±105.3d	6,448.0±2.8a
FV	1,521.0±42.4a	424.0±4.2d	692.5±44.5c	1,414.0±0.0b	4,066.5±19.4b	2,904.5±13.0c	2,490.0±19.4c	8,713.5±1.5a
Setback	349.0±8.4	-	-	-	2,471.5±10.7b	916.5±91.2c	1,455.5±14.7bc	5,036.0±10.9a
PT	67.9±0.3c	80.8±0.5b	79.8±0.0b	84.4±0.9a	69.2±0.4d	93.5±0.1a	90.5±0.5b	77.0±0.2c

[1] Means within the same row with the same letter are not significantly different.

[2] DW = distilled water; PV = peak viscosity (cP); FV = final viscosity; PT = pasting temperature.

[3] M = potassium phosphate molarity.

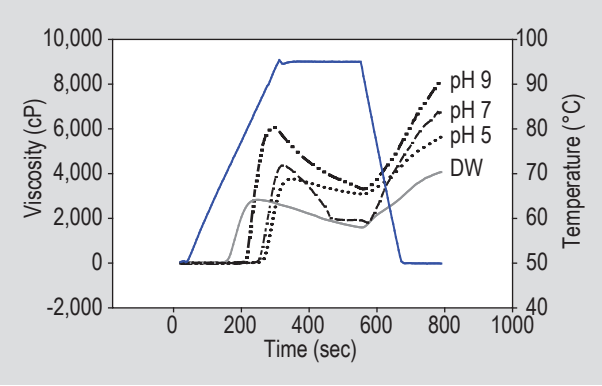

Figure 1. Rapid visco analyser profile of chickpea starch cooked in distilled water (DW) or 1 M NaH_2PO_4 solutions of pH = 5, 7 or 9.

change in molarity. Conversely, CPS exhibited PV values significantly ($P \leq 0.05$) higher than the control at all pH and salt concentrations except for 1.5 M at pH 9. The low PV at high molarity, except for pH 9, could be due to inhibitory action of the salt in the absence of repulsion noted for PS starch. Once again, high pH gave high PV for CPS. The difference between the granule structure of two starches (granule size, amylose and mineral contents) can be the cause of the different behaviour at the same experimental conditions, where PS granules are larger and CPS is higher in amylose content. Obviously, the presence of phosphate groups in PS is a major difference between PS and CPS. The dissimilarity in PV between the two starches was reflected in the final viscosity (FV), where 1.5 M pH 9 presented the highest FV value (8,713.5 cP) (Table 1). Since starch setback increases as a function of amylose content, the setback of CPS was much higher than PS due to the higher amylose content of CPS (Table 1). Although PS exhibited setback in distilled water, it showed no setback at 1.5 M sodium phosphate (Table 1) at all pH. Pasting temperature (PT) is one of the starch characteristics that determine the first step of the gelatinisation process. The PT of PS was lower than CPS. The higher PT at higher molarity could be attributed to the swelling inhibitory action of the salts at high salt concentration, which was also noted for the PV. The low PT of PS at high pH was observed, except for 1.5 M at pH 9, which is in line with PV pattern, where the inhibitory action of the salt at higher pH was decreased, causing starch to gelatinise earlier (at lower temperature). The reduction in PT for CPS as a function of higher pH could be attributed to high swelling power at high pH which resulted in lower PT. The highest PT (93 °C) was recorded at 1.5 salt concentration and pH 5 for CPS. The PT of both starches in sodium phosphate was higher than the control regardless of salt concentration or pH. The highest setback was obvious at pH 9 in 1.0 M for PS, whereas CPS exhibited the highest value at 1.5 M pH 9. PS starch did not exhibit setback at 1.5 M irrespective of pH.

The ΔH of PS as determined by DSC was significantly higher ($P \leq 0.05$) than the control at pH 5 regardless of molarity (Table 2). This is consistent with the pH dependency of PS pasting properties stated above. The CPS showed ΔH trend similar to PS except for 1.5 M and exhibited less pH dependency, where ΔH decreased significantly at pH 5

Table 2. General effect of sodium phosphate (0.5, 1.0, 1.5 M) and pH (5, 7, 9) on the differential scanning colorimeter properties of potato and chickpea starches. Means ± standard deviations are given.[1,2]

	Potato starch				Chickpea starch			
	DW	pH=5	pH=7	pH=9	DW	pH=5	pH=7	pH=9
0.5 M[3]								
ΔH	16.7±0.0c	18.1±0.2a	17.3±0.1b	17.5±0.2b	11.5±0.4c	14.8±0.3a	13.6±0.0b	13.4±0.1b
PT	63.3±0.0d	68.1±0.0a	67.6±0.3b	65.6±0.1c	62.8±0.1d	69±0.0a	67.4±0.0b	66.9±0.1c
OT	58.6±0.5c	62.7±0.1a	62.2±0.3a	60.1±0.1b	56.1±0.6b	59.3±0.0a	59.3±0.0a	58.5±0.0a
1 M								
ΔH	16.7±0.1b	18.0±0.2a	17.8±0.14a	16.9±0.1b	11.5±0.4c	13.8±0.1a	13.7±0.0a	13.0±0.0b
PT	63.3±0.1d	72.4±0.0a	71.7±0.02b	69.0±0.1c	62.8±0.1c	82.5±0.1a	80.6±0.2b	68.9±0.0c
OT	58.6±0.1d	66.9±0.1a	66.1±0.02b	63.2±0.2c	56.1±0.6c	63.2±0.1a	62.4±0.0a	60.5±0.6b
1.5 M								
ΔH	16.7±0.1ab	17.8±0.2a	17.6±0.3ab	16.3±0.3b	11.5±0.4a	10.1±0.1b	9.6±0.4b	11.1±0.1a
PT	63.3±0.1d	77.4±0.1a	77.0±0.1b	74.1±0.1c	62.8±0.1d	90.2±0.1a	89.3±0.5b	85.2±0.2c
OT	69.9±0.1c	71.5±0.1a	70.8±0.2b	67.9±0.1d	56.1±0.5d	77.3±0.3a	67.2±0.3b	65.5±0.4c

[1] Means in the same row with same letters are not significantly different.
[2] DW = distilled water; ΔH = enthalpy (J/g); PT = peak temperature (°C); OT = onset temperature (°C).
[3] NaH_2PO_4 molarity.

and pH 7. At the same pH value, the drop in ΔH is obvious and was more prominent for the CPS (Figure 2). Although it was higher than the control, the peak temperature was molarity dependent and decreases significantly at higher pH (Table 2). The high peak temperature at higher molarity is another demonstration of the protective action of salts. Nevertheless, the drop in peak temperature at higher pH could be attributed to the loss of granule integrity in alkaline environment which causes faster gelatinisation, thus lower peak temperature. Similar effect on peak temperature of CPS was noted, but the peak temperature of CPS was much higher than PS due to the higher amylose content which is found in the outer layer of the granules causing greater integrity and compactness. The highest peak temperature for PS and CPS controls were 63.4, 77.4 and 90.2 °C, respectively (Table 2). The onset temperature exhibited a trend comparable to the peak temperature.

Reports in the literature stated that anionic salts have more impact on starch granule swelling than cationic salts prompting direct effect on the thermal properties of the starch (Ahmad and Williams, 1999). Therefore, at 0.5 M NaH_2PO_4, starch was more stable compared to higher molarity indicating protective action of the sodium ions. As mentioned earlier in the introduction, NaCl, as well as other salts, was reported to increase peak temperature only at lower concentration (Ahmad and Williams, 1999; Chinachoti*et al.*, 1991; Jane 1993; Lii and Lee, 1993; Maaurf *et al.*, 2001; Wootton and Bamunuarachchi, 1980). The data presented here showed just that. For comparison, wheat and corn starches were reported to be more resistant to changes caused by salts, where the ΔH of these starches started decreasing at 1 M NaCl and higher, while a drop in the ΔH of rice starch was also reported in the literature at 0.3 M NaCl (Wootton and Bamunuarachchi, 1980). Structural differences between these starches, as verified by X-ray diffraction patterns, could explain the different effect of NaCl on these starches (Baik *et al.*, 2010; Bello-Perze and Paredes-Lopez, 1995). Conversely, other studies showed that the ΔH of corn starch as a function of NaCl or $CaCl_2$ started increasing at 0.1 M NaCl or $CaCl_2$ (Jane, 1993). The instability of starch granules in presence of anions could be attributed to the disruption of hydrogen bonding within the granule. Literature reports indicated the effectiveness of $CaCl_2$ is caused by the release of two Cl^- ions as opposed to one Cl^- from NaCl.

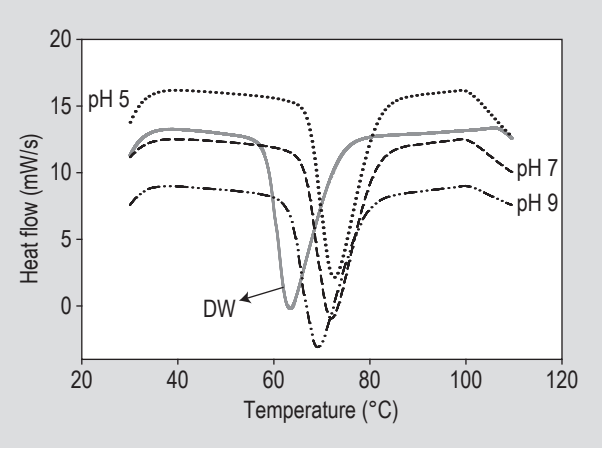

Figure 2. Differential scanning calorimetry thermograms of potato starch cooked in distilled water (DW) or 1 M NaH_2PO_4 solutions at pH = 5, 7 or 9.

The apparent viscosity of the RVA-cooked gels was determined using Brookfield viscometer at 50 °C. The RPM of the viscometer were converted to shear rate and the power law model ($T = K\dot{\gamma}^n$) was fitted to the experimental data. Hence, T is designated as shear stress (Pas), K as consistency coefficient (Pas), $\dot{\gamma}$ as shear rate (s^{-1}), and n as flow behaviour index (dimensionless). The natural log of the power law model allows calculation of k and n from the shear stress and shear rate plot. The n value is the slope of the line determined by linear regression of the shear rate against shear stress, whereas the intercept represents k (graphs not shown) (Table 3 and 4). The n values of both starches were less than 1 (Table 3 and 4). It means that starch gels were pseudoplastic material irrespective of salt molarity, pH, and the type of starch, as pointed out by previous researchers (Razavi *et al.*, 2007). Gels with n =1 are considered to follow Newtonian flow. The high R^2 shown in Table 3 and 4 indicates that the power law is appropriate for relating the flow behaviour of the gels within the range of viscosities of the starches cooked under the stated experimental conditions. It has been reported that pseudoplasticity of solutions, such as starch gels, is caused by disentanglement of long chain molecules which results in decrease in intermolecular resistance to flow under limited shear (Nurul *et al.*, 1999). The presence of sodium phosphate reduced the n value making PS more pseudoplastic. The flow behaviour index (n) of potato starch decreased at higher temperature under all salt molar concentrations, but at higher pH and same molarity, potato starch gels generally became less pseudoplastic (higher n). However, at higher salt molarity and same pH, potato starch gels turned into a more pseudoplastic material (lower n) compared to lower salt concentration, which can be attributed to limited disentanglement of starch fractions due water-mobility restriction by the salt. Conversely, CPS became less pseudoplastic at higher temperature and 0.5 or 1.5 salt concentration (Table 4) indicating direct influence of sodium phosphate concentration on CPS pseudoplasticity. This could mean increase in starch fractions disentanglement. The K value was greatly influenced by the high solutes content (higher molarity) in the liquid phase of the system, which could mean that a large portion of the phosphate was located in the liquid phase. Since the value of K is indicative of viscosity, it is temperature dependent and drops at higher temperatures. The K value of potato starch dropped at higher temperature

Table 3. General effect of sodium phosphate (0.5, 1.0, 1.5 M) and pH (5, 7, 9) on n and K values for potato starch.[1]

Temperature (°C)	DW			pH=5			pH=7			pH=9		
	n	K	R^2	n	K	R^2	n	K	R^2	n	K	R^2
0.5 M												
Ramping up												
30	0.666	2.874	0.998	0.440	2.688	0.999	0.211	1.102	0.988	0.454	3.256	0.994
40	0.581	2.922	0.998	0.428	2.621	0.996	0.191	1.131	0.999	0.444	3.099	0.997
50	0.552	2.905	0.993	0.414	2.490	0.996	0.183	1.077	0.997	0.430	2.970	0.994
Ramping down												
30	0.604	3.074	0.996	0.510	2.393	0.997	0.523	2.376	0.999	0.518	2.297	0.998
40	0.568	2.952	0.998	0.505	2.297	0.999	0.511	2.302	0.997	0.504	2.856	0.999
50	0.545	2.953	0.996	0.470	2.270	0.999	0.474	2.267	0.998	0.496	2.715	0.999
1 M												
Ramping up												
30	0.666	2.874	0.998	0.380	2.704	0.990	0.420	3.025	0.994	0.468	3.412	0.997
40	0.581	2.922	0.998	0.366	2.646	0.994	0.378	2.983	0.995	0.464	3.169	0.996
50	0.552	2.905	0.993	0.382	2.517	0.993	0.376	2.826	0.995	0.444	3.042	0.995
Ramping down												
30	0.604	3.074	0.996	0.396	2.664	0.999	0.466	2.847	0.998	0.520	3.200	0.999
40	0.568	2.952	0.998	0.394	2.552	0.999	0.432	2.775	0.999	0.509	2.996	0.999
50	0.545	2.953	0.996	0.391	2.508	0.999	0.410	2.710	0.999	0.478	2.919	0.999
1.5 M												
Ramping up												
30	0.666	2.874	0.998	0.118	1.804	0.850	0.195	2.437	0.994	0.451	3.514	0.985
40	0.581	2.922	0.998	0.065	2.170	0.836	0.180	2.476	0.996	0.532	2.572	0.994
50	0.552	2.905	0.993	0.032	2.630	0.695	0.157	2.687	0.996	0.528	2.353	0.994
Ramping down												
30	0.604	3.074	0.996	0.530	0.128	0.977	0.430	1.438	0.996	0.508	3.312	0.995
40	0.568	2.952	0.998	0.655	0.305	0.998	0.475	1.238	0.998	0.541	2.582	0.999
50	0.545	2.953	0.996	0.597	0.251	0.999	0.453	1.442	0.998	0.551	2.335	0.992

[1] DW = distilled water; n = flow behaviour index (dimensionless); K =consistency index (Pa).

and salt molarity. Overall, higher K was noted at higher pH. This was also obvious, as well, on the higher RVA peak viscosity of PS at high pH. The drop in K value at higher temperature indicates lower viscosity which is consistent with the general behaviour of biomaterials. The effect of salt solution concentration and higher temperature was more evident for CPS, where K value dropped at higher temperature within the same salt concentration. Conversely, across different salt concentrations the K values were as follows; at pH 5 1.0 M < 1.5 M < 0.5 M, whereas at pH 7 1.5 M < 1.0 M < 0.5 M. On the other hand, pH 9 exhibited higher K at higher sodium phosphate concentration (Table 4). Once again, chickpea starch showed higher K values compared to potato starch due to the higher amylose content. This observation was true for the RVA peak viscosity and DSC peak temperature mentioned above.

The temperature dependence of the viscosity of potato and chickpea starch at the specific pH and salt solution molarity was determined by fitting the data to Arrhenius model. The reciprocals of temperatures (30, 40, and 50 °C) were plotted against the log of K obtained from the power law model as shown in Table 3 and 4. The high R^2 (coefficient of determination) listed in Table 5 implies good correlation between the apparent viscosity and the specified temperatures and obeys Arrhenius model. The activation energy (Ea) of potato starch in sodium phosphate was much higher than in distilled water during ramping up and down. Potato starch exhibited Ea at 1.5 M as follows; pH 9 > pH 5 > pH 7 (Table 5) indicating the least coherent gel (weak network) at pH 7. The average ramping up Ea of potato starch for the same molarity, regardless of pH, showed increase at higher molarity, where ramping down exhibited mixed results (Ea of 1.5 M > 0.5 M > 1.0 M); a gel with high Ea is resilient gel. Conversely, CPS exhibited

Table 4. General effect of sodium phosphate (0.5, 1.0, 1.5 M) and pH (5, 7, 9) on n and K values for chickpea starch.[1]

Temperature (°C)	DW			pH=5			pH=7			pH=9		
	n	K	R^2	n	K	R^2	n	K	R^2	n	K	R^2
0.5 M												
Ramping up												
30	0.186	2.664	0.99	0.188	3.266	0.99	0.224	3.115	0.99	0.262	3.257	0.99
40	0.163	2.580	0.96	0.228	2.779	0.99	0.212	2.976	0.98	0.267	3.089	0.99
50	0.261	1.793	0.99	0.235	2.417	0.99	0.255	2.556	0.99	0.299	2.826	0.99
Ramping down												
30	0.281	2.451	0.95	0.282	2.864	0.99	0.348	2.591	0.99	0.361	2.850	0.99
40	0.292	2.190	0.99	0.322	2.356	0.99	0.358	2.376	0.99	0.372	2.656	0.99
50	0.430	1.423	0.99	0.368	1.854	0.99	0.386	2.014	0.99	0.381	2.493	0.99
1 M												
Ramping up												
30	0.186	2.664	0.99	0.282	2.714	0.93	0.305	2.728	0.97	0.269	3.453	0.99
40	0.163	2.580	0.96	0.192	2.458	0.97	0.186	2.762	0.98	0.262	3.236	0.99
50	0.261	1.793	0.99	0.120	2.196	0.92	0.197	2.471	0.99	0.292	2.943	0.99
Ramping down												
30	0.281	2.451	0.99	0.426	2.100	0.95	0.412	2.243	0.99	0.331	3.199	0.99
40	0.292	2.190	0.99	0.423	1.491	0.99	0.381	1.937	0.99	0.351	2.879	0.99
50	0.430	1.423	0.99	0.448	0.840	0.99	0.439	1.453	0.99	0.364	2.654	0.99
1.5 M												
Ramping up												
30	0.186	2.664	0.99	0.028	3.007	0.90	0.337	1.498	0.75	0.197	3.656	0.93
40	0.163	2.580	0.96	0.234	2.073	0.65	0.380	0.847	0.90	0.410	2.694	0.97
50	0.261	1.793	0.99	0.029	0.667	0.89	0.694	0.420	0.93	0.191	2.870	0.98
Ramping down												
30	0.281	2.451	0.95	0.071	2.591	0.77	0.442	1.129	0.91	0.356	2.980	0.99
40	0.292	2.190	0.99	0.129	2.588	0.35	0.595	0.031	0.88	0.538	1.920	0.99
50	0.430	1.423	0.99	0.469	0.958	0.91	0.318	0.061	0.90	0.389	2.038	0.99

[1] DW = distilled water; n = flow behaviour index (dimensionless); K =consistency index (Pa).

the lowest Ea at 1.0 M salt concentration followed by 0.5 M and 1.5 M. The behaviour of both starches in 1.0 M sodium phosphate appeared to be different compared to the other salt concentrations (Table 5), which requires further investigation. The controversy of the effect of salts on starch physicochemical properties is widely discussed in the literature. Depending on the starch type and concentration, salts can depress or increase gelatinisation parameters; this was reflected on the Ea values presented here. Once again, the data presented here showed that pH 9 exhibited the highest Ea for both starches at 1.5 M followed by pH 5. The different behaviour of starch at high pH is consistent with Maaurf *et al.* (2001), who reported higher peak viscosity of starch at 0.1 M NaOH. The difference between Ea values, K, and setback results for both starches investigated here at 1.0 M sodium phosphate is worth further investigation; in other words, why these starches behave differently at 1.0 M salt compared to 0.5 and 1.5 M. The apparent viscosity (μ_0) of potato starch at the specified temperature appeared not to follow any specific pattern, but it was lower at high molarity (Table 5).

Potato starch gel in distilled water exhibited the lowest hardness, however at all sodium phosphate concentrations, samples showed significant ($p \leq 0.05$) increase in hardness (Table 6). This was consistent with the increase in gel hardness of potato starch gel in sodium chloride, calcium chloride, and calcium lactate (Yifang *et al.*, 2014). The same authors reported higher storage modulus (G') and loss modulus (G''). Therefore, salts significantly affect the textural and rheological properties of starches. Gel hardness was higher at pH 9, except for PS in 0.5 M salt, which is consistent with the high peak viscosity at higher pH. Potato starch cooked in 1.5 M sodium phosphate pH 9 was the hardest of all (Table 6). This indicates stronger amylose network formation within the gel. This data was

Table 5. General effect of sodium phosphate on the Arrhenius equation parameters (activation energy Ea, the apparent viscosity μ_0, and the coefficient of determination R^2) of potato starch and chickpea starch.

	Ramping up			Ramping down		
	Ea (J/mol/K)[1]	μ_o (Pas^n)[2]	R^2	Ea (J/mol/K)	μ_o (Pas^n)	R^2
Potato starch						
Distilled water	675.8	0.2660	0.96	1,651.0	0.6305	0.76
0.5 M / pH=5	3,100.8	0.7886	0.96	2,158.8	0.9888	0.92
0.5 M / pH=7	1,991.1	0.5130	0.99	1,917.1	0.9030	0.97
0.5 M / pH=9	3,742.3	0.7366	0.99	6,916.7	0.0267	0.56
1.0 M / pH=5	2,902.5	0.8586	0.94	2,465.0	0.0752	0.95
1.0 M / pH=7	2,750.4	0.9780	0.88	2,006.7	0.7791	0.99
1.0 M / pH=9	4,682.8	0.5292	0.97	3,755.5	0.7164	0.95
1.5 M / pH=5	15,329.4	0.0012	0.99	27,850.5	0.0014	0.57
1.5 M / pH=7	3,943.5	0.0867	0.85	6,267.6	0.0655	0.78
1.5 M / pH=9	16,408.9	0.0050	0.92	14,281.1	0.0111	0.95
Chickpea starch						
Distilled water	15,960.1	0.0049	0.80	21,976.9	0.0004	0.89
0.5 M / pH=5	12,253.5	0.0251	0.99	17,668.2	0.3561	0.99
0.5 M / pH=7	7,998.3	0.1326	0.90	10,212.2	0.0456	0.96
0.5 M / pH=9	5,757.6	0.3335	0.97	5,446.4	0.3297	0.99
1.0 M / pH=5	8,608.0	0.0893	0.99	37,167.8	0.0001	0.97
1.0 M / pH=7	3,970.8	0.5757	0.64	17,598.5	0.0021	0.96
1.0 M / pH=9	6,487.0	0.3220	0.98	7,606.8	0.1557	0.99
1.5 M / pH=5	60,919.3	0.0001	0.91	40,037.5	0.0001	0.74
1.5 M / pH=7	51,663.8	0.0001	0.99	120,542.1	0.0010	0.60
1.5 M / pH=9	10,004.8	0.0650	0.60	15,670.0	0.0054	0.65

[1] Ea = activation energy parameters were obtained by fitting experimental data to Arrhenius equation ($\ln \mu_a = \ln \mu_o + Ea/RT$); the Ea data was based on three different heating rates 30, 40, and 50 °C.
[2] μ_o = is the apparent viscosity at a reference temperature.

not in agreement with the setback data shown in Table 1 which is also dependent on amylose network formation, but evidently PS at 1.5 M pH 9 showed no setback at all. As we make such a comparison, one should take into account that setback was determined in the RVA at 50 °C while gel hardness at room temperature. This is particularly important because in the RVA the energy in the system was still high causing high molecular mobility and prevented strong amylose network formation, therefore lower or no setback was recorded. In contrast, at room temperature, the lower energy of the system allows molecular proximity and facilitates amylose-network formation. The gel hardness of CPS starch was much higher than potato starch due to higher total solids (1.5 g for PS and 2.8 g for CPS) and higher amylose content of CPS. Unlike potato starch, the gel hardness of CPS starch dropped at higher molarity except for pH 9, which exhibited increase in hardness (Table 6). One can expect higher syneresis of chickpea starch due to the higher amylose content. The cohesiveness of potato starch gel was significantly lower than the control which is in agreement with the lower setback. The CPS starch cohesiveness increased at higher molarity for all three pHs, but at 0.5 M it was not significantly changed compared to the control. At pH 5, 1.0 M and 1.5 M it was significantly higher than the control, but for 1.0 M at pH 7 and 9 no change was observed. The sample with the highest cohesiveness was CPS at pH 7 1.5 M. In general, the mechanical property of any starch gel is reliant on factors such as amylose network characteristics, the volume fraction, and the flexibility of amylose and amylopectin chains. In addition, the interactions between the dispersed and continuous phases in the gel are critical as well (Biliaderis, 1998). Basically, gel hardness at 1.0 M and 1.5 M in Table 6 for potato starch increased at higher pH and molarity, where the highest chewiness, adhesiveness, and cohesiveness were listed at pH 9. However, CPS starch exhibited higher textural parameters at higher pH but not strictly.

Table 6. General effect of sodium phosphate on the texture profile analysis of potato and chickpea starches.[1]

	Potato starch				Chickpea starch			
	DW[2]	pH=5	pH=7	pH=9	DW[2]	pH=5	pH=7	pH=9
0.5 M								
Hardness (g)	11.0±1.4d	34.5±0.7a	22.00±1.4b	14.5±0.7c	33.0±1.4d	45.0±0.0c	54.0±2.8b	68.5±2.1a
Cohesiveness	0.8±0.0a	0.6±0.0c	0.64±0.0c	0.7±0.0b	0.4±0.0a	0.4±0.1a	0.4±0.1a	0.3±0.0a
Springiness (mm)	8.5±0.2b	10.0±0.0a	9.90±0.0a	9.9±0.2a	9.5±0.3ab	9.5±0.4b	9.9±0.1ab	10.2±0.1a
Adhesiveness (mJ)	0.2±0.1b	0.2±0.1b	0.15±0.1b	0.4±0.1a	0.2±0.1b	0.2±0.0b	0.2±0.0b	0.4±0.1a
Chewiness	69.9±11.9d	215.6±19a	139.39±8.9b	102.1±1.8c	123.8±2.1d	155.2±2.8c	191.7±18.9b	236.4±8.9a
1 M								
Hardness (g)	11.0±1.4d	18.0±1.4c	34.5±0.7b	52.0±5.6a	33.0±1.4c	49.0±1.4b	35.5±0.7c	69.00±4.2a
Cohesiveness	0.7±0.0a	0.5±0.1b	0.5±0.0c	0.5±0.1bc	0.4±0.1b	0.6±0.0a	0.4±0.1b	0.36±0.1b
Springiness (mm)	8.5±0.2c	9.1±0.5c	10.0±0.1a	9.9±0.1b	9.5±0.3a	9.1±0.5ab	8.1±0.6b	7.95±0.5b
Adhesiveness (mJ)	0.2±0.1c	0.2±0.1c	0.3±0.1ab	0.5±0.1a	0.2±0.1ab	0.1±0.0b	0.1±.0b	0.25±0.1a
Chewiness	69.8±11.9c	87.3±10.5c	175.9±1.1b	270.7±36.9a	123.8±2.0c	265.7±5.6a	122.1±8.1c	197.09±23.08b
1.5 M								
Hardness (g)	11.0±1.4d	19.0±1.4c	30.00±1.4b	108.0±1.4a	33.0±1.4b	14.0±1.4c	22.0±2.8c	121.5±6.4a
Cohesiveness	0.8±0.0a	0.3±0.0c	0.21±0.1c	0.5±0.0b	0.4±0.1c	0.7±0.0b	0.9±0.0a	0.5±0.1c
Springiness (mm)	8.5±0.3b	4.5±0.2c	5.05±0.2c	9.7±0.2a	9.5±0.3a	9.3±0.1a	9.6±0.1a	7.6±0.2b
Adhesiveness (mJ)	0.2±0.1bc	0.1±0.0c	0.35±0.1b	1.2±0.1a	0.2±0.1b	0.1±0.1b	0.1±0.1b	0.6±0.1a
Chewiness	69.9±11.9b	22.2±2.8c	31.80±6.5c	484.2±1.9a	123.8±2.1c	94.5±10.2c	180.8±27.3b	413.0±9.5a

[1] Means carrying same letters in a row are not significantly different; ± is standard deviation

[2] DW = distilled water.

4. Conclusions

The RVA pasting temperature and the DSC peak temperature decreased at higher pH. Potato starch exhibited more pseudoplastic behaviour (higher n value) than chickpea starch, whereas the shear stress and shear rate obeyed the power law. Both starches showed different interaction with sodium phosphate in terms of peak viscosity, setback, and gel texture. This data showed that Arrhenius equation is the appropriate method to point out the association between temperature and the viscous property of the two starches. Generally, CPS required higher activation energy than PS. The average activation energy of PS across pH increased at higher salt concentration with much higher value during ramping down, while CPS exhibited lower activation energy at higher pH. Gel hardness of both starches increased at higher pH, but CPS starch gels were much harder than potato starch due to higher amylose content. PS and CPS starches behaved differently in 1.0 M sodium phosphate compared to 0.5 M and 1.5 M, which requires further investigation.

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