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ORIGINAL ARTICLE

Physicochemical, morphological and pasting properties of acid treated starches from different botanical sources

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Abstract Effect of acid modification on physico-chemical, morphological and pasting properties of banana, sweet potato, lotus stem and wheat starches were studied. Results revealed that swelling power, solubility and water binding capacity of all starches decreased by acid modification. By acid modification starch granules from different varieties tended to appear fused and less smooth than the native starch granules. The pasting properties of native starches of all different varieties have significantly decreased following acid modification. Acid modified starches showed higher syneresis as compared to native starches.

Keywords Starch · Acid modification · Pasting properties · Morphological properties

Introduction

Starch is the major reserve polysaccharide in plants and is in the form of granules that exist naturally within the plant cells. Starch can be used as thickener, an adhesive, binder, encapsulating agent, film former, gelling agent, water binder, texturizer and fat-sparing agent and with numerous other applications both in the food and non-food areas (Mauro 1996). Starch granules are composed of a mixture of two polymers, an essentially linear polysaccharide called amylose and a highly branched polysaccharide called amylopectin (Bemiller and Whistler 1996). Starch can be modified by acid hydrolysis, oxidation, etherification, esterification and cross-linking. Various methods such as acid, phosphate and H_2O_2 treatments can be employed to modify starch (Akubor 2007). The objective of starch modification is to alter the physico-chemical characteristics of native starch to improve functional characteristics. Modification is important for the continued and increased use of starch to provide thickening, gelling, binding, adhesiveness and film forming characteristics.

Swelling power indicates the water holding capacity of starch, which has generally been used to demonstrate differences between various types of starches (Crosbie 1991). Swelling volume is the ratio of the sedimented gel to the dry weight of starch. Solubility is the percent amount of starch leached out into the supernatant in the swelling volume determination (Singh et al. 2005). The water binding capacity in commercial starches is important to the quality and texture of some food products because it stabilize them against effects such as syneresis, which sometimes occurs during retorting of freezing (Baker et al. 1994). Scanning electron microscopy has been used to relate granules morphology to starch genotype (Fannon et al. 1992). Rapid visco-analyser (RVA) has been extensively used for measuring starch paste viscosity. Pasting properties helps in comparison between cooking behaviour of different starches with the aid of RVA thermal viscous graph. The present study was undertaken to investigate the effect of acid modification on physicochemical, morphological and pasting properties of starches extracted from different plant sources.

Material and methods

Freshly harvested sweet potato (*Ipomoea botatas*), green un-ripened bananas (*Musa acuminata*) and lotus stem

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(*Nelumbo nucifera)* was procured from the local market, Amritsar, India. 'PBW-373' wheat (*Triticum aestivum*) variety was procured from PAU, Ludhiana, India.

Isolation of starch Banana fruits were peeled, cut into 6 mm slices, each slice was cut into four pieces and dipped immediately in sodium bisulphate solution (0.25 g/l) in 2:1 (v/w) proportion. Fruit pieces were macerated and blended (Sujata, New Delhi, India) at low speed 100 rpm for 5 min. The resultant slurry was sieved through 100 mesh until the waste solution was clean. Starch suspension was left overnight at refrigerated temperature (4 °C) and washed with tap water, centrifuged (Eltek, Mumbai, India) at 3000 rpm for 15 min. The white sediment was dried at 40 °C in a convection oven (Universal, New Delhi, India) for 24 h, ground in a pestle and mortar, passed through a 100 mesh screen and stored in sealed glass jars at room temperature (25–30 °C) (Waliszewski et al. 2003).

The lotus stems were washed, peeled, cut into small pieces and ground in a blender to form a paste. The resultant slurry was sieved through 100 mesh linen cloth into a beaker. Starch suspension was left overnight and extracted by washing with distilled water by four times. The resultant slurry was centrifuged at 3200 rpm for 10 min. The isolated starch was dried in an air oven at 40 °C for 24 h.

Sweet potatoes were washed thoroughly, immersed in ice cold water for 1 h, peeled, sliced and steeped in 0.1% (w/v) potassium metabisulphite to control enzymatic browning. It was then ground in a blender to form a paste. The recovery of the paste was done in 4% (w/v) NaCl solution. The solution was sieved and allowed to settle overnight. The starch was extracted by washing with distilled water four times. The resultant slurry was centrifuged at 3200 rpm for 10 min. The isolated starch was dried in an air oven at 40 °C for 24 h (Collado and Corke 1997).

Stiff dough was prepared by mixing 100 g of wheat flour with 45–55 ml of distilled water, left for 1 h at room temperature (30 °C), covered with moistened cheese cloth. It was washed under water gently to separate starch from gluten, over the mouth of beaker over a nylon mesh (240 μm). The water-starch suspension was allowed to settle for 5–7 h at 7 °C. The supernatant was decanted and starch slurry was recovered. The slurry was centrifuged at 3000 rpm for 10 min. The upper pigmented fraction was removed carefully by scraping with metal spatula. The sediment was re-suspended in distilled water and centrifuged again, scrapped-off in same way after 3–4 washings to obtain purified starch. The resultant starch sediments were dried at 40 °C in hot-air-oven overnight and clumps were broken manually to prevent formation of hard masses. The clumps were finally ground and passed through 72 mesh sieve (Wolf 1964).

Acid modification Dry starch (100 g) was taken in a beaker and 150 ml of 6% (w/v) HCl at 40 °C were added in batches over 48 h with occasional stirring. Acid addition was done carefully to accomplish a uniform dispersion of starch throughout the acid. After each hydrolysis, the suspension was neutralized with 2% (w/v) NaOH solution and washed three times with distilled water. The water was then removed by centrifugation at 3000 rpm for 10 min and decantation. The wet acid-modified starch was dried in hot-air-oven at temperature not exceeding 40 °C. The dried powder was ground and sieved through 100-mesh sieve to obtain acid-modified starch powder.

Physico-chemical properties

Swelling power and solubility A solution of starch slurry (1%) was made and heated in water-bath (Gupta Pvt Ltd, Ambala Cantt, India) maintained at 90 °C for 30 min with constant stirring and cooled. The suspension was centrifuged at 3200 rpm for 10 min and the supernatant collected in a pre-weighed aluminium dish, which was evaporated at 110 °C for 24 h. The dried aluminium dishes were weighed for calculation of solubility. The weight of wet sediment in centrifuge tube was noted to determine the swelling power (Leach et al. 1959).

Water binding capacity A suspension of 2.5 g native and treated sample in 20 ml of distilled water was agitated for 30 min in a shaker (Narang Scientific Works, New Delhi, India). The suspension was poured into pre-weighed centrifuge tube. Then 10 ml of distilled water used for rinsing starch from beaker, which was also added to centrifuge tube and centrifuged at 3000 rpm for 10 min. Supernatant was decanted and wet starch was weighed to determine water binding capacity (Anderson et al. 1969).

Scanning electron microscopy (SEM) Scanning electron micrographs were obtained at 800x with a SEM (Joel JSM-6100, Jeol Ltd., Tokyo, Japan). Starch samples were suspended in ethanol to obtain a 1% suspension. One drop of the starch-ethanol solution was applied on an aluminium stub using double-sided tape and the starch was coated with gold-palladium (60:40). An accelerating potential of 10 KV was used during micrography.

Pasting properties of native and acid modified starches were studied using RVA (RVA-4 D, Warriewood, Australia). Three g sample along with 25 g distilled water were weighed into aluminium canister. Paddle was placed into canister and vigorously jocked 10 times up and down to break any lumps on water surface. The sample was equilibrated at 50 °C for

1 min, heated to 95 °C in 4.42 min, held at 95 °C for 3.1 min before cooling to 50 °C and holding at 50 °C for 2 min. The mixture was stirred at 960 rpm for 10 s and then at 160 rpm for remainder of the test.

Starch suspension (2%, w/w) was heated at 85 °C for 30 min in a temperature controlled water bath (Gupta Pvt Ltd, India), followed by rapid cooling in an ice water bath to the room temperature. The starch sample was stored for 1, 2, 3, 4 and 5 days at 4 °C. Syneresis was measured (triplicates) as amount (%) of water released after centrifugation at 3000 rpm for 15 min.

Statistical analysis The mean and standard deviation of morphological and pasting properties parameters were calculated. One way ANOVA and least significant difference were employed to check the significant effect ($p<0.05$) of acid modification on the morphological and pasting properties of starches extracted from different botanical sources (Cochran and Cox 1957).

Results and discussion

Swelling power and solubility index The swelling power of acid modified starches was lower in all cases as compared to native starches (Table 1). Kaur et al. (2007) studied the effect of aqueous HCl on properties of wheat starch and found that swelling power decreased from 15.4 to 5.0 g/g with increase in modification time. The reduction in swelling power of acid modified starches was reported due to increase in high proportion of soluble dextrins of both small and medium chain lengths in starch granules (John et al. 2002).

Solubility index of native starches was lower compared to acid modified starches (Table 1). Fannon et al. (1992) reported that channels present in starch granules may also be responsible for aiding permeation and increase the potential surface area available for reaction and penetration of reagents in granules. During acid modification hydroxonium ion (H_3O^+) attacks glycosidic O_2 atom and hydrolyse glycosidic linkage, therefore acid preferentially attacks at amorphous regions. This causes increase in solubility index of acid modified starches.

Water binding capacity The acid modification reduced water binding capacity of starch (Table 1). Acid thinning impaired both hydrophilic and hydrophobic capacities of native starch. The acid thinning reduces water absorption capacity because of increase in crystalline region and decrease in amorphous region in starch granules that reduced the number of available binding sites thus lowering the water binding capacity (Lawal 2004).

Pasting properties The pasting temperature of acidified starches was higher than native starches (Table 2). The acid hydrolysis removed amorphous regions of starch granules leading to lowering of viscosity. The final viscosity of acid thinned modified starches was more than peak and hot paste viscosity. Acid modified starches show higher final viscosity supporting the fact that amylose terminated the short term gel structure development (Wang and Wang 2001). Kaur et al. (2007) studied on the effect of aqueous HCl on properties of wheat starch and found that the pasting temperature, peak viscosity, hot paste viscosity and cold paste viscosity showed a decline with acid modification.

Morphological properties The starches have been reported to differ in granules size, shape, presence of phosphate esters, amylose to amylopectin ratio that in turn is responsible for rheological, thermal and other functional properties (Wisenborn et al. 1994). The granules size and geometry differed significantly among various starches. The granules from different starches showed spherical, oval, cylindrical and oblong shapes but oval, cylindrical and irregular shapes were predominant. The native banana starch granules showed size in the range of 10–27 μm with higher proportion of medium size granules (Fig. 1). After

Table 1 Swelling power, solubility and water binding capacity of native and acid modified starches

Starch from	Swelling power, g/g		Solubility,%		Water Binding capacity,%	
	Native	Acid modified	Native	Acid modified	Native	Acid modified
Banana	$16.0^{c}\pm0.41$	$0.48^{c}\pm0.03$	$8.1^{b}\pm0.35$	$51.1^{d}\pm0.97$	$107.7^{a}\pm3.04$	$71.7^{a}\pm1.01$
Lotus Stem	$31.0^{a}\pm1.26$	$0.80^{b}\pm0.7$	$2.4^{c}\pm0.42$	$62.4^{b}\pm1.13$	$100.5^{b}\pm3.03$	$61.6^{b}\pm1.17$
Sweet Potato	$21.5^{b}\pm0.15$	$0.72^{b}\pm0.14$	$7.8^{b}\pm0.76$	$67.7^{a}\pm0.83$	$78.1^{c}\pm2.66$	$57.0^{c}\pm0.70$
Wheat	$9.8^{d}\pm0.60$	$1.05^{a}\pm0.07$	$12.9^{a}\pm1.03$	$55.7^{c}\pm1.06$	$66.8^{d}\pm2.05$	$46.4^{d}\pm1.20$

* Means with different superscripts are significantly different ($p\leq0.05$) along the rows ($n=3$)

Table 2 Pasting properties of native and acid modified starches

Starch	PT, °C	PV, cp	HPV, cp Native	CPV, cp	BD, cp	SB, cp
Banana	$75.1^{a}\pm0.29$	$4577^{b}\pm2.9$	$3532.7^{d}\pm18.5$	$4364^{d}\pm59$	$1045^{b}\pm33.9$	$832^{b}\pm41.1$
Lotus stem	$74.9^{a}\pm0.89$	$8314^{d}\pm330$	$2673^{b}\pm35.1$	$3471^{b}\pm60.8$	$564^{a}\pm35.9$	$798^{b}\pm90.5$
Sweet potato	$74.2^{a}\pm0.02$	$5964.3^{c}\pm43.5$	$2960^{c}\pm23.2$	$3587^{c}\pm93.1$	$3005^{c}\pm23.7$	$627^{a}\pm27.8$
Wheat	$81.6^{b}\pm4.33$	$2810.0^{a}\pm7.0$	$2243^{a}\pm48.3$	$3370^{a}\pm32.5$	$567^{a}\pm51.8$	$1127^{c}\pm27.9$
Acid modified						
Banana	$78.2^{a}\pm0.01$	$285.7^{a}\pm6.66$	$210.7^{a}\pm7.02$	$316.3^{a}\pm4.04$	$75^{a}\pm1$	$105.7^{a}\pm3.21$
Lotus stem	$77.4^{a}\pm0.77$	$516.0^{d}\pm67.6$	$318.0^{d}\pm47.8$	$624.0^{c}\pm2.04$	$198^{c}\pm20.2$	$306^{c}\pm156.8$
Sweet potato	$76.4^{a}\pm0.46$	$466.7^{c}\pm33.2$	$241.3^{c}\pm14.15$	$453.0^{b}\pm11.53$	$225.3^{d}\pm24$	$205^{b}\pm9$
Wheat	$94.9^{b}\pm0.5$	$329.7^{b}\pm44.6$	$235.0^{b}\pm48.6$	$635.7^{c}\pm48$	$94.7^{b}\pm4.04$	$400.7^{d}\pm4.16$

PT Pasting temperature; *PV* Peak viscosity; *HPV* Hot paste viscosity (Minimum viscosity at 95 °C); *CPV* Cold paste viscosity (Final viscosity at 50 °C); *BD* Breakdown viscosity; *SB* Setback viscosity; *cP* Centipoise

* Means with different superscripts are significantly different ($p \leq 0.05$) along the rows ($n=3$)

acid modification the granule size increased and reported a range of 15–34 μm. The native lotus starch granule showed uniform size, oblong structure having average granule size in the range of 14–31 μm which increased to 25–37 μm after acid modification. The native sweet potato show granule size range of 8–20 μm which after acid modification was reported to vary from 8 to 18 μm (Fig. 2). There was slight reduction in size in case of sweet potato granules after acid modification. The native wheat starch granules had sizes in the range of 9–28 μm with fairly uniform proportion of large, medium and small granules. After acid modification, the modified wheat granules had sizes in the range of 12–21 μm. Wheat granules seem to be affected more by acid hydrolysis compared to other acid modified granules.

Syneresis The syneresis of the native and acid modified banana, lotus stem, sweet potato and wheat after 1, 2, 3, 4 and 5 days (Table 3) was analyzed. The acid modified sample showed higher syneresis compared to native starches. The initial gel firmness during gelation is attributed to the formation of amylose matrix gel and slow increase in gel firmness due to amylopectin crystallization.

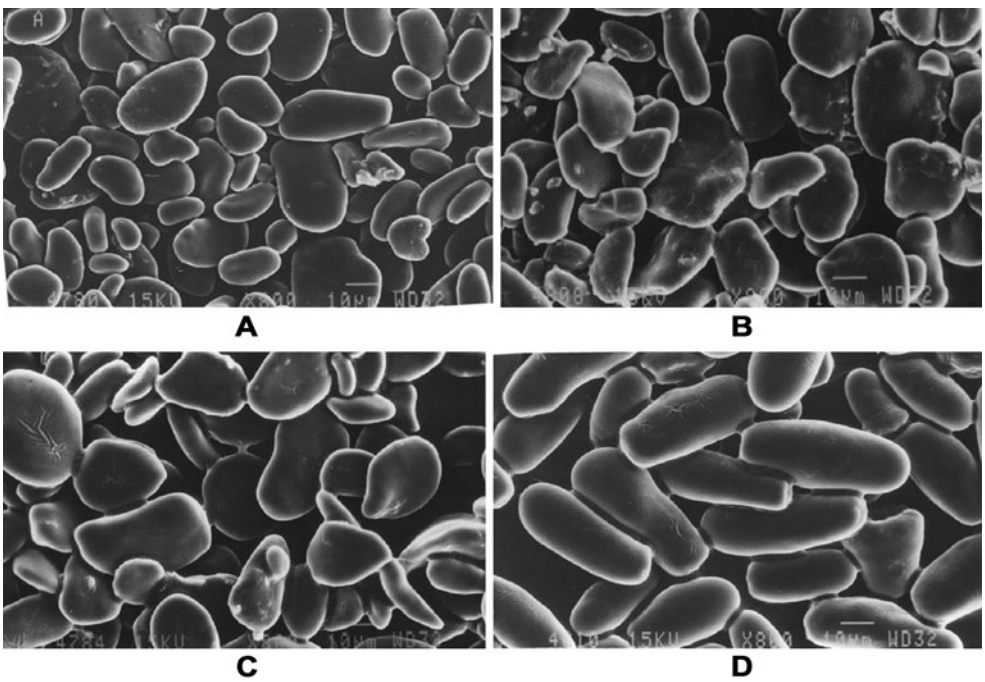

Fig. 1 Scanning electron microscopy photographs of banana starch. (A-Native, B-Acid modified) and Lotus stem starch (C-Native, D-Acid modified)

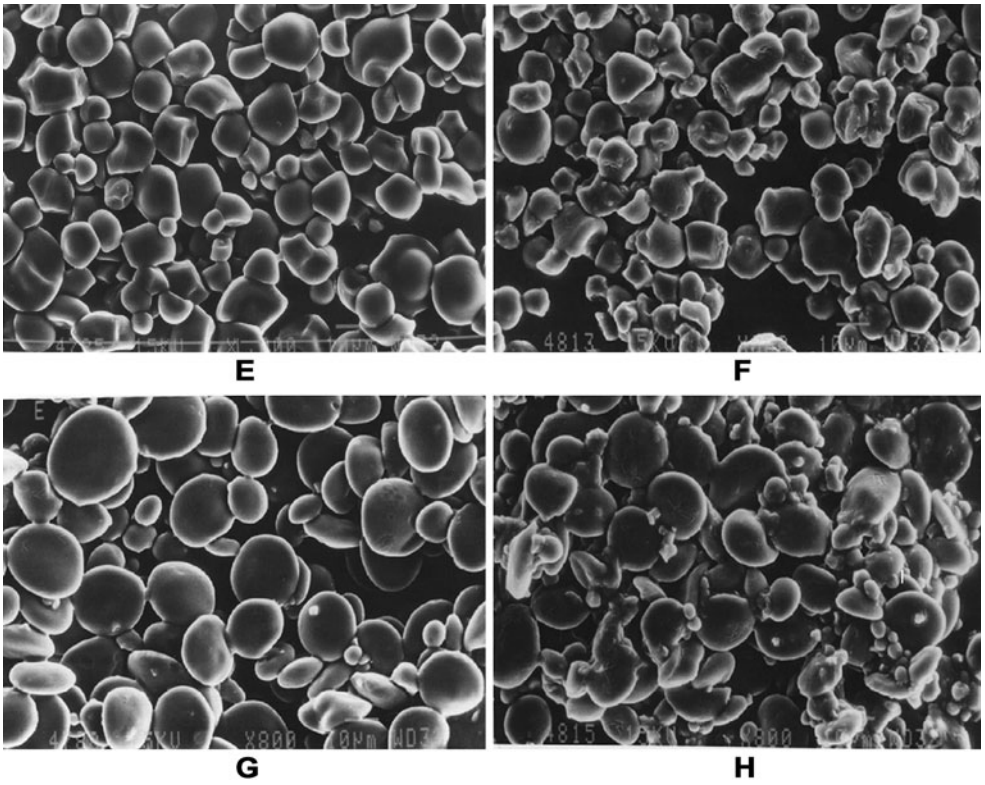

Fig. 2 Scanning electron microscopy photographs of sweet potato starch. (E-Native, F-Acid modified) and wheat starch (G-Native, H-Acid modified)

The amount of water excluded from the starch gel system stored at low temperature is due to increase in intermolecular and intra molecular hydrogen bonding due to interaction between amylose- amylose, amylose- amylopectin and amylopectin- amylopectin chains.

Conclusion

Swelling power, solubility and water binding capacity of starches decreased following acid modification. The morphological properties revealed hydrolysis of starch granules due to attack of acid on amorphous regions, which come in contact with the acid leading to fusion of granules. After modification, starch granules tended to appear fused and less smooth than the native starch granules. The acid modified starches reported slightly higher pasting temperature compared to their native counterparts. The acid modification reduced thickening ability of starches that is based on swollen capacity of undamaged granules as revealed by pasting properties. The syneresis increased with acid modification of starches.

Table 3 Effect of acid modification on the syneresis (%) of native and modified starches during storage

Starch from	Storage (4 °C) period, days				
	1	2	3	4	5
	Native				
Banana	$15.7^{ap}\pm0.71$	$18.8^{aq}\pm0.37$	$20.2^{ar}\pm0.34$	$21.2^{as}\pm0.78$	$21.3^{bs}\pm0.35$
Lotus stem	$11.4^{bp}\pm0.39$	$13.8^{bq}\pm0.28$	$15.7^{br}\pm0.28$	$21.9^{as}\pm0.50$	$23.8^{as}\pm0.22$
Sweet potato	$9.8^{cp}\pm0.40$	$13.8^{bq}\pm0.24$	$15.8^{br}\pm0.32$	$18.7^{bs}\pm0.65$	$18.8^{cs}\pm0.33$
Wheat	$7.2^{dp}\pm0.25$	$8.4^{cq}\pm0.23$	$10.8^{cr}\pm0.37$	$12.7^{cs}\pm0.42$	$13.9^{dt}\pm0.21$
	Acid modified				
Banana	$29.7^{ap}\pm0.40$	$31.9^{ap}\pm0.50$	$32.8^{ap}\pm0.30$	$35.8^{aq}\pm0.34$	$41.6^{br}\pm0.60$
Lotus stem	$28.5^{bp}\pm0.24$	$30.0^{bp}\pm0.29$	$32.8^{aq}\pm0.28$	$35.8^{ar}\pm0.39$	$43.6^{as}\pm0.20$
Sweet potato	$18.6^{dp}\pm0.32$	$19.6^{dp}\pm0.21$	$23.7^{cq}\pm0.15$	$26.9^{cr}\pm0.32$	$32.3^{ds}\pm0.34$
Wheat	$22.8^{cp}\pm0.20$	$23.7^{cp}\pm0.30$	$25.8^{bq}\pm0.37$	$28.7^{br}\pm0.18$	$33.8^{cs}\pm0.85$

* Means with different superscripts are significantly different ($p\leq0.05$) along the rows (a,b,c...) and columns (p, q, r...) ($n=3$)

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