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EFFECT OF CHEMICAL MODIFICATIONS ON THE FUNCTIONAL AND RHEOLOGICAL PROPERTIES OF POTATO (*SOLANUM TUBEROSUM*) STARCHES

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ABSTRACT

The present study investigated the effect of single and dual chemical modifications on functional and rheological properties of starch isolated from Pakistani potato cultivar. The isolated starch was chemically modified to produce acid-thinned, acetylated, oxidized and acetylated-oxidized starch. The objective of chemical modification was to tailor and improve the native properties of potato starch so that it could be used in wide variety of food products. The chemicals used for acid-thinning, acetylation and oxidation were hydrochloric acid, acetic anhydride and sodium hypochlorite, respectively. The scanning electron micrographs revealed the presence of small, medium and large sized granules ranging from 7-111 µm. Except acid-thinning, all other chemical modifications increased swelling power of potato starch. Each of the modifications reduced time to reach viscosity while pasting temperature of acetylated and dual modified acetylated-oxidized potato starch was also significantly reduced. Acid-thinning reduced peak viscosity of potato starch suggesting its use as a bulking agent in confectionery products. Shear stability also declined due to acetyl-oxidation of potato starch, owing to substitution of hydroxyl groups by acetyl, carboxyl and carbonyl groups. Oxidized and acid-thinned starch formed gels with higher firmness and rupture strength compared to native starch. However, increase in rupture strength with cold storage of starch gels declined significantly due to chemical modifications.

Key words: potato starch, modification, functional, rheological

INTRODUCTION

Different genotypes of potato contain 9 to 20% starch along with 75 to 80% water, 2.5 to 3.2% protein, 0.1 to 0.2% fats and 0.6% fiber (Abbas *et al*., 2011). Potato starch has its own usage because of its particular characteristics which are significantly different from starches of other sources. Starch from a particular genotype is required for desired functionality and unique physicochemical properties (Singh and Singh, 2001). Potato starch granules are oval or round with a very smooth exterior (Thomas and Atwell, 1999). Research papers on nutritional quality of potato genotypes in Pakistan showed that upto 20% starch is present in local Pakistani potatoes. As per the report MINFAL (1993) the farm price of Pakistani potatoes which would be the best for processing was 60% to 65% of the farm price of similar varieties in Eastern Europe and Turkey, and 40% to 50% of US or Western European farm prices.

Local food processing and manufacturing companies are importing modified potatoes starches from Europe and other countries to improve the quality of their existing products and processes and to develop new products as per the emerging customer needs not only due to changing life style but also due to availability of imported ready to use food products in our local retail stores.

Therefore, the aim of present study was to extract starch from local Diamont variety of potato, modification of extracted starch, and comparison of native and modified starches by evaluating their different functional and rheological properties.

MATERIALS AND METHODS

Selection and procurement of materials: Diamant variety of potato was purchased from main wholesale fruit and vegetable market of Karachi, Pakistan. All chemicals used in starch extraction and modifications were of reagent grade.

Extraction of starch from potato: Starch was extracted by the method of Kallabinski and Balagopalan (1994).

Chemical modifications: Acid thinning was carried out in accordance to the method of Gunaratne and Corke (2007) to produce acid thinned potato starch.

Acetylation: The method described by Philips *et al* (1999) was used to prepare acetylated potato starch by adding 12 g acetic anhydride.

Oxidation: The method of Forssel *et al*. (1995) with some modifications was employed for preparation of oxidized potato starch. Starch and water were mixed in a ratio of 1:2. The pH of the slurry was adjusted to 9.5 using 2M NaOH solution. Sodium hypochlorite (10% based on starch weight) was added while maintaining the pH between 9.0-9.5. After addition of NaOCl the reaction was allowed to continue for 10 minutes after complete addition of NaOCl. The pH was then adjusted to 7.0 followed by sedimentation of starch. Water was decanted and starch was washed three times with distilled water before drying at 45°C in a forced air oven.

Dual modification (Acetylation-oxidation): Dual modified acetylated-oxidized starch was produced by using the procedure of Philips *et al*. (1999). The previously produced oxidized potato starch is used for further acetylation which was done as per the above mentioned procedure.

Morphological characteristics: Morphology of native and modified starches was studied by using scanning electron microscope (JSM 6380A, Jeol Japan). Starch samples placed in aluminum specimen holder were coated with gold and examined under different magnifications.

Swelling power: The procedure of Bello-Perez *et al*. (2010) was used for determination of swelling power of native and modified starches.

Solubility: The procedure of Bello-Perez *et al*. (2010) with few modifications was used for determination of solubility of native and modified starches. Starch slurry (2% w/v, starch dry basis) was heated at 60°C, 70 °C, 80 °C and 90 °C for 30 minutes. The slurry was directly made in centrifuge tubes. After cooling the tubes were centrifuged at 1290g for 30 minutes. The supernatant was decanted and residue was dried at 85 °C overnight and weighed. Solubility was calculated as

$$\text{Solubility} = \frac{\text{Initial weight of starch-weight of starch after drying}}{\text{Initial weight of starch}}$$

Water retention capacity: Water retention capacity of potato starch was determined by using the method of Bryant and Hamaker (1997).

Paste clarity: Paste clarity of starch slurry was determined by using method as described by Perera and Hoover (1999).

Pasting properties: Pasting properties of starches like pasting temperature, time to reach peak viscosity, peak viscosity, hot plate viscosity, cold paste viscosity, break down viscosity, setback viscosity of native and modified potato starches were measured by using Brabender microviscoamylograph (Model 803201, Brabender, Germany) equipped with 300 cmg sensitivity cartridge. Ten percent (w/v) starch slurry made in distilled water was heated from 40 °C to 95 °C at a heating rate of 3 °C per minute. The slurry was kept at 95 °C for 10 minutes and then cooled to 50 °C at a cooling rate of 3 °C per minute. The slurry was held at 50 °C for 10 minutes.

Rheological characteristics: Rheological characteristics like firmness, rupture strength, and elasticity were measured on starch gels prepared through micro-visco-amylograph which were poured in 3.5 cm internal diameter plastic bowls, covered with parafilm, and stored in refrigerator. The above mentioned texture based properties were measured by using Universal Testing Machine (Zwick /Roell, GmbH, and Co, D-89079 Ulm). The gel was compressed at a speed of 1 mm per second to a distance of 5 mm using a 5 mm cylindrical probe. The texture based properties of the gel were measured after 24, 48, and 72 hours of storage at 6 °C.

Statistical analysis: All analysis was done in triplicate. One way analysis of variance (ANOVA) was carried out using SPSS (SPSS, Inc, USA) followed by Duncan's multiple range test to separate the mean values.

RESULTS AND DISCUSSION

Morphological characteristics: Morphological properties of native and modified potato starches were studied using SEM (Scanning Electron Microscope) at different magnifications ranging from 100x to 2200x. Approximate size and shape of native and modified starches are presented in Table 1. Generally, the granules of native and modified starches were present in three prominent sizes; small, medium, and large. Similar results were also mentioned by Das *et al*. (2010) for sweet potato. Population of medium sized granules > Population of large sized granules > Population of small sized granules. The small, medium and large sized granules ranged between 7-22 µm, 24-50 µm and 58-111 µm, respectively for native and modified starches. Modifications were not found to significantly affect the granular sizes. Ali and Hasnain (2011) also reported the similar results for modified white sorghum starch. However, acetylated-oxidized starch significantly increased the granular size which could be due to the disruption of intra –granular interactions which subsequently lead to size increment. Spherical, oval and spherical, and oval- shaped granules were observed in small, medium, and large granules, respectively in native and modified starches. All modifications retained the granular structure of potato starch. Similar results are also reported by Singh *et al*. (2004) and Das *et al*. (2010) for potato starch and sweet potato starch, respectively.

Swelling Power: Swelling power of native and modified starches at temperatures from 60 °C to 90 °C are compiled in Table 2. Swelling power is very important phenomenon of starch because it results in higher viscosity in starch-water systems. The results showed that swelling power increased with the rise in temperature both for native and modified starches. Similar results were also reported by Ali and Hasnain (2011) for modified white sorghum starch and by Lawal (2004) for cocoyam starch. At 60 °C, swelling power of all modified starches except acid-thinned starch was significantly higher than native starch. Similarly, at 90 °C, swelling power of all modified starches including acid thinned starch was found significantly higher than native starch. The results are in agreement with those reported by Adebowale *et al*. (2006) for modified sword bean starches. Swelling power of dual modified acetylated-oxidized starch was almost 1.5 times higher than swelling power of native starch at 60 °C and 90 °C. The rise in swelling power could be due to the gelatinization of starches which unfolds the structure of starch granules. The swelling power of acetylated, oxidized, and acetylated-oxidized starches improved due to insertion of functional groups. Since acetyl as well as carboxyl and carbonyl groups are introduced in acetylated-oxidized starch, therefore it demonstrated the highest improvement in swelling power. Acid thinned starches showed the least increment in swelling power which may be due to the fact that acid molecules preferably attack the amorphous region of the starch granule and disrupts it which in turn results in relatively higher crystallinity of starch granule and thus restricts its swelling power. Similar results of acid-thinned starches have also been reported by Ali and Hasnain (2011) for modified white sorghum starch and by Lawal (2004) for cocoyam starch.

Solubility: Solubility of native and modified starches are compiled in Table 3. Generally, all modified starches showed significant increase in solubility as compared to native starch with solubility of dual modified (acetylated-oxidized) starch more than two times as compared to native starch. Increase in solubility of modified starches as compared to native starch may be due to increase in leaching of amylose from the starch granules. Similarly, increase in temperature resulted in leaching of higher amount of amylose from the starch, as granules gelatinize at higher temperature. Solubility of oxidized starch was initially same as native starch but then at higher temperatures it increased significantly as compared to solubility of native starch. Interestingly, it was observed that further acetylation of oxidized starch improved the solubility suggesting further weakening of starch granules. Solubility of acid thinned starch was found significantly higher than native starch within temperature range from 60 °C – 80 °C.

Water retention capacity: Water retention capacity of native and modified starches are compiled in Table 4. Water retention capacity of oxidized and acetylated oxidized starches was found 2.4 times and 1.6 times higher than native starch at 60 °C. Similar results were also reported by Lawal (2004) for cocoyam starch. This behavior of oxidized and acetylated oxidized starches could be due to introduction of functional groups on the starch molecules which facilitate a more enhanced binding capacity than the native starch. Increase of water retention capacity of modified starches as compared to native starch could due to increase in the availability of water binding sites.

Paste Clarity: Paste clarity of native and modified starches are compiled in Table 5. Paste clarity of all modified starches was significantly different than native starch with the paste clarity of acid-thinned and oxidized starches was found prominently higher than all other type of native and modified starches during cold storage. Highest paste clarity (three times than native starch) of acid-thinned starch could be due to low molecular weight, short chains which increased light transmission. Whereas, paste clarity of oxidized starch was higher due to substitution of carbonyl and carboxyl groups combined with depolymerization. Paste clarity of native and all type of modified starches decreased during storage at refrigeration temperature. Increase in opacity of starch pastes on storage is attributed to many factors like leached amylose and amylopectin chains, granular remnants, granular swelling, amylose and amylopectin chain length (Jacobson *et al*. 1997). With the passage of time, paste clarity of all native and modified starches was showing a decreasing trend. Decrease in paste clarity of native, acid thin, acetylated, oxidized, and acetylated-oxidized starches was 23.7%, 26.5, 8.3%, 15.2%, 2.7%, respectively during storage at refrigeration temperature.

Pasting Properties: Pasting properties of native and modified starches are compiled in Table 6. The phenomenon of pasting, following gelatinization of starch, involves granular swelling, exudation of molecular components from the granules and, eventually, total disruption of the granules (Aishat *et al*, 2007). Significantly lower pasting temperature of acetylated and acetylated-oxidized starches than native starch could be due to insertion of acetyl, carbonyl and carboxyl groups. Acetylated-oxidized starch caused a reduction of more than 3 °C in pasting temperature as compare to native starch. The decline in pasting temperature on acetylation is also reported for Thai green and Thai purple canna starches (Saartrat *et al*. 2005). Time to reach peak viscosity of all modified starches was found to be lower than the native starch with acetylated-oxidized starch having half the time required to attain peak viscosity. This reduction in time to reach peak viscosity could be the result of weakening of starch granules owing to chemical modifications. Significant reduction in peak viscosity of acid thinned starch as compared to native starch could be the result of increase in relative crystallinity due to disruption of amorphous region of starch granule. Acid thinned sorghum starches also exhibited low peak viscosity after acid modification owing to the formation of low molecular weight dextrin (Singh *et al*. 2009). Some low peak viscosity Caribbean sweet potato starches are used as ideal types for use in the manufacture of weaning foods (Abegunde *et al*, 2012).

Whereas increase in peak viscosity of acetylated, oxidized and dual modified starches could be the results of increase in their swelling power (Table 2). Liu *et al*. (1999) attributed increased in viscosity of acetylated starches to the weakening of associative forces in amorphous areas of the granules. Hot paste viscosity of acid thinned, and oxidized starches was lower than native starch. Whereas hot paste viscosity of acetylated – oxidized was found to be 1.4 times higher than native starch and might be due to insertion of bulky functional groups. Results showed that acetylated-oxidized starch had the highest break down viscosity indicating weakening of granules due to disruption of electrostatic interactions between the starch chains. Cold paste and set back viscosity shows the tendency of reassociation of starch polymers. Results showed lower cold paste viscosity of acid thinned, acetylated, and oxidized starches whereas higher cold paste viscosity of acetylated-oxidized starch as compared to native starch. This behavior of acid thinned starch may be due to the formation of short chains which has lower tendency of reassociation as compared to long chains. Similarly, the presence of functional groups in acetylated and oxidized starches reduces the tendency of reassociation of starch polymers. This behavior of acid thinned, acetylated, and oxidized starches is also evident from the results of setback viscosity.

Rheological Characteristics: Results of the rheological characteristics of starch gel such as firmness, rupture strength, gel elasticity are shown in Table 7-9. Firmness of oxidized starch > acid thinned starch > acetylated - oxidized starch > native starch > acetylated starch after 24 hours of storage in refrigeration conditions. Depolymerization could be the reason of the highest firmness in oxidized starch. On cold storage for 72 hours, firmness of native starch > acid thinned starch > oxidized starch > acetylated starch > acetylated-oxidized starch. The lowest increment in aging the gel for three days was observed in acetylated-oxidized starch which was 39% lower than native starch and could be attributed to the dual substitution that allows the starch to remain hydrated.

Rupture strength of potato starches followed the order oxidized starch > acid thinned starch > native starch > acetylated-oxidized > acetylated starch after 24 hours of storage under refrigerated conditions. Reason of high rupture strength in oxidized and acid-thinned starch could be due to depolymerization of starch chains. Kim and Ahn (1996) also reported higher gel strength of acid-thinned red bean starches. Increase in rupture strength of starch gels was of the order: native starch > acetylated starch > acid thinned starch > acetylated oxidized > oxidized when measured after 72 hours of cold storage. Reason of the lowest retrogradation in oxidized starch could be the presence of carbonyl and carboxyl groups on polymeric chains of starch

The gel elasticity of native starch after 24 hours storage under refrigeration conditions was higher than modified starches. It was also observed that the gel elasticity of acetylated starch was higher than dual-modified starch. Similar results were also reported by Das *et al*. (2010) and Raina at el. (2006). After storage of 72 hours under refrigeration conditions, the least change in elasticity was observed in native starch whereas the highest change was observed in dual modified starch.

Table 1: Morphological characteristics of native and modified potato starches

Type of starch	Small Granules		Medium Granules		Large Granules	
	Size (µm)	Shape	Size (µm)	Shape	Size (µm)	Shape
Native	7-14	Spherical	28-36	Oval, Spherical	58-64	Oval
Acid Thin	7-14	Spherical	28-36	Oval, Spherical	64-81	Oval
Acetylated	8-13	Spherical	26-38	Oval	62-67	Oval, deformed
Oxidized	7-17	Spherical	24-36	Spherical	71-81	Polygonal
Acetylated- Oxidized	11-22	Spherical	44-50	Oval	94-111	Oval, deformed

Table 2: Swelling power of native and modified diamant potato starch submitted to heat from 60 to 90 °C (g water g^{-1} dry starch)

Temperature(°C)	60 °C	70 °C	80 °C	90 °C
Native	7.14a,1	11.02a,2	14.69a,3	14.31a,3
Acid Thin	7.03a,1	11.6b,2	13.22b,3	15.83b,4
Acetylated	9.03b,1	11.64b,2	14.13c,3	17.65c,4
Oxidized	8.11c,1	13.97,2	18.79d,3	18.89d,3
Acetylated-oxidized	10.99d,1	15.43d,2	19.28e,3	19.44d,3

Values with the same alphabets in a column and same numbers in a row did not differ significantly at ($p< 0.05$).

Table 3: Solubility of native and modified diamant potato starch submitted to heat from 60 to 90 °C (g water g^{-1} dry starch)

Temperature(°C)	60 °C	70 °C	80 °C	90 °C
Native	0.07a,1	0.12a,2	0.12a,2	0.17a,3
Acid Thin	0.11b,1	0.16bc,2	0.17b,2	0.16a,2
Acetylated	0.12b,1	0.14ab,2	0.19bc,3	0.26b,4
Oxidized	0.07a,1	0.17c,2	0.17b,2	0.22ab,2
Acetylated Oxidized	0.15c,1	0.18c,2	0.20c,2	0.21ab,2

Values with the same alphabets in a column and same numbers in a row did not differ significantly at ($p < 0.05$).

Table 4: Effect of temperature and modification on water retention capacity (g/g) of diamant potato starch

Temperature(°C)	60 °C	70 °C	80 °C	90 °C
Native	1.07a,1	1.66a,1	4.35a,2	8.33a,3
Acid Thin	1.24a,1	1.26b,1	9.07b,2	8.43a,3
Acetylated	1.12a,1	1.49c,1	5.45c,2	9.18b,3
Oxidized-	2.6c,1	1.6ac,1	10.73d,2	9.54b,2
Acetylated Oxidized	1.67b,1	2.3d,2	2.34e,2	14.78c,3

Values with the same alphabets in a column and same numbers in a row did not differ significantly at ($p < 0.05$).

Table 5: Effect of storage and modification on paste clarity (%T) of diamant potato starch

	0h	24h	48h	72h
Native	22.09a	19.82a	17.75a	16.85a
Acid Thin	63.59b	58.14b	54.59b	46.74b
Acetylated	19.19c	18.90c	18.06c	17.60c
Oxidized	24.53d	24.19d	22.75d	20.81d
Acetylated Oxidized	20.35e	20.32e	20.23e	19.80e

Table 6: Pasting properties of native and modified diamant potato starch

	T_p (°C)	TTP (Min)	P_v (BU)	H_v (BU)	C_v (BU)	B_d (BU)	S_b (BU)
Native	61.05a	19.725a	545a	445a	831a	100a	386a
Acid Thin	60.8a	16.1b	475b	407.5b	678.5b	67.5b	271b
Acetylated	58.3b	16.75b	685c	493.5c	709b	191.5c	215.5c
Oxidized	60.4a	10.7c	817d	394.5b	678.5b	422.5d	284d
Acetylated Oxidized	57.95b	10.425c	1181e	617d	901c	564e	284d

T_p= Pasting Temperature, TTP, Time to reach peak viscosity, P_V= Peak viscosity, H_v= Hot paste viscosity after 10 minutes holding at 95 °C, C_v= Cold paste viscosity at 95 °C, B_d= Breakdown viscosity (Bd=Pv-Hv), Setback viscosity (S_b=Cv-Hv).

Values with the same alphabets in a column did not differ significantly ($p < 0.05$).

Conclusion: The results revealed that Diamant potato (Solanum *tuberosum*) can be considered as a very potential source of edible native and modified starches for commercial purposes. Ample and cheap availability of raw potato; ease of starch extraction and modification; quality of native and modified potato starches, vast gap in local supply and demand of potato starches, local purchase of imported modified potato starches on a very high price makes it a feasible study not only for large business organizations but also for medium and small size organizations to earn handsome profits, generate economic activities, provide employment opportunities, reduce national burden of payment in foreign currencies and generate foreign exchange through its exports.

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