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Review

Effect of succinylation on the corn and amaranth starch pastes

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Abstract

Succinylation of corn and amaranth starches altered their swelling power as a function of temperature and degree of substitution (DS). Brabender viscoamylogram showed decrease in gelatinisation temperature with increase in DS from 0.05 to 0.20. While the peak viscosity increased with DS for corn starch (CSS), the reverse was found to be true for amaranth starch (ASS). CSS showed good stability in acidic as well as salt containing medium. Both the CSS and ASS exhibited very good freeze-thaw stability and paste clarity. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Corn starch; Amaranth starch; Freeze-thaw; Succinylation

1. Introduction

Succinylation of starch modifies its physicochemical properties, which widens its range of applications in food and non-food industries like pharmaceuticals, paper and textile industries. Modification of native starch to its succinate derivatives reduces its gelatinisation temperature and the retrogradation, improves the freeze-thaw stability as well as the stability in acidic and salt containing medium (Tessler & Wurzburg, 1983; Trubiano, 1987)

Succinylation imparts a hydrophilic character to starch, and it is known to weaken the internal bonding that holds the granules together (Tuschhoff, 1987). Moreover, succinate derivatives of starch show a typical increase in cold-water solubility. The degree of swelling and solubility is dependent on the starch species, and on the type and extent of modification (Buchholz, Dorn & Thomas, 1996; Rutenberg, Jarowenkow & Ross, 1961). For a complete, but very time consuming characterisation of starch, the swelling power may be calculated with 10°C interval in the entire pasting range. Swelling power is a measure of hydration capacity since it is a weight measure of swollen granules and their occluded water.

In the Brabender amylograph, swelling of starch as a function of temperature is continuously recorded as a change in viscosity. At the peak viscosity, some of the granules are broken due to stirring. With continuous stirring, these granules rupture leading to reduction in viscosity. On cooling, starch molecules re-associate or set back to form a gel, the firmness of which depends on the extent of interference; these are altered by modification. The effect of succinylation on the Brabender viscosity characteristics is of great importance to study the viscosity stability. The stability of succinates in acidic and salt containing medium on the Brabender viscosity characteristics is also an important aspect of study. When pasted starch is employed as a thickening agent in foods, the accelerated retrogradation at low temperature may produce undesirable physical changes including gel formation and synerisis. This is particularly objectionable in pie-filling and frozen foods. The freeze-thaw stability of 5% starch paste is determined by alternative freezing and thawing of starch paste. One freeze-thaw cycle is equivalent to 2–3 weeks of frozen storage. The unique property of modified starch is also evidenced in improved freeze-thaw stability (Tuschhoff, 1987). In the case of starch succinate, the hydrophilic groups prevent synerisis when subjected to freeze- thaw cycles (Trubiano, 1987; White, Abbas & Johnson, 1989).

Paste clarity is related to the state of dispersion and the retrogradation tendency of the starch and hence will influence other technologically important qualities of starch. A fairly transparent paste is desirable in fruit pie-fillings, whereas opacity is desirable in salad dressing and instant desserts. Paste clarity is influenced by many factors like concentration, pH, extent of modification and type of modification. Chemical substitution of side chains with succinate group results in the inhibition of ordered structure of starch paste, thus retarding retrogradation and resulting in more fluid paste with improved clarity (Craig, Maningat, Seib & Hoseney, 1989).

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The effect of succinylation on the thermal behavior of starches can also be studied by differential scanning calorimetry (DSC). It is an important technique to study the thermodynamics of starch gelatinisation, and shows the endothermic phenomena during heating of moistened starch in terms of initial, peak and end point of gelatinisation temperatures and thermal energy changes. During gelatinisation, DSC measures the extent of disruption of primary hydrogen bonds that stabilize the double helix within the starch granules and quantifies the heat energy, i.e. the enthalpy involves the transition of the starch from a semi crystalline granule to an amorphous gel (Biliaderis, 1990; Biliaderis, Maurice & Vose, 1980). DSC characteristics are affected by starch type, and by the type and extent of modification (Wootton & Bamunuarachchi, 1979a,b).

In the present work, starch succinates prepared from corn and amaranth starches having a degree of substitution (DS) of 0.05, 0.10, 0.15, and 0.20 were chosen for characterisation and compared to their native counterparts. They have been studied for swelling power, Brabender characteristics, freeze-thaw stability, paste clarity, and thermal behavior by DSC.

2. Materials and methods

Starch succinates of DS ranging from 0.05 to 0.20 prepared from corn and amaranth starches under previously optimised conditions (Bhandari and Singhal, unpublished work) were analysed as follows:

2.1. Determination of swelling power

The swelling power of starches was determined as described by Subramanian, Hoseney and Bramel-cox (1994) with minor modifications. 0.5% suspension (w/w) of starch in distilled water was heated to the desired temperature (45–95°C) for 30 min. Lump formation was prevented by stirring the mixture. It was then centrifuged at 3000 rpm for 15 min. The supernatant was carefully removed and the swollen starch sediment was weighed. Swelling power is the ratio of weight of the wet sediment to the initial weight of the dry starch.

2.2. Brabender characteristics of starch succinates

Viscosity–temperature-relationship was studied on 5% suspension (22.5 g in 450 ml of water) of starch sample on Brabender Viscoamylograph (model PT-100). The samples were heated from 30 to 95°C at 1.5°C/min at a bowl speed of 75 rpm, held at 95°C for 30 min, and then cooled back to 30°C at 1.5°C/min. The results were interpreted with respect to gelatinisation temperature, peak viscosity, viscosity at 95°C, viscosity after holding at 95°C for 30 min and cold paste viscosity at 30°C.

Similarly, studies were carried out with all the starches in the study at pH 4, and in a medium containing 3% sodium chloride. The pH was adjusted with 0.5N acetic acid (Lopez, Carabez & Gonzalez, 1988; Stone & Lorenz, 1984; Trubiano, 1987).

2.3. Freeze-thaw stability

The freeze-thaw stability of the starch succinates was studied by subjecting 5% starch paste to repeated cycles of freezing and thawing and measuring the amount of water separated on centrifuging the thawed paste. The starches were frozen at −10°C for 18 h, and then thawed at room temperature for 6 h. The starch suspension was then centrifuged at 3000 rpm for 10 min. The percentage of water separated after each freeze-thaw cycle was measured and expressed as the percentage of water separated (Eliasson & Kim, 1992; Kite, Maywald & Schoch, 1963; White et al., 1989). All the samples were studied for 10 successive cycles.

$$\% \text{ Synerisis} = \{\text{Water separated (g)}/\text{Total weight of sample (g)}\}100$$

2.4. Determination of clarity

This was carried out as per the method of Craig et al. (1989). If possible '500mg' of the starch succinates from corn and amaranth starches was passed through a standard sieve of 60 mesh and suspended in 5 ml of water in a 10 ml of cotton plugged test tubes. The tubes were then placed in a boiling water bath for 30 min, shaken thoroughly every 5 min, and then cooled to room temperature for about 10 min. The percent transmittance (*% T*) was determined at 650 nm against water blank in a Hitachi U-2001 spectrophotometer.

2.5. Differential scanning calorimetry of native and succinate derivatives of corn and amaranth starches

The instrument used was a DSC 2010 Differential scanning calorimeter from TA instruments, New castle, DE 19720 USA. The following procedure was found to be suitable for the production of gelatinisation endotherms. Four hundred milligrams of starch was intimately mixed with 8 ml of distilled water to give a starch water ratio of 1:20 (this ratio was chosen since it corresponds with formulation of most desserts) in a small glass sample bottle. 8-15 mg sample of the well-mixed starch water suspension was weighed in an aluminium DSC pan, which was then hermetically sealed. The sample pan was placed in a sample holder with the sealed empty pan as a reference pan. A steam of dry nitrogen was flushed through the DSC head at 60–70 ml/min throughout the procedure. All the samples were heated at 10°C/min from room temperature to 130°C (Biliaderis, 1990; White, Abbas, Pollak & Johnson, 1990).

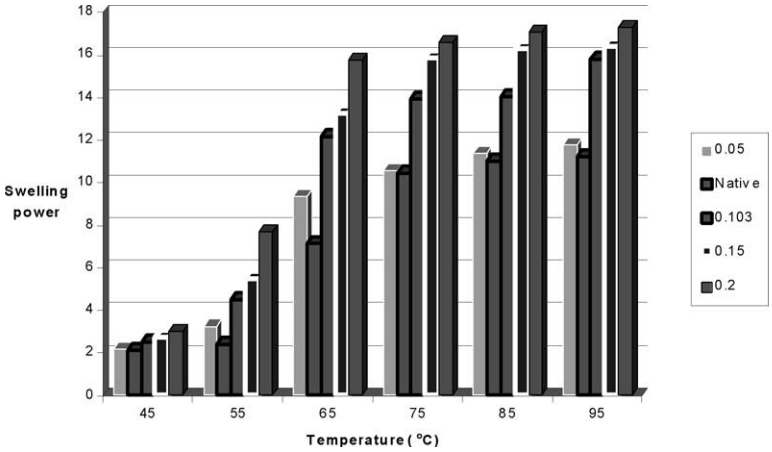

Fig. 1. Swelling power characteristics of native and succinylated corn starch.

3. Results and discussion

Swelling power of native corn, corn starch succinates (CSS) and native amaranth and amaranth starch succinate (ASS) were determined over a temperature range 45–95°C. Fig. 1 indicates that the swelling power of native corn starch increased from 2.1 to 11 in the temperature range of the study, whereas the swelling power of CSS was in the range 2.14 at 45°C for CSS of DS 0.05 to 17 at 95°C for CSS of DS 0.20 for the same temperature range. Moreover swelling power increased substantially from the temperature range 65–95°C for both native corn and CSS. Fig. 2 shows the swelling power data of native amaranth starch and ASS. Native starch showed swelling power in the upward range 3.89–20 with constant increase in temperature from 45 to 95°C, where as ASS showed the range 4.72–22 with increase in DS from 0.05 to 0.20. Swelling of starch was found to be a function of temperature for both the native and the modified derivative; it increased considerably more between the temperature range 60–90°C (Perez, Bahnassey & Breene, 1993; Stone & Lorenz, 1984). The increase in swelling power with the DS could be due to easy hydration. This in turn is a manifestation of the increasing number of hydrophilic groups incorporated in the starch (Wootton & Manatsathit, 1983) It was also observed that amaranth starch and ASS showed greater swelling power than that of corn starch and CSS (Leach, McCowen & Scoch, 1959). This may be attributed to the waxy nature of amaranth starch. Amylopectin is well known to hydrate to a greater extent. The effect appears to become more pronounced after succinylation Lopez & Carabez, 1988).

Tables 1 and 2 show the Brabender characteristics of both the native starches and their succinate derivatives in water and in an acidic medium of pH 4.0. Table 3 shows similar data in a medium containing 3% sodium chloride. From all the tables, it is clear that the amaranth starch showed lower gelatinisation temperature and lower cold paste viscosity than that of corn starch. Although previous work from our laboratories have found relatively higher viscosity for amaranth starch as compared to corn starch, several other researchers have reported the reverse (Lorenz, 1981; Russell, 1987; Sugimoto, Sakamoto, Yamada & Fuwa, 1981; Zobel, 1984). The lower peak viscosity of amaranth starch as compared to corn starch in this study may be due to unknown factors attributed to biological

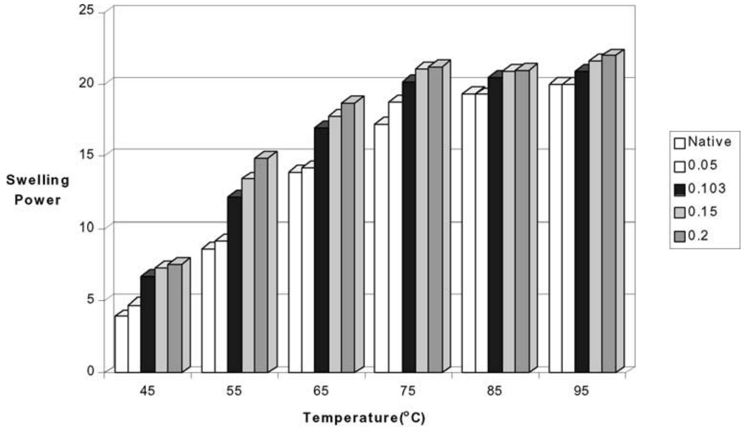

Fig. 2. Swelling power characteristics of native and succinylated amaranth starch.

Table 1
Brabender characteristics of corn starch succinate (CSS) and amaranth starch succinate (ASS) at neutral pH

Sample	Gelatinisation temperature/ pasting temperature (°C)		Peak viscosity (BU)		Viscosity at 95°C (BU)		Viscosity after holding at 95°C for 30 min (BU)		Viscosity after cooling to 30°C (BU)	
	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS
Native	78–85	66.7	120	90	105	90	100	100	190	150
0.05	78–84	65	130	85	90	85	100	90	210	130
0.10	78–82	63	130	75	95	75	95	75	220	115
0.15	80	61	140	75	100	75	100	75	240	100
0.2	76	58	150	70	95	70	100	70	290	85

variations in samples frequently encountered. This could in turn be due to variations in agronomical conditions that may have been employed prior to harvesting. Table 1 indicates CSS to exhibit lower gelatinisation temperature, increased peak and cold paste viscosity with increase in DS, whereas ASS showed exactly opposite behavior of decreasing peak and cold paste viscosity. Amaranth starch consisting wholly of amylopectin is expected to give swollen granules that are fragile. Lack of linear amylose also does not permit amaranth starch to have any setback value. Succinylation increases the hydrophilicity of the starch granules further, making them even more fragile. This feature of amaranth starch in combination with lower cohesive forces could be responsible for decreased peak and cold paste viscosity with increase in DS (Islam & Azemi, 1997; Lorenz, 1981).

Table 2 demonstrates the Brabender characteristics for naïve as well as CSS and ASS for pH 4. For all the samples, irrespective of the starch type and the DS, the peak viscosity, viscosity at 95°C and that obtained after holding at 95°C for 30 min, cold paste viscosity decreased as compared to similar values at neutral pH (in distilled water). This can be explained by the fact that at lower pH the substituent groups exist mostly in acidic form which is less hydrophilic and contribute to considerable drop in viscosities (Trubiano, 1987). However, these viscosities were maximum at 0.2 DS. This indicates CSS to be stable at pH 4. On the other hand, at pH 4 the peak and cold paste viscosities decreased with increase in DS for ASS. Again, the higher amylopectin content of amaranth starch may be responsible for this observation.

Table 3 shows the Brabender characteristics of CSS and ASS and their native starches in a medium containing 3% sodium chloride. Salt is an electrolyte, and also inhibits the swelling of starch. Hence the temperature required for swelling and gelatinisation increased for all the samples including native and CSS and ASS (Trubiano, 1987). The peak and cold paste viscosities for corn starch increased with an increase in DS from 0.05 to 0.20. This shows that the hydrophilicity increased with increase in DS, hence the higher viscosities. However, the gelatinisation temperature increased with an increase in the DS of both CSS and ASS. This is because salt initially retards the absorption of water molecules to the granules, but as the temperature increases, enhances swelling and higher was observed than that of native derivatives (Trubiano, 1987). This again indicates the stability of CSS in a medium containing sodium chloride. The setback value of the starch after cooling reflects the retrogradation tendency of the starch. The role of amylose in this phenomenon is clear from the results obtained with amaranth starch. A very low amylose content of amaranth starch is responsible for weak intermolecular forces within the granules on cooling giving lower cold paste viscosity. Therefore, amaranth showed decreased setback value with increase in DS in the medium containing 3% sodium chloride.

Freeze-thaw stability of native and CSS and ASS,

Table 2
Brabender characteristics of CSS and ASS at pH 4

Sample	Gelatinisation temperature/ pasting temperature (°C)		Peak viscosity (BU)		Viscosity at 95°C (BU)		Viscosity after holding at 95°C for 30 min (BU)		Viscosity after cooling to 30°C (BU)	
	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS
Native	74	62	100	80	80	80	80	85	160	130
0.05	74	62	110	75	90	75	80	80	180	110
0.10	72	60	110	70	90	70	85	72	180	100
0.15	70	58	100	67	95	67	90	70	200	90
0.2	68	55	100	62	100	62	100	65	220	75

Table 3
Brabender characteristics of CSS and ASS in a medium containing 3% sodium chloride

Sample	Gelatinisation temperature/ pasting temperature (°C)		Peak viscosity (BU)		Viscosity at 95°C (BU)		Viscosity after holding at 95°C for 30 min (BU)		Viscosity after cooling to 30°C (BU)	
	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS	CSS	ASS
Native	87	70	100	80	90	80	90	85	170	135
0.05	90	70	100	75	90	75	90	75	170	120
0.10	90	72	110	70	90	70	85	75	190	100
0.15	91	75	120	65	110	65	100	70	200	90
0.2	95	78	130	62	120	62	110	65	200	75

subjected to alternate freeze-thaw cycles is shown in Table 4. Native corn and amaranth showed synerisis from first and second cycle, respectively. Corn starch showed 66% synerisis after 10 cycles, where as native amaranth showed 50.31%, respectively. With increase in DS resistance to synersis improved as indicated by the increased number of cycles showing the resistance to synerisis (Kim & Eliasson, 1993). The poor freeze-thaw stability exhibited by native starches indicates extensive retrogradation during frozen storage. However substitution improved water-holding capacity of the starch gel by decreasing the extent of retrogradation (White et al., 1989). The ability of succinate groups in reducing retrogradation could be attributed to steric effect imposed by the bulky hydrophilic succinyl groups, which prevent alignment of starch chain (Islam & Azemi, 1997).

It was also observed that percent synerisis decreased with increase in DS for both CSS and ASS. CSS with 0.20 DS showed excellent freeze-thaw stability, whereas ASS with 0.20 DS was found to relatively less stable to freeze thaw cycles. These results suggest CSS of 0.2 DS to be useful as excellent thickener for canned, frozen foods, and for soups (Kite et al., 1963).

Percent transmittance of 5% starch paste is a measure of clarity. This was measured for both the starches, CSS and ASS. The results are as shown in Table 5. It was observed that CSS showed better clarity at all the DS chosen in the study as compared to ASS. At a fixed DS, CSS showed better clarity than ASS. Chemical substitution of the hydroxyl group by succinyl moiety causes the inhibition of ordered structure of the starch paste, thus retarding retrogradation and resulting in a more fluid paste with improved clarity. The paste clarity of native amaranth was better as compared to native corn starch. This is because of greater swelling of amaranth, which allows more light to pass through the granules, instead of being reflected. The changes to the granular and molecular structure induced by succinylation facilitated better penetration and absorption of the starch granules, which ultimately lead to more swelling of starch and resulted in more transmittance of light (Craig et al., 1989; Trubiano, 1987).

Table 6 compiles the various parameters that have been obtained from the DSC thermograms of the native starches and the effect of succinylation thereon. The gelatinisation energy is defined as the enthalpy change (ΔH_g) that occurs when a starch–water system is heated under the defined conditions, and is obtained directly as the area under the endotherms in J/g. T_o (onset temperature), T_p (peak temperature), and T_c (conclusion temperature) are the other parameters which can be observed on a DSC thermogram. T_c–T_o indicate the range of temperatures over which the gelatinisation of starch takes place. T_g is the glass transition temperature and indicates the step at which the starch changes from its semi-crystalline state to the plasticised state. T_m is the crystallite melting temperature and indicates the step at which the crystalline structure totally breaks down (Biliaderis et al., 1980; Miller, Gordon & Davis, 1991; Stevens & Elton, 1971; Wootton & Bamunuarachchi, 1979a,b).

ΔH_g value represents the amount of thermal energy involved in the gelatinisation process. At the molecular level, this may be expected to involve the cleavage of existing hydrogen bonds between starch molecules and the formation of new bond involving water to give a less ordered structure with increased entropy (Paton, 1987; Stevens & Elton, 1971). The result of endotherms obtained for native starches, CSS and ASS, showed higher ΔH_g values for both native corn and amaranth starches than their succinylated derivatives. T_p (gelatinisation temperature) values also decreased with an increase in DS upto 0.20 for both CSS and ASS (Wootton & Manatsathit, 1984). T_p, T_c, and T_o values are found to be comparable to that of earlier reports for native corn and amaranth starches (Baker & Rayas-Duart, 1998; Wootton & Bamunuarachchi, 1979a,b). T_p of amaranth starch was found to be lower than corn starch. This may be due to higher swelling power and high amylopectin, which is well known for greater and early hydration. The decreased T_p for all samples of CSS and ASS with increase in DS may be due to bulky hydrophilic groups, which resides in the amorphous regions in the starch molecule. This effectively increases the number of hydrophilic side chains of amylose molecule. This in turn increases the flexibility and reduces the temperature of gelatinisation (T_p).

Table 4
Effect of succinylation on freeze-thaw stability (results are mean ± SD of three determinations) of native and modified derivatives of corn and amaranth starches

Sample	Percent synerisis after freeze-thaw cycles									
	First	Second	Third	Fourth	Fifth	Sixth	Seventh	Eighth	Ninth	Tenth
Native Corn Starch	25.5 ± 0.10	32 ± 0.13	40.56 ± 0.09	48.36 ± 0.04	51.39 ± 0.10	56.78 ± 0.10	61.37 ± 0.21	63.41 ± 0.04	65.19 ± 0.09	66.61 ± 0.19
Modified corn starch derivatives										
0.05	Nil	Nil	5.36 ± 0.04	12.84 ± 0.18	20.49 ± 0.29	24.65 ± 0.32	29.12 ± 0.10	35.91 ± 0.16	37.02 ± 0.01	43.21 ± 0.15
0.10	Nil	Nil	Nil	Nil	3.94 ± 0.08	9.27 ± 0.12	15.37 ± 0.17	19.59 ± 0.18	22.47 ± 0.09	26.94 ± 0.75
0.15	Nil	Nil	Nil	Nil	Nil	2.07 ± 0.08	5.84 ± 0.1	6.99 ± 0.08	9.74 ± 0.10	12.67 ± 0.07
0.20	Nil	Nil	Nil	Nil	Nil	Nil	Nil	1.01 ± 0.14	1.24 ± 0.13	3.06 ± 0.09
Native amaranth starch	Nil	6.04 ± 0.09	12.38 ± 0.18	18.94 ± 0.42	27.91 ± 0.10	34.24 ± 0.34	39.62 ± 0.50	42.56 ± 0.41	47.19 ± 0.13	50.31 ± 0.13
Modified amaranth starch derivatives										
0.05	Nil	3.54 ± 0.08	8.26 ± 0.13	12.85 ± 0.06	17.85 ± 0.10	21.93 ± 0.39	27.71 ± 0.46	31.48 ± 0.12	35.87 ± 0.29	42.63 ± 0.14
0.10	Nil	Nil	Nil	1.87 ± 0.18	3.08 ± 0.05	7.36 ± 0.03	11.91 ± 0.10	16.37 ± 0.08	21.98 ± 0.34	27.26 ± 0.33
Nil	Nil	Nil	Nil		Nil	1.28 ± 0.27	5.92 ± 0.33	10.73 ± 0.32	14.54 ± 0.30	19.46 ± 0.47
0.20	Nil	Nil	Nil	Nil	Nil	Nil	2.07 ± 0.07	4.31 ± 0.33	7.94 ± 0.17	10.54 ± 0.44

Table 5
Effect of succinylation on the paste clarity (results are mean ± SD of three determinations) of corn and amaranth starches

Sample	% Transmittance (at 650 nm)	
	Corn starch	Amaranth starch
Native	6.85 ± 0.03	7.58 ± 0.07
Starch succinates of DS		
0.05	10.85 ± 0.13	9.54 ± 0.08
0.103	16.75 ± 0.22	11.65 ± 0.24
0.15	21.73 ± 0.02	15.94 ± 0.02
0.2	25.39 ± 0.04	18.07 ± 0.07

Further work on evaluating the heterogeneity of degree of succinylation, and the role of the two polymers, i.e. amylose/amylopectin with respect to the polymer properties need to be investigated.

4. Conclusions

Succinylation of corn and amaranth starches improved their swelling power, as well as functional properties like paste clarity and freeze thaw stability. The two starches chosen in the study exhibited good stability at pH 4.0 as well as in a medium containing 3% sodium chloride. These are all attributed to the increased hydrophilicity obtained on succinylation. Such derivatives could find promising industrial applications.

Table 6
Effect of succinylation on the transition temperatures and enthalpy associated with gelatinisation of corn and amaranth starch

Sample	ΔH_g (J/g)	Transition temperature (°C)			
		T_o	T_p	T_c	$T_c - T_o$
Corn starch					
Native	3.827	69.20	73.72	83.06	13.86
0.05	1.113	68.05	72.92	78.06	10.01
0.1	1.089	68.33	72.53	79.53	11.2
0.15	2.005	67.89	72.76	79	11.11
0.20	0.900	68.54	71.55	80	11.46
Amaranth starch					
Native	3.991	67.15	73.14	85.5	18.35
0.05	2.135	60.51	68.15	76	15.49
0.10	1.431	61.65	67.74	75	13.35
0.15	1.614	65.63	72.22	80	14.35
0.2	0.969	59.10	64.65	73	13.9

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