

# Effect of Steam Pressure Treatment on the Physicochemical Properties of *Dioscorea* Starches<sup>†</sup>

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*Dioscorea alata* and *Dioscorea rotundata* starches were subjected to steam pressure treatments (0.35, 0.7, and 1.05 kg cm<sup>-2</sup>) for different periods. The total amylose content in the treated starches was not significantly affected, but the soluble amylose content decreased 3–5-fold. Reducing values and  $\lambda_{\max}$  of the iodine complexes were unaffected. Viscosity of the starch paste was reduced by the treatments, and at higher pressures and longer time of treatments, the peak viscosity values were reduced to very low values. Pasting temperatures were enhanced considerably. Swelling volumes underwent reduction, but no change in solubility occurred. Clarity and paste stability were markedly lowered. The results suggest that starch granules are compressed by the steam pressure treatment, leading to vast changes in physicochemical properties.

**Keywords:** *Dioscorea alata*; *Dioscorea rotundata*; steam pressure treatment; starch properties

## INTRODUCTION

Modification of starch can be achieved by various physical and chemical treatments. The physical treatments which can bring about modifications include  $\gamma$ -irradiation (Raffi et al., 1981; Sokhey and Hanna, 1993), UV irradiation (Soyer and Semenov, 1973), pressure (Mercier et al., 1968; Muhr and Blanshard, 1982; Stute et al., 1996), and heat moisture treatments (Sair and Fetzer, 1944; Sair, 1967; Kulp and Lorenz, 1981; Raja et al., 1987; Larsson and Eliasson, 1991; Hoover and Vasanthan, 1994). The extent of modification by pressure and heat moisture treatments depends on the pressure, temperature, and moisture levels. Mercier et al. (1968) observed depolymerization of potato starch at  $0.6 \times 10^9$  Pa. Kim and Hamdy (1987) found that potato starch was depolymerized in the presence of acid and heat at  $0.3 \times 10^9$  Pa. Kudla and Tomasik (1992a,b) have also studied the effect of pressure and moisture level on starch breakdown. The quantity of water used in heat treatments can be varied to produce different effects (Stute, 1992; Larsson and Eliasson, 1991; Schierbaum, 1966). If excess water is used and the heat treatment is carried out for a long period below the gelatinization temperature of starch, the treatment is termed annealing and the main effects on starch are enhancement in gelatinization temperature and reduction in viscosity. However, if the starch is subjected to heat treatments at moisture levels of 30% and below, the observed effects are less pronounced compared to annealing treatments (Stute, 1992). Similarly the heat moisture treatments changed the X-ray diffraction patterns of potato starch from "B" to "A" (Sair and Fetzer, 1944; Hoover and Vasanthan, 1994), but in annealing treatments, such a change was not observed. On the basis of differential scanning calorimetry, vis-

cography, and X-ray diffractometry, it has been inferred that heat moisture treatments always involve structural alterations, while annealing brings about modifications of binding forces between crystallites and amorphous matrix (Stute, 1992).

In our earlier studies on the effect of steam pressure treatment on cassava starch, considerable reduction in viscosity was observed as the steam-pressure and time of treatment were increased. However, there was only a minor increase in the reducing values of the treated starches, indicating that the major alteration occurred in the granular structure rather than any large breakdown of the starch molecules (Moorthy, 1982). *Dioscorea* starches differ from cassava starch in that they possess a "B" X-ray diffraction pattern and their viscosity properties are different compared to cassava starch. In fact, the *Dioscorea* starches resemble potato starch in many of their properties (Moorthy, 1994). Potato starch is known to undergo considerable changes in its properties during heat moisture treatment (Sair and Fetzer, 1944), and it was desired to find out whether *Dioscorea* starches also undergo similar changes during steam pressure treatments. This paper describes the effect of steam pressure on the physicochemical properties of *Dioscorea alata* and *Dioscorea rotundata* starches.

## MATERIALS AND METHODS

*D. alata* and *D. rotundata* starches were extracted from fresh tubers as previously described (Moorthy, 1991). The starches were subjected to steam pressure treatments (0.35, 0.7, and 1.05 kg cm<sup>-2</sup>) for different periods (5, 15, 30, and 60 min) in an autoclave. The moisture level in the samples was determined by drying in an air oven at 60 °C for 16 h. The reducing values were found out by the method of Schoch (1964) and expressed as ferricyanide numbers. The total and soluble amylose contents were estimated by the procedure of Sowbhagya and Bhattacharya (1971) and Shanty et al. (1980), respectively. For these estimations, six replicates were used. X-ray diffraction patterns were obtained with a Phillips X-ray diffractometer with a chart speed of 5 mm degree<sup>-1</sup> over a scanning range of  $2\theta = 5-40^\circ$  using Cu K $\alpha$  radiation. Crystallinity values were calculated from the graphs. Paste viscosities

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**Table 1. Moisture Content, Blue Values, and Reducing Values of Steam Pressure Treated *Dioscorea* Starches<sup>a</sup>**

steam pressure (kg cm <sup>-2</sup> )	treatment time (min)	moisture content <sup>b</sup> (%)	blue values <sup>c</sup>		reducing values <sup>d</sup>
			total amylose	soluble amylose	
<i>D. alata</i>					
0	0	7.3	0.371 ± 0.0055	0.127 ± 0.0095	0.58 ± 0.05
0.35	5	7.3	0.369 ± 0.0056	0.118 ± 0.0075	0.60 ± 0.03
0.35	15	7.1	0.370 ± 0.0120	0.117 ± 0.0120	0.81 ± 0.04
0.35	30	7.1	0.358 ± 0.0033	0.110 ± 0.0055*	0.69 ± 0.02
0.35	60	6.9	0.351 ± 0.0195	0.094 ± 0.0017**	0.61 ± 0.01
0.70	5	7.3	0.346 ± 0.0235	0.097 ± 0.0010**	0.70 ± 0.05
0.70	15	7.0	0.342 ± 0.0185	0.091 ± 0.0010**	0.72 ± 0.03
0.70	30	7.0	0.339 ± 0.0220	0.079 ± 0.0024**	0.72 ± 0.02
0.70	60	6.8	0.340 ± 0.0170	0.067 ± 0.0022**	0.92 ± 0.03*
1.05	5	7.1	0.350 ± 0.0087	0.096 ± 0.0035**	1.12 ± 0.05**
1.05	15	7.1	0.345 ± 0.0064*	0.076 ± 0.0018**	1.00 ± 0.03**
1.05	30	6.9	0.354 ± 0.0245*	0.075 ± 0.0013*	1.10 ± 0.07**
1.05	60	6.7	0.336 ± 0.0190*	0.060 ± 0.0013**	1.00 ± 0.04*
<i>D. rotundata</i>					
0	0	7.7	0.363 ± 0.0065	0.118 ± 0.0075	0.89 ± 0.06
0.35	5	7.7	0.347 ± 0.0120	0.108 ± 0.0017*	1.00 ± 0.04**
0.35	15	7.6	0.340 ± 0.0082*	0.108 ± 0.0025*	1.16 ± 0.07**
0.35	30	7.5	0.338 ± 0.0075**	0.098 ± 0.0013*	1.00 ± 0.07
0.35	60	7.3	0.340 ± 0.0125**	0.070 ± 0.0014*	1.14 ± 0.05*
0.70	5	7.6	0.335 ± 0.0250**	0.104 ± 0.0075*	1.40 ± 0.1*
0.70	15	7.3	0.340 ± 0.0175**	0.085 ± 0.0012**	1.60 ± 0.06**
0.70	30	7.2	0.328 ± 0.0055**	0.076 ± 0.0022**	1.46 ± 0.07*
0.70	60	7.0	0.338 ± 0.0170**	0.056 ± 0.0007**	1.60 ± 0.11*
1.05	5	7.3	0.334 ± 0.0050**	0.100 ± 0.0018*	1.50 ± 0.06**
1.05	15	7.1	0.339 ± 0.0135**	0.069 ± 0.0033**	1.45 ± 0.05**
1.05	30	6.9	0.341 ± 0.0175*	0.051 ± 0.0009**	2.00 ± 0.11**
1.05	60	6.8	0.340 ± 0.0205**	0.020 ± 0.0022**	2.11 ± 0.06**

<sup>a</sup> Statistical comparison was made for each species with 0.0 sample by Student's *t*-test. One asterisk (\*) indicates significance at  $p < 0.05$ , two asterisks (\*\*) indicate significance at  $p < 0.01$ , and no asterisk means nonsignificant. <sup>b</sup> Moisture content after the treatment. Before treatment it was 7.5% for *D. alata* starch and 7.8% for *D. rotundata* starch. <sup>c</sup> Six replications. <sup>d</sup> Three replications.

were monitored using a Brabender viscoamylograph model 801202 using a 350 cmg cartridge and at a heating rate of 1.5 °C min<sup>-1</sup>. The starch concentration used was 5% (db) in distilled water, and one run was carried out for each sample. Differential scanning calorimetry (DSC) of representative samples of *D. rotundata* starch was obtained with DuPont DSC equipment at a heating rate of 5 °C min<sup>-1</sup> in air using indium as standard. For making starch slurry, the ratio of water to starch was 3:1. For each sample three runs were made.

Viscosities (2%) of the starches were determined in a Redwood viscometer no. 1 (AIMIL, India) at 75 °C according to standard procedure (ISI, 1970) with three replications for each sample. The same solution was used for determination of swelling volume by centrifugation at 2200 rpm for 15 min, and the volume of gelatinous precipitate obtained was noted. Clarity of the solution has been reported as the reciprocal of the absorbance of the 2% solution at 500 nm compared to that of distilled water. The paste stability was the time taken for the starch gel to just start settling as observed visually.

## RESULTS AND DISCUSSION

The blue values corresponding to total and soluble amylose contents in the treated and untreated samples are presented in Table 1. For both *D. alata* and *D. rotundata* starches, there was a slight reduction in blue values corresponding to total amylose for the treated samples, but the reduction was small and became significant only at higher levels of treatment. For *D. rotundata* starch the effect was more significant compared to *D. alata* starch. The effect appeared at the 0.35 kg cm<sup>-2</sup> level, and thereafter the reduction was less pronounced. Thus total amylose content is not very much affected by the treatment. However results of blue values corresponding to soluble amylose showed a definite trend. The soluble amylose content decreased significantly as a function of pressure and time of treatment, and at a pressure of 1.05 kg cm<sup>-2</sup> for 60 min,

the blue values were nearly one-sixth the original values. The effect was more pronounced with *D. rotundata* starch compared to *D. alata* starch. Soluble amylose is distributed in the amorphous regions of the starch granules and hence is easily leached out into hot water. It appears that steam pressure treatment compresses the granular structural organization of the starch, rendering the soluble amylose molecules less mobile. Most of the compressive effect seems to be concentrated in the amorphous regions, since the X-ray diffraction pattern was hardly affected by the treatments. The crystallinity was also not changed to any noticeable extent during the treatments (Table 2). Potato starch undergoes change in the X-ray diffraction pattern from "B" to "A" during heat moisture treatment (Sair and Fetzer, 1944). The absence of such a change in *Dioscorea* starches may be due to a difference in the moisture levels used in steam pressure and heat moisture treatments. Recently Hoover and Vasanthan (1994) observed that heat moisture treatment of yam starches changes the X-ray diffraction pattern from "B" to "A" + "B", which also indicates the difference between these two treatments on the same starch. Another reason for the observed reduction in blue values corresponding to soluble amylose may be the tendency of the compressed amylose molecules in the granules to retrograde easily, leading to a reduction in leaching out into hot water. The absence of change in the X-ray diffraction pattern and the reduction in soluble amylose content point out that the major change takes place in the amorphous regions of starch during the steam pressure treatments.

Heat treatment of starch can either lead to partial or total degradation of starch or may involve only disruption of granular organization of starch. The former effect brings about a perceptible increase in reducing values.

**Table 2. X-ray Diffraction (XRD) Patterns and Crystallinity of the Steam Pressure Treated *Dioscorea* Starches**

steam pressure (kg cm <sup>-2</sup> )	treatment time (min)	XRD pattern	crystallinity (%)
<i>D. alata</i>			
0	0	B	11.7
0.35	5	B	11.2
0.35	15	B	11.4
0.35	30	B	11.2
0.35	60	B	11.3
0.70	5	B	11.5
0.70	15	B	11.0
0.70	30	B	11.3
0.70	60	B	10.9
1.05	5	B	11.0
1.05	15	B	10.9
1.05	30	B	10.9
1.05	60	B	11.2
<i>D. rotundata</i>			
0	0	B	12.2
0.35	5	B	12.2
0.35	15	B	12.4
0.35	30	B	12.1
0.35	60	B	12.3
0.70	5	B	12.0
0.70	15	B	12.1
0.70	30	B	11.9
0.70	60	B	12.1
1.05	5	B	11.9
1.05	15	B	11.9
1.05	30	B	12.2
1.05	60	B	12.0

The results with the steam pressure treatments show only a marginal increase in reducing values during the treatment (Table 1), indicating that starch degradation does not take place to any noticeable extent. Though the increase in reducing values shows some significance at higher levels of treatment, the values do not indicate any high breakdown of starch. The treatments were carried out at low levels of moisture and there was only very little change in the moisture content (Table 1) during the treatment; hence water activity may not be an important factor in these modifications. Depolymerization of amylopectin would also lead to a similar effect, but this may not be very significant since the  $\lambda_{\max}$  of the treated starch was not different from that of untreated starch. High-pressure extrusion of cassava starch (Kim and Hamdy, 1987) and sonification of potato starch (Azar and Hamdy, 1979) have also been found to bring about only small increases in reducing values, while other properties are affected. The results have been explained in terms of changes occurring at the granular level rather than at the molecular level. Treatment of potato starch to high pressures was found to introduce a new band at 630 nm (Kudla and Tomasik, 1992a). However, such an effect was not observed in the present study. The pressure used in the steam pressure treatments was much lower and not enough to bring about the depolymerization observed at higher pressures, which would have led to the new peak.

Viscosity data from Brabender viscosography also point out vast changes due to steam pressure treatments (Table 3). The peak viscosity progressively decreased from 700 to 550 BU at 0.35 kg cm<sup>-2</sup>, 280 BU at 0.7 kg cm<sup>-2</sup>, and 0 BU at 1.05 kg cm<sup>-2</sup> for *D. alata* starch. For *D. rotundata* starch, the corresponding values were 820, 420, 340, and 0 BU, respectively. The significant effect observed in the viscosity cannot be attributed to the breakdown of starch, since the reducing values and gel characteristics of the starch are not affected to any

**Table 3. Viscosity Properties of Steam Pressure Treated *Dioscorea* Starches**

steam pressure (kg cm <sup>-2</sup> )	treatment time (min)	pasting temp (°C)	peak viscosity (BU)	V95 <sup>a</sup> (BU)	VH <sup>b</sup> (BU)	2% solution viscosity <sup>c</sup> (s)
<i>D. alata</i>						
0	0	80–97	750	540	780	180
0.35	5	80–97	800	520	750	140
0.35	15	82–97	750	450	850	55
0.35	30	82–97	620	400	780	40
0.35	60	83–97	550	370	750	35
0.70	5	82–97	650	440	750	100
0.70	15	83–97	450	210	520	40
0.70	30	85–97	400	200	510	35
0.70	60	88–97	280	60	350	30
1.05	5	82–97	580	500	700	45
1.05	15	89–97	120	40	200	38
1.05	30	92–97	80	20	140	30
1.05	60				20	28
<i>D. rotundata</i>						
0	0	80–97	820	570	890	145
0.35	5	81–97	800	540	850	120
0.35	15	81–97	780	550	820	80
0.35	30	82–97	650	450	620	60
0.35	60	85–97	450	270	440	50
0.70	5	83–97	600	320	620	
0.70	15	83–97	540	250	520	65
0.70	30	88–97	460	160	480	55
0.70	60	93–97	340	30	320	40
1.05	5	82–97	580	500	700	45
1.05	15	89–97	120	40	200	38
1.05	30	92–97	80	20	140	30
1.05	60				20	28

<sup>a</sup> Viscosity at 95 °C. <sup>b</sup> Viscosity after holding for 30 min at 95 °C. <sup>c</sup> Redwood viscometer values.

**Table 4. DSC Data<sup>a</sup> on Untreated and Steam Pressure Treated *D. rotundata* Starch**

steam pressure (kg cm <sup>-2</sup> )	treatment time (min)	$T_{\max}$ (°C)	$\Delta H$ (cal g <sup>-1</sup> )
0	0	74.9	2.66
0.35	60	75.1	2.59
0.70	30	84.1	2.77
0.70	60	82.3	2.53
1.05	15	88.9	2.63
1.05	60	87.8	2.73

<sup>a</sup> Mean of three values. <sup>b</sup>  $\Delta H$  = enthalpy of gelatinization.

noticeable extent by the treatments. Raja et al. (1987) also found that viscosity of cassava starch was reduced by steam hydrothermal treatments. Similar reduction in viscosity during heat moisture treatments has been reported for yam and potato starches (Hoover and Vasanthan, 1994). Azhar and Hamdy (1979) observed that the viscosity of potato starch was reduced drastically by sonification but reducing values showed only slight changes. Thus steam pressure treatment brings about only physical changes rather than any major chemical changes in contrast to high-pressure treatments which lead to dextrinization observed by Kudla and Tomasik (1992a). The 2% solution viscosity values also confirm the results obtained with the Brabender viscosograph (Table 3).

Pasting temperatures were steadily enhanced by the treatments. The increase was up to 12 °C. The enhancement in pasting temperatures can be attributed to the compressive effect of the treatment leading to stronger associative bonding between the starch molecules in the granules.

The DSC data (Table 4) also show that onset of gelatinization is delayed by steam pressure treatments. The  $T_{\max}$  increased from 74.9 °C for the untreated

**Table 5. Swelling Volume, Clarity, Paste Stability, and Solubility of Steam Pressure Treated *Dioscorea* Starch<sup>a</sup>**

steam pressure (kg cm <sup>-2</sup> )	treatment time (min)	swelling volume <sup>b</sup> (mL g <sup>-1</sup> )	clarity <sup>b,c</sup>	paste stability (h)	solubility <sup>b</sup> (%)
<i>D. alata</i>					
0	0	18.75 ± 0.73	1.03 ± 0.02	48	10.80 ± 0.26
0.35	5	18.10 ± 1.0	0.83 ± 0.04*	48	10.60 ± 0.36
0.35	15	18.10 ± 0.63	0.76 ± 0.04*	48	10.20 ± 0.35
0.35	30	16.90 ± 0.76	0.74 ± 0.05*	36	11.20 ± 0.26
0.35	60	15.00 ± 0.5*	0.70 ± 0.04*	36	12.00 ± 0.06*
0.70	5	17.50 ± 0.5	0.84 ± 0.06	48	9.80 ± 0.42
0.70	15	13.10 ± 0.14**	0.61 ± 0.06**	36	10.20 ± 0.83
0.70	30	11.20 ± 0.76**	0.59 ± 0.06**	24	10.60 ± 0.15
0.70	60	10.00 ± 0.25**	0.55 ± 0.05**	24	10.50 ± 0.40
1.05	5	18.15 ± 0.28	0.72 ± 0.05*	24	10.20 ± 0.66
1.05	15	10.60 ± 0.14**	0.61 ± 0.04**	12	10.80 ± 0.75
1.05	30	10.00 ± 0.5**	0.59 ± 0.01**	6	12.00 ± 0.75
1.05	60	8.20 ± 0.29**	0.52 ± 0.03**	2	10.20 ± 0.50
<i>D. rotundata</i>					
0	0	25.00 ± 0.25	0.98 ± 0.03	48	12.40 ± 0.42
0.35	5	22.50 ± 0.50*	0.79 ± 0.08	48	12.70 ± 0.86
0.35	15	21.25 ± 0.25**	0.72 ± 0.06*	36	11.80 ± 0.75
0.35	30	20.00 ± 0.50**	0.65 ± 0.08*	36	12.70 ± 0.5
0.35	60	16.25 ± 0.66**	0.65 ± 0.05*	24	13.20 ± 0.29
0.70	5	20.70 ± 0.58**	0.69 ± 0.07*	48	12.66 ± 0.5
0.70	15	18.20 ± 0.29**	0.68 ± 0.04*	36	11.80 ± 0.29
0.70	30	13.80 ± 0.29**	0.62 ± 0.04**	24	11.20 ± 0.29
0.70	60	10.60 ± 0.28**	0.61 ± 0.05*	24	12.6 ± 0.69
1.05	5	20.00 ± 1.00*	0.77 ± 0.10	24	12.20 ± 0.28
1.05	15	16.25 ± 0.75**	0.57 ± 0.07*	6	12.80 ± 0.50
1.05	30	10.50 ± 0.50**	0.55 ± 0.05**	4	12.40 ± 0.50
1.05	60	8.20 ± 0.29**	0.52 ± 0.05**	2	13.00 ± 0.90

<sup>a</sup> Statistical comparison was made for each species with 0.0 sample by Student's *t*-test. One asterisk (\*) indicates significance at  $p < 0.05$ , two asterisks (\*\*) indicate significance at  $p < 0.01$ , and no asterisk means nonsignificant. <sup>b</sup> Three replications. <sup>c</sup> Clarity is the reciprocal of the absorbance of 2% solution at 500 nm relative to water.

sample to 89 °C for the *D. rotundata* starch treated at 1.05 kg cm<sup>-2</sup> for 15 min. The enthalpy of gelatinization was not affected to any noticeable extent, showing that crystallinity is not changed during the treatments.

Swelling volumes of the starches were significantly reduced by the treatments and decreased from 18.75 to 8.2 mL g<sup>-1</sup> for *D. alata* and from 25 to 8.2 mL g<sup>-1</sup> for *D. rotundata* starch (Table 5). This is in contrast to the enhancement in sedimentation volumes observed in the steam hydrothermal treatment of cassava flour (Raja et al., 1987). The reduction in swelling volumes may be due to compression of the granules and thereby strengthening of the associative bonds between the starch molecules. The clarity of the solution of the treated starches was noticeably lower, and the absorbance of the solution of the highest level of treatment was almost twice that of the untreated sample (Table 5). Similarly, the paste stability was also affected very drastically by the treatments, although it was not very evident at lower levels of treatment. At higher levels of treatment, viz. 1.05 kg cm<sup>-2</sup> and 60 min, the paste stability was so poor that the starch gel started settling from the solution within 2 h on standing, in contrast to the solution of the untreated starch which required over 48 h for the starch gel to start settling (Table 5). Steam pressure treatment compresses the starch granules to bring about faster association between the starch molecules. Solubility was not enhanced by the treatments, implying that the starch granules do not undergo breakdown during the treatments (Table 5).

The study thus reveals that starch granules can withstand breakdown during steam pressure treatments but undergo physical changes manifested by change in many physicochemical properties. The changes are quite different from those brought about by heat moisture treatments.

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