

Physicochemical Characterization of Starch From Peruvian Sweetpotato Selections*

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Laboratory starches, isolated from seven sweetpotato selections grown at two locations in Peru, and commercially available Peruvian starch were characterized with regard to physicochemical properties. Chemical analyses included determination of nitrogen, ash, and amylose content. Physical characterization was based on results from differential scanning calorimetry (DSC), swelling power, solubility, and rheology of the pastes. We found that nitrogen and amylose content were not affected by location or selection, while ash content was affected by location. Results from the DSC analysis showed that selection identity

(SI) did not affect DSC enthalpy, onset, peak, and final temperatures. Location affected only peak temperature. Investigation of starch swelling power showed that SI and location did not affect swelling power above 75 °C, and that laboratory starch samples had greater swelling power than commercial samples. Solubility was affected by location, but not by SI. Maximum viscosities of heated starch slurries were not affected by SI or location. Although laboratory and commercial samples did not differ significantly in chemical composition, physical behavior of their pastes was significantly different.

1 Introduction

As is the case for most root crops, sweetpotatoes are a staple food for many developing countries. World production of this crop was seventh after wheat, rice, maize, potatoes, barley, and cassava [1]. Since the starch content of this crop ranges from 30 % to 70 % of the dry matter, developing countries such as China convert a significant part of their production into starch [2]. However, more developed countries are decreasing production of starch from this crop as tariff barriers are lowered. Developing countries continue to have a strong incentive to make starch from this crop as a means to decrease the need for imported materials and, thus, conserve monetary resources for use within the country itself. The incentive is strongest in those countries which can grow this crop year-round because the leafy vines can be used as the "seed" material, instead of using a portion of the roots themselves as the source of transplants. This significantly lowers production costs. Peru falls into this latter category. The purposes of this research were to measure the chemical and physical properties of starch from two types of Peruvian sweetpotato selections and to determine the effect of production location on starch rheological properties.

2 Materials and Methods

2.1 Materials

Starch from two types of Peruvian sweetpotato (*Ipomoea batatas*) selections was used in this study. The DLP designated selections are indigenous and have been grown for long

periods of time in Peru using traditional agricultural practices. Selections of this type may have primitive characteristics [3]. The RCB-designated selections are breeding lines selected to include a variety of valuable traits. The roots of these selections were provided by the International Potato Center, and two replicate samples of each selection were harvested from two locations in Peru. These were in the Cañete River Valley near Cañete and La Molina. Both locations are in the coastal desert of Peru and, thus, require irrigation. La Molina is located in the vicinity of Lima, and the Cañete Valley is located approximately 190 km south of La Molina. Soil types in both locations are medium texture clay loams. To compare the effect of commercial processing to laboratory processing on starch properties, a commercially available sweetpotato starch was procured from a Peruvian source.

2.2 Methods

2.2.1 Starch isolation

Once harvested, the roots were washed, cut into slices (0.5 cm from each end were discarded), frozen at -20 °C, and freeze-dried under vacuum (platen temperature was 20 °C) for 1 week. To obtain the starch, the dried sweetpotato slices were comminuted in a Waring blender at high speed for 30 s with distilled water, and the resulting slurry sieved through four layers of cheesecloth and through a 140 mesh (150 micron) sieve. The sieved slurry was centrifuged at 10,000 g for 16 min at 6 °C, the supernatant was discarded, the settled starch was washed with distilled water, filtered through cheesecloth in a 140-mesh sieve, and centrifuged again. The centrifuging and washing steps were repeated four times. After washing, the starch was allowed to settle under refrigeration for 4 h. After settling, the excess water was decanted, and the discolored top portion of the starch was removed and discarded. The starch was allowed to dry overnight at room temperature and stored in glass containers until used. One

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commercial sweetpotato starch sample from Peru was included and used as standard (ASA Alimentos S.A.).

Starch samples were analyzed for moisture content, Kjeldahl nitrogen, amylose content, energy flow during controlled heating (DSC), swelling power and solubility, and selected rheological properties (Brabender Viscoamylograph and Bohlin Rheometer).

2.2.2 Moisture determination

Approximately 3.8 g of starch (in duplicate) were dried at 105 °C overnight or until constant weight was attained.

2.2.3 Nitrogen determination

Nitrogen content was determined in approximately 1.3 g of starch using a Kjeltac apparatus. Hydrochloric acid, 0.2144N, was used for the titration. Nitrogen was calculated as:

$$\% N = \frac{14.01 (\text{mL acid to titrate sample} - \text{mL to titrate blank}) (\text{normality})}{10 \times \text{sample weight in g} \times \text{dry matter}}$$

The nitrogen content (%) was multiplied by the factor 6.25 to convert it to per cent protein.

2.2.4 Potentiometric determination of amylose

Amylose content was measured by the potentiometric method of Schoch [5]. Contrary to previous reports [6], our preliminary studies showed that defatting the starch samples prior to potentiometric titration with iodine was not required.

2.2.5 Swelling power and solubility

The method cited by Schoch [7] was used. Briefly, weighed starch samples were mixed with water (1 part starch to 60 parts water) and incubated at 8 temperatures, ranging from 60 to 95 °C. After incubation, the amount of water absorbed by the starch (swelling power) and the starch dissolved in the supernatant (solubility) at each incubation temperature were determined.

2.2.6 Differential scanning calorimetry (DSC)

DSC measurements were made with a Perkin Elmer-4 Calorimeter with thermal analysis data station. The instrument was calibrated using Indium, H (fusion) = 6.80 cal · g⁻¹. Starches of known moisture content were weighed (6 to 10 mg of dry starch) into stainless-steel pans, 60 to 70 mg of deionized water were added with a micropipet, and the pans were hermetically sealed. Sealed pans were allowed to equilibrate for 1 h before heating. The reference material was deionized water in a sealed pan. The heating rate used was 10 °C/min from 25 °C to 100 °C. A stream of nitrogen was allowed to flush through the DSC at 30 ml/min throughout the procedure. For each starch sample, three to five determinations were obtained and the mean calculated. The conversion factor, 4.186, was used to convert calories per g to Joules per g (absolute) (1 cal = 4.186 Joules) [8].

2.2.6 Rheology

2.2.6.1 Brabender viscoamylograph

27.6 g of starch (dry weight basis) were weighed and slurried in distilled water (approximately to 6.4 % w/w), giving a total volume of 460 mL, transferred to a Brabender Viscoamylograph, and heated from 25 °C to 95 °C at a rate of 1.5 °C/min. The starch slurry was maintained at 93° to 95 °C for 15 min and cooled at 1.5 °C/min to 50 °C. The bowl of the

Tab. 1. Temperature treatment of starch suspension required for attainment of 50 % of the maximum swelling power.

Sample	Temperature (°C)		Time (min)	
	72		14	
Cultivar	Cañete	La Molina	Cañete	La Molina
DLP2247	71.5	68.2	14.0	13.0
DLP2335	70.5	66.5	11.5	11.5
DLP2478	66.5	65.7	10.0	14.0
RCB25IN	69.2	68.2	14.0	9.0
RCB49IN	69.2	68.2	14.0	10.0
RCB106IN	69.2	65.7	14.0	10.0
RCB179IN	69.2	64.0	14.0	12.0

viscoamylograph was rotated at 75 rpm, and the 700 cmg cartridge was used.

2.2.6.2 Bohlin rheometer

Duplicate amounts of starch were weighed and slurried with distilled water in centrifuge tubes. The final concentration of the slurries was 5.66 % (by weight) starch and 94.34 % (by weight) water. To avoid settling of the starch during the test in the rheometer, each sample was heated in a water bath to the temperature at which 50 % of the maximum swelling power was attained before placing the sample in the rheometer. Times and temperatures used in the pre-treatment are shown in Table 1. After heating in the water bath, the slurries were poured into the cup of a C-14 measuring system of a VOR Bohlin Rheometer operating in the oscillation mode. The experimental conditions used were: torque element, 1.757 g/cm; amplitude, 10 %; strain, 0.0206; frequency, 1 Hz; heating rate, 1.5 °C/min. The samples were heated from the temperature at which 50 % of the maximum swelling power was attained to 95 °C, held at this temperature for 10 min, and cooled at the same rate as in heating, from 95° to 35 °C. Storage and loss moduli (G' and G'') were measured using the oscillatory mode of the Bohlin rheometer. G' is associated with the periodic storage and complete release of energy in a sinusoidal deformation process. G'' reflects the non-recoverable use of applied mechanical energy to cause flow in the specimen.

2.2.7 Statistical analysis

Statistically significant differences (P ≤ 0.05) in the parameters of interest due to selection, location, and the location by selection were determined by analysis of variance using General Linear Models Procedure of the Statistical Analysis System [9].

3 Results and Discussion

The results are reported by the selection identity (SI) first, followed by the location after a semicolon. For example, DLP2247; Cñ or DLP2247; LM, where Cñ refers to Cañete and LM to La Molina.

3.1 Starch composition and thermal properties

The proximate composition and amylose content of the starches is given in Table 2. Starch moisture levels extended from 9.81 to 15.34 %. The protein content, which ranged from 0.04 % to 0.30 %, was affected by neither location nor

Tab. 2. Chemical composition of sweetpotato starch samples.

Sample	Moisture	Dry matter	Protein concentration ^{a)}	ash ^{a)}	Amylose content
	(%)	(%)	(%)	(%)	(%)
ASA	12.63	87.37	0.28	1.765	22.74
DLP2247; Cñ	12.73	87.27	0.29	0.344	25.54
DLP2247; LM	15.34	84.66	0.30	1.321	25.54
DLP2335; Cñ	13.85	86.15	0.13	0.295	23.47
DLP2335; LM	14.17	85.83	0.07	0.324	22.74
DLP2478; Cñ	14.61	85.39	0.25	0.289	21.94
DLP2478; LM	13.00	87.00	0.13	0.805	24.07
RCB25IN; Cñ	11.07	88.93	0.30	0.345	22.21
RCB25IN; LM	12.68	87.32	0.07	0.401	24.74
RCB49IN; Cñ	14.32	85.68	0.35	0.174	20.48
RCB49IN; LM	13.66	86.34	0.04	0.382	24.47
RCB106IN; Cñ	13.45	86.55	0.10	0.177	23.81
RCB106IN; LM	14.41	85.59	0.19	0.411	23.34
RCB175IN; Cñ	14.64	85.36	0.15	0.281	24.54
RCB175IN; LM	9.81	90.19	0.13	0.492	24.00

^{a)} Dry weight basis.

SI. The ash content was affected by location being higher in samples from La Molina (0.59 %) than in those from Cañete (0.27 %). The average percent amylose in the starch ranged from 20.48 to 25.54 %, with neither location nor cultivar having any effect. Others have reported that the amylose content of sweetpotato starch was not affected by the manner of cultivation, nor the year of harvest [7].

Gelatinization enthalpies of the starches as determined by DSC ranged 14.8 to 18.7 J/g (Table 3). This range of enthalpies is slightly greater than those previously reported for sweetpotato starch (11, 12). Onset, peak, and final temperatures ranges of our samples were broader than those reported previously [10, 11, 12, 13]. SI did not have an effect on the

Tab. 3. DSC Onset, peak, and final temperatures and enthalpies for sweetpotato starch samples.

Sample	Temperature (°C)		Enthalpy	
	onset	peak	final	(J/g)
ASA	64.91	75.40	90.07	17.20
DLP2247; Cñ	61.91	71.97	85.30	16.53
DLP2247; LM	61.43	65.81	82.90	18.67
DLP2335; Cñ	58.82	73.40	85.25	15.45
DLP2335; LM	61.63	66.92	83.35	17.46
DLP2478; Cñ	58.03	63.22	80.17	17.87
DLP2478; LM	58.32	66.19	83.90	17.54
RCB25IN; Cñ	60.93	68.71	82.85	15.11
RCB25IN; LM	61.81	66.43	78.50	15.74
RCB49IN; Cñ	61.57	68.54	86.77	16.33
RCB49IN; LM	59.98	66.81	81.00	15.36
RCB106IN; Cñ	62.14	68.82	81.85	14.82
RCB106IN; LM	59.37	64.01	78.33	16.41
RCB179IN; Cñ	63.18	70.25	83.33	16.87
RCB179IN; LM	61.12	65.52	78.77	15.15

endothermic temperature values, nor on enthalpies. However, location did affect peak temperatures, with La Molina samples being about 3 °C lower than those from Cañete. Our results differ from those reported previously in which the enthalpy and the onset and peak temperatures varied with the selection and the environmental conditions in which the plants were grown [10].

Evaluation of the swelling power and % solubility of the starch samples revealed that at temperatures below 75 °C the laboratory samples from La Molina had higher swelling rates than samples from Cañete (Figure 1), while at temperatures above 75 °C, both locations tended to have similar swelling rates. Higher swelling power at lower temperatures might indicate that the amorphous areas in samples from La Molina were more readily available for hydration than starch samples from Cañete. SI did not have an effect on the swelling power. The degree of swelling increased as the temperature approached the gelatinization temperature (58–75 °C) [14]. DLP2247 and DLP2335, both from Cañete, showed a significant increase in swelling between 65° and 75 °C, while all other samples had a linear swelling pattern (data not shown). Selection and location did not have a significant effect on the pattern of the swelling power curves. In comparing the slopes of the swelling power curves, the highest slope value was 0.87 for RCB179IN, and the smallest slope value was 0.59 for DLP2478, indicating a higher degree of association within the granules of the latter sample [15, 16]. ASA reached its maximum swelling power of 25 units at 85 °C, while the highest swelling values for the laboratory samples were at 95 °C.

Starch is used in the food industry as a thickening and stabilizing agent in gravies, soups, puddings, salad dressings, etc. In all these applications, water absorptive capacity of the starch, as well as the texture of the paste formed, are of major importance. If only moisture retention or water-binding capacity of the starch is important, as is the case for surimi (restructured fish protein) or extruded foods, then SI and/or location would have no effect because neither variable affected the swelling power of the laboratory samples. When the objective is to keep the swollen starch granules as fully intact as possible without major dispersion of the internal material, as is the case for ready-to-eat puddings (retrogradation is a negative quality factor), neither location nor SI would have to be taken into consideration because the amount of soluble solids leached from the granule, mainly amylose, was not affected by these variables. If a food processor is interested in whether the maximum swelling capacity of the granules or the temperature at which maximum swelling occurs, then the starch extraction process has to be taken into consideration. Dispersion of individual starch molecules in ASA occurred at higher temperatures than for the laboratory samples. But above 75 °C, the soluble solids of the commercial samples were released more rapidly and in higher amounts than was the case for the laboratory samples. In this regard, if one desires to have a higher water-binding capacity and a small amount of soluble material in the continuous phase of the starch paste, then the conditions followed for starch extraction are an important contributing factor.

The commercial sample, ASA, showed by far the most leaching of any of the starch samples, reaching a maximum of 28 % at 85 °C (Fig. 1). Leaching from this sample was significantly greater than was leaching from the laboratory samples. The maximum percent soluble solids (PSS) for the laboratory samples occurred at 95 °C. The means for Cañete and La Molina were 9.47 % and 10.49 %, respectively. Sta-

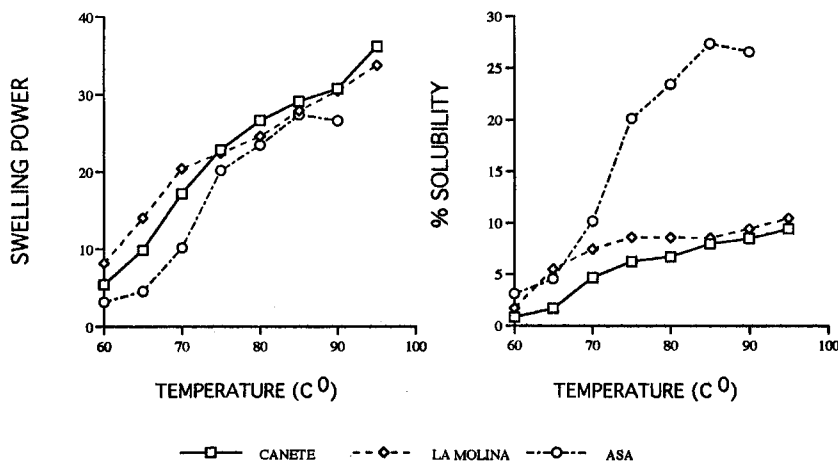


Fig. 1. Swelling power and solubility of sweetpotato starch from seven cultivars grown in Cafete and La Molina, Peru compared with ASA, a commercially available Peruvian starch.

tistical analysis showed that location had a significant effect on the PSS at temperatures higher than 60 °C and that SI had no effect.

3.2 Starch rheological properties

An ideal starch for many food products is one that at low concentrations produces a smooth texture with a heavy-bodied paste, which remains soft and flexible at low temperature and retains its thickening power at high temperatures and high shear. In this respect, the study of rheological properties of major importance.

Due to the small amounts of starch, only the ASA and five selections from La Molina were run in the Brabender Visco-Amylograph. The commercial sample had a pasting temperature of 73 °C, which was within the range of 71 °C to 77 °C reported for sweetpotato starch [17] (Table 4). Selection pasting temperatures ranged from 58.5 °C to 73 °C. Peak viscosities were 640 to 825 BU, close to the range reported of 585 to 715 BU [18]. Since the peak viscosity value indicates how readily the starch granules are disintegrated, cohesive forces within the granules having the higher values are stronger than those having the lower values.

The consistency of the paste after holding at 93 °C for 15 min (breakdown viscosity) provides an estimate of the resistance of the paste to disintegration in response to heating and stirring. Viscosity values for DLP2478, RCB25IN, and RCB49IN (La Molina) increased constantly throughout the analysis without showing a consistency breakdown; thus, the intermolecular forces within the granules are very strong.

Tab. 4. Brabender Viscoamylograph, pasting temperatures, and paste viscosities.

Sample	Pasting temperature (°C)	Peak viscosity (BU)	Peak Viscosity temperature, °C	Viscosity at 93 °C 15 min (BU)	viscosity at 50 °C (BU)
ASA	73.0	680	85.0	570	680
DLP2335; LM	58.5	720	72.0	720	925
DLP2478; LM	73.0	630	93.0	690	860 ¹⁾
RCB25IN; LM	68.5	720	82.0	750	1000
RCB49IN; LM	67.8	825	93.0	870	1000 ¹⁾
RCB106IN; LM	64.5	640	78.3	580	680

¹⁾ at 60 °C.

The commercial sample, ASA, had the lowest breakdown value, meaning that its paste viscosity thins very rapidly with prolonged cooking. Thus DLF2478, RCB25IN and RCB49IN samples from La Molina would be preferred over ASA in those applications in which a prolonged period of high consistency is required.

Setback, defined as the difference between the breakdown viscosity and the viscosity at 50 °C, has been directly related to the amount of amylose leached from the granule [19]. RCB25IN; LM exhibited the highest setback, while RCB106IN; LM showed the lowest setback value. In practical terms, upon cooling, RCB25IN; LM would tend to have stiffer pastes than RCB106IN; LM due to higher amounts of amylose in solution.

With regard to basic rheology parameters, the average behavior of G' , G'' , and $\tan \delta$ summed over all starch samples are shown in Figures 2 and 3. During heating the storage (G') and loss moduli (G'') increased, and the phase angle decreased, indicating a phase change from sol to gel was occurring. Both moduli reached a maximum during heating after which their values decreased. Gelatinized starch systems have been described as suspensions of swollen starch granules in a continuous phase [20, 21]. During heating and before G' and G'' reached maximum values, the starch granules act as rigid fillers within the continuous phase. The initial increase in G' and G'' is due to a progressive swelling of the starch granules so that they finally become close-packed. When the starch granules become very soft, deformable and compressible a decrease in G' and G'' is observed [4]. Figure 2 demonstrates that the elastic nature of the material exceeds the viscous nature of the paste (G' predominates over G''). This is in agreement with previous reports that describe starch pastes like soft solids in which deformations within the linear range imposed by a low strain, will be essentially recoverable [21, 22].

Most of the reported values for G' and G'' refer to starch pastes that have been held at room temperature for several hours after heating. G' for 6% corn and potato starch solutions at 60 °C have been reported to be 132 Pa and 124 Pa, respectively [21]; while G' values of 10% corn starch suspensions at 70 °C have been found to be 1000 Pa [23]. Reported values of G'' for 5% corn starch Suspension at 20 °C and for 4% cassava starch pastes at 25 °C are 497 Pa and 16.57 Pa, respectively [24]. The G' range of our samples at 60 °C was between 82.5 Pa and 229 Pa, with most of the values being between 100 Pa and 198 Pa. The average G'' value of our samples at 35 °C was 5.92 Pa.

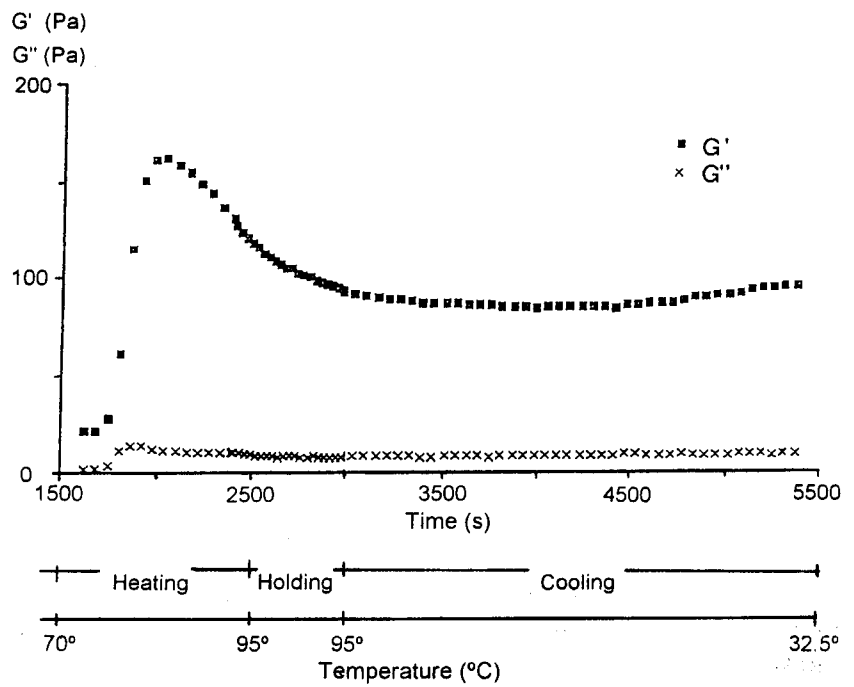


Fig. 2. Mean storage and loss moduli (G' and G'' , respectively) for Peruvian laboratory sweetpotato starch samples.

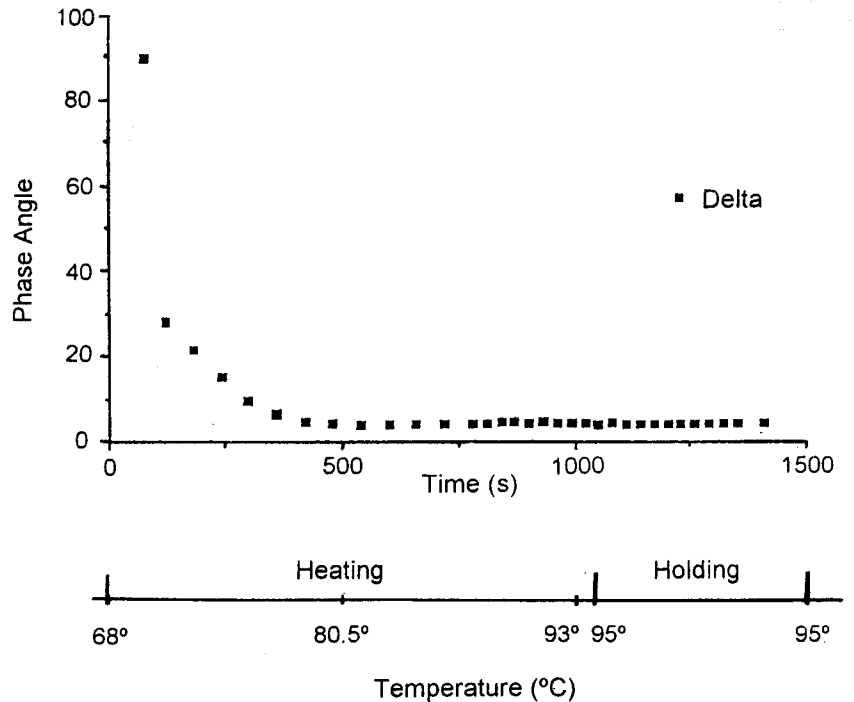


Fig. 3. Mean loss tangent, δ ; (G''/G') for Peruvian laboratory sweetpotato starch samples.

The complex viscosity is defined as the mathematical representation of a viscosity [$\eta^* = (G'/w)^2 + (G''/w)^2$]. To compare our samples, we selected five points from the complex viscosity curves which we designated as follows: peak viscosity (highest viscosity registered), initial and final viscosity at 95 °C (initial and final viscosities during the holding period), lowest viscosity (minimum viscosity value obtained during the entire treatment), and viscosity at 35 °C (viscosity at the end of the cooling period). The lab samples had higher peak viscosity values than ASA (Table 5). If the end use depends on relatively low peak viscosities, as is the case for pumping or sterilizing can products, ASA would be preferred. If on the other hand, the

product requires high viscosity values, the lab samples would be preferred.

Location was found to have a significant effect on the values of initial and final complex viscosities at 95 °C, lowest complex viscosity, and complex viscosity at 35 °C. These complex viscosity values were lower for samples from La Molina. It is possible that location influenced the complex viscosity after attaining a maximum value by affecting the temperature at which the peak complex viscosity was reached. Peak complex viscosity temperatures were lower in samples from La Molina than in those from Cañete (Table 5). The lab samples had higher initial and final complex viscosities and complex viscosity at 95 °C, as well as lower com-

Tab. 5. Complex viscosity values of sweetpotato starch samples.^{a)}

Sample	Complex Viscosity (Pa.s)					Peak viscosity temperature (°C)
	heating	holding		cooling		
	peak	initial at 95 °C	final at 95 °C	lowest	at 35 °C	
ASA	25.75	20.30	14.85	13.55	15.35	
DLP2247; Cñ	54.35	44.65	37.60	35.85	38.15	84.0
DLP2247; LM	58.3	27.30	19.60	19.00	23.75	81.1
DLP2335; Cñ	49.35	34.25	26.15	25.45	28.95	78.5
DLP2335; LM	47.00	28.80	23.10	22.60	24.90	81.1
DLP2478; Cñ	63.20	42.10	31.20	30.00	34.30	81.1
DLP2478; LM	37.05	32.60	25.90	24.10	25.80	79.6
RCB25IN; Cñ	44.05	32.80	26.60	25.30	27.60	81.4
RCB25IN; LM	50.45	28.90	22.20	21.70	25.15	77.1
RCB49IN; Cñ	50.00	34.50	26.95	25.75	27.80	77.8
RCB49IN; LM	56.43	35.85	26.10	25.40	30.20	87.9
RCB106IN; Cñ	50.87	36.20	29.00	28.00	31.75	77.1
RCB106IN; LM	47.20	23.45	16.15	15.85	20.50	79.2
RCB179IN; Cñ	51.40	39.10	30.40	29.50	33.30	78.5
RCB179IN; LM	43.50	20.20	16.00	15.80	20.20	75.4

^{a)} determined by oscillatory rheometry using the Bohlin VOR Rheometer.

plex viscosities and viscosities at 35 °C than ASA. The former samples would be preferred over ASA in applications that require high consistency at elevated temperatures for prolonged periods of time as in the case of ready-to-eat food.

4 Conclusions

In this research, starches from sweetpotato roots of seven selections from two locations in Peru were extracted in the laboratory and their physicochemical characteristics were determined and compared with a commercially available sweetpotato starch. We found that SI and location of the laboratory starches affected some DSC parameters, indicating that starch granule organization was somewhat dependent on SI and location.

Swelling power and solubility of the laboratory starches were only slightly affected by location, while SI had a minimal effect on swelling power but a larger effect on solubility, again suggesting starch granule organizational differences. Rheological differences of the gelatinized laboratory starches, on the other hand, were affected by location alone, indicating that it was responsible for differences in the particle to particle interaction of the gelatinized material.

Although laboratory and commercial starch samples did not differ significantly in chemical composition, the physical behavior of their pastes was significantly different. Commercial starch samples had lower swelling power and viscosity and higher endothermic temperatures and solubility than did the laboratory samples.

Laboratory and commercial starch samples did not differ significantly in chemical composition, but physical behavior of their pastes was significantly different. Commercial starch samples had lower swelling power and viscosity, and higher endothermic temperatures and solubility.

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