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Influence of the surface/volume ratio on the rheological properties of starch dispersions

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Abstract: This study aims to evaluate the influence of the surface/volume granule ratio of amaranth, corn, and potato starches on the rheological properties of pastes and gels obtained at 5 and 10% solids concentration through granulometry, scanning electron microscopy, rotational, and dynamic rheological measurements. The granule size distribution and the surface/volume ratio drove the rheological behaviour as a function of temperature and concentration. At a concentration of 10%, the consistency index of corn starch (CS) paste was 147.25 Pa·s^n (n – flow behavior index), a higher value compared to pastes from potato starch (PS), 86.54 Pa·s^n, and amaranth starch (AS), 44.48 Pa·s^n. The lowest values of the loss angle tangent (Tan δ) in CS (0.052 and 0.035) at both solids concentrations suggested a better gel conformation. Noticeable changes in consistency index and storage modulus were observed in CS. A theoretical analysis of the surface/volume ratio change showed that CS reached a 2.2 value, much lower than the 4.5 and 5.8 values for PS and AS, respectively. These findings provide additional criteria for the food industry when choosing starches with suitable rheological behaviour as a function of surface/volume granule ratio and solids concentration.

Keywords: granulometry; Pasting properties; polymer gels; rheology; solids concentration

Starch is a renewable biopolymer with several industrial applications, as it is widely used to modify texture properties (Vamadevan and Bertoft 2015) as a thickening or gelling agent. When the starch-water mixture is heated above the gelatinisation temperature, the gelatinisation allows absorbing large amounts of water, swelling to several folds over their initial size and reaching a high volume and superficial area. Upon cooling, the system forms a gel composed of swollen starch granules in an amylose matrix (Genovese et al. 2003), or a starch paste conformed by a mixture of swollen granules suspended in a continuous phase composed mainly of amylose (Lagarrigue et al. 2001).

The granule size distribution and mean diameter are important parameters determining the viscosity of gelatinised dispersions (Okechukwu et al. 1996). The rheological behaviour of starch dispersions depends on their size, shape, size distribution, volume, and the interaction between the granules and the solids concentration. The swelling behaviour of starch granules is hindered at high concentrations, and the rheological properties, including flow and viscoelastic behaviour, are strongly influenced by the physical state of the granules (Genovese et al. 2003). Rheological characterisation of the starch dispersion would allow selecting starch granules of specific size distribution

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associated with surface and volume quantities to obtain different rheological properties in new product development (Rao et al. 1997).

The granule size and its ability to swell (reaching a specific volume) determine the functional and physical properties, as demonstrated in a study where the evolution of granule size during swelling was independent of the solids concentration up to 8%. However, at 10%, the swelling decreased because of the limited availability of water. These results suggest that the solids concentrations higher than 8% have an important effect on the surface/volume ratio affecting the rheological properties (Waterschoot et al. 2016).

There is no information on how the interactions due to changes in the surface/volume ratio of starch granules of different sizes are affected by the solids concentration and their influence on the rheological behaviour. Assessing these impacts could help the industry to select the best alternative when seeking a desirable texture or specific rheological properties. The objective of this study was to evaluate the influence of the surface/volume granule ratio of amaranth, corn, and potato starches on the rheological properties of pastes and gels obtained at 5 and 10% solids concentrations. Also, the relationship between the starch suspension properties and the change in the surface/volume ratio for each starch type is described.

MATERIAL AND METHODS

Material. Corn starch (CS) was provided by Cargill (USA). Potato starch (PS) was purchased from Sigma-Aldrich (CAS 9005-25-8, USA). Amaranth starch (AS) was isolated from grains using the method reported by Wang and Wang (2004). For this study, 5 and 10% solids contents were chosen based on the rheological behaviour of starch suspensions with no restriction of available water.

Scanning electron microscopy (SEM). The samples were placed on the sample holder using a conductive double-sided carbon tape and observed with an electron beam of 20 kV/15 A in an ambient scanning electron microscope (JEOL JSM-6300; JEOL Ltd., Japan).

Particle size distribution. The measurement was carried out at different temperatures by triplicate using laser light scattering granulometry (Cilas 1640; France) following the methodology described elsewhere (Herrera-Gómez et al. 2005).

Rheological measurements. Pasting profiles were obtained from a rotational test using a rheometer (Physica MCR 101; Austria). The samples in triplicate were placed between two 25 mm diameter parallel plates (upper plate sandblasted) with a 1 mm gap. The test was conducted at a 50 s^{-1} shear rate and a 2.5 °C·min^{-1} rate for heating and cooling periods. To avoid water loss by evaporation, mineral oil was used to cover the plates during the test. The temperature profile started with a heating ramp from 25 to 90 °C, maintaining for 5 min, cooling to 25 °C, and maintaining 5 min before applying 3 measurement cycles: the first cycle up from 0.01 to 100 s^{-1} and the second and the third cycle up-down from 100 to 0.01 s^{-1}. The Ostwald-de Waele equation was used to calculate the consistency index (k, Pa·s^n) and the flow behaviour index (n, dimensionless).

The same temperature profile and operation parameters were used to measure the viscoelastic properties by oscillatory tests at 1 Hz and 0.3% strain. The linear viscoelastic region (LVR) was defined using oscillation stress sweeps at 25 °C. Frequency sweeps (0.1–10 Hz) were carried out at a 2% strain value.

Analysis of the surface and volume of starch granules. A simplified model was defined based on spherical approximation used in granulometry as follows (Equations 1–6):

$$S = 4\pi r^2 \tag{1}$$

$$V = \frac{4\pi r^3}{3} \tag{2}$$

$$S/V = \frac{3}{r} \tag{3}$$

where: S – surface; V – volume; S/V – surface/volume; r – radius.

Suppose granules are simplified to spheres with d_i diameter. In that case, we can define N_{d_i} as the quantity of granules per 1 g of starch, calculated by

$$N_{d_i} = \frac{P_{d_i} \times V_{1g}}{V_{exp}} \tag{4}$$

where: V_{1g} – volume of 1 g of starch; V_{exp} – volume of 100 particles measured by granulometry.

The four parameters were calculated using the following procedure:

The total surface of granule distribution for a specific granule size S_i is the sum of particular surfaces s_i multiplied by the quantity of granules N_{d_i}:

$$S_i = N_{d_i} \times s_i = N_{d_i} \times \frac{\pi d_i^2}{4} \tag{5}$$

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The integrated surface IS_j is the cumulative sum of total surface values for all size granules lower than or equal to the specific size value d_j.

$$IS_j = \sum_{i=1}^{j} S_i = \sum_{i=1}^{j} \frac{\pi d_i^2}{4} \qquad (6)$$

The maximum size for each starch granule is the biggest granule size measured. At this point, the integrated surface parameter reaches its maximum value.

The total surface change is the difference between the maximum of the integrated surface for swollen granule distribution and the native granule distribution.

Statistical analysis. Statistical analysis was carried out through the analysis of variance (ANOVA) using Statistica 7.0 (Statsoft Inc., USA). To determine the significant difference between values, the Fisher's LSD (least significant difference) test was used at $P = 0.05$.

RESULTS AND DISCUSSION

Particle size distribution and shape of granules. Starch granules had different sizes depending on the source, and their particle size distribution was unimodal or multimodal (Tester et al. 2004). In this study, CS showed large and polygonal granules, while the small granules were circular with a rough surface (Figure 1). For PS, small granules had a circular shape, while large granules had an elliptic shape with a smoother surface than CS. These morphologies are similar to those reported by Tester et al. (2004). AS granules had a polygonal shape with a regular size distribution like those reported by Kong et al. (2009).

AS showed a granule size distribution ranging from 0.1 to 9.0 µm, with an average size of 1.5 µm. For CS, a multimodal population, ranging from 0.7 to 28 µm, was observed, with a predominant population averaging 17 µm. The granule size in PS showed a noticeable unimodal distribution with a population averaging 50 µm. Some starches have a multimodal distribution, and each population could have different physicochemical properties, resulting in different end-use potential. In this study, AS and PS samples showed a unimodal distribution, while CS had a multimodal particle size distribution, which is consistent with previous reports (Singh et al. 2003).

Rheological properties – pasting formation. Pasting includes the morphological modifications of starch granules, from swelling to rupture, and the lixiviation of granular molecular components, particularly amylose. The kinetics of these events determines the characteristics of starch pastes (Matignon and Tecante 2017). Figure 2 shows the pasting profiles of native starches. PS had the highest viscosity values, and the unexpected behaviour of CS was noticeable. At 5% solids content, both CS and AS showed a weak response, and the differences in granule sizes were not responsible for this behaviour because their gelatinisation temperatures

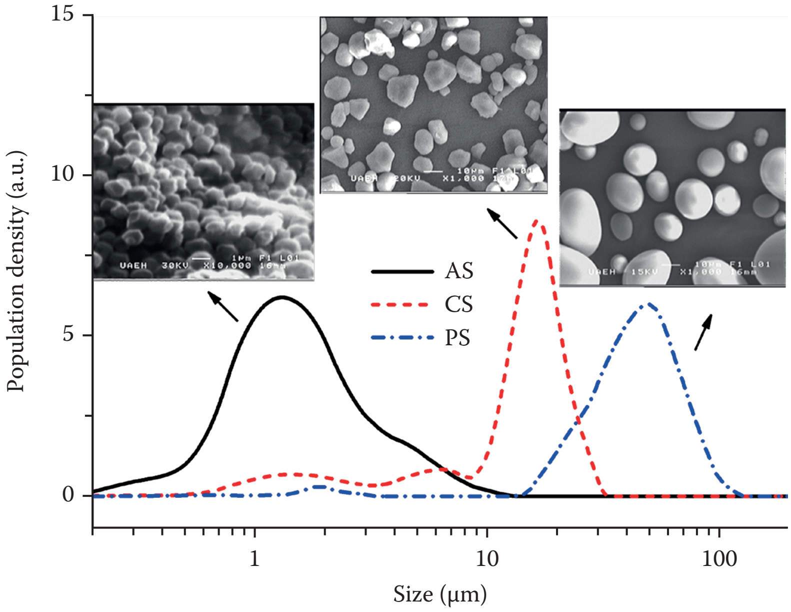

Figure 1. Scanning electron microscopy (SEM) images and population density as a function of the size by granulometry for native starches

AS – amaranth starch; CS – corn starch; PS – potato starch; a.u. – arbitrary units

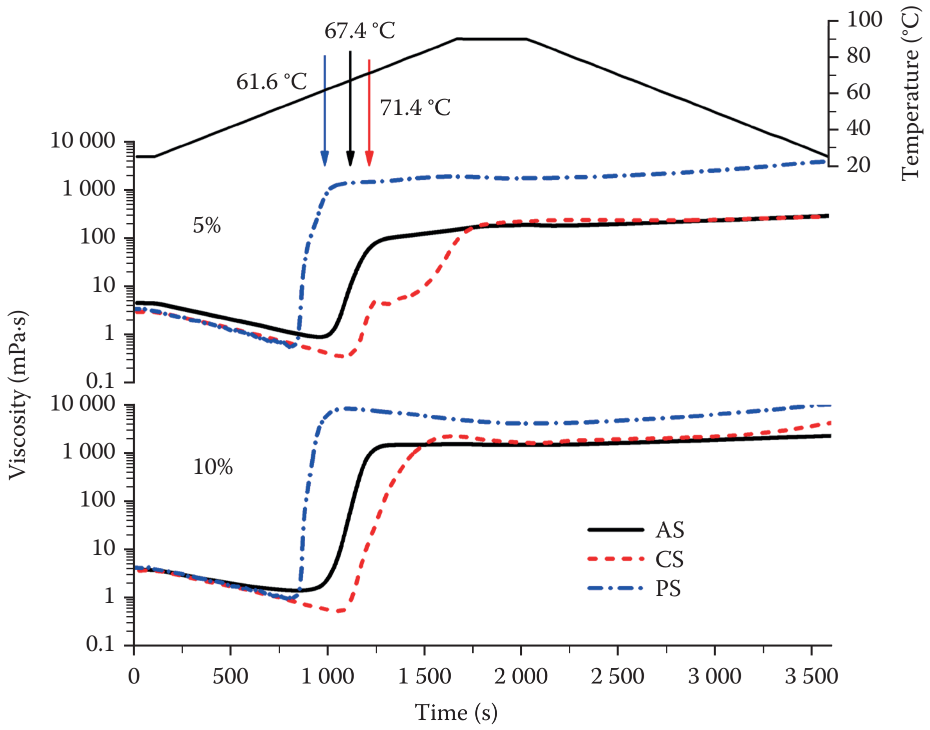

Figure 2. Paste formation for native starches at 5 and 10% of total solids

AS – amaranth starch; CS – corn starch; PS – potato starch

were different. At 10% solids content, the pasting profiles of both CS and AS showed a significant increase. This behaviour, as a function of solids concentration, could be explained by an increment in the contact surface of the granules when the surface/volume ratio granule increased, which is proportional to shear forces among granules. In the present study, the granule size distribution significantly affected the rheological properties during paste formation as the flocculated swollen starch granules fill the system's total volume, promoting granular interactions and notoriously increasing the viscosity values. Also, this effect was closely related to the behaviour observed in the theoretical analysis of the surface/volume granule ratio, as described next.

During swelling, granules keep their integrity, and the change in viscosity is due to space-filling effects arising from changes in their surface/volume granule ratio. A large change in viscosity is expected when big granules swell, increasing intergranular contact at a lower concentration than smaller granules. Waterschoot et al. (2016) reported a low peak viscosity for their blends at low starch concentrations (< 6%) due to reduced swelling. At high concentrations (> 6%), close packing was reached early during the temperature-time profile. The behaviour observed at 10% solids concentration could be attributed to a mixed effect of swelling, packing concentration, size distribution, and surface/volume granule ratio. The high values of peak viscosity of CS could have been due to the multimodal wider size distribution resulting in different swelling patterns in the heterogeneous starch system and promoting the entanglement of the polymeric chains in the starch dispersion. Wang et al. (2022) reported that the gelation mechanism influenced the reassociation and structure alignment, modifying the matrix conformation. Cabrera-Ramirez et al. (2020) explained the effect of different starch granule sizes on the properties of popped sorghum. A similar approach was followed to present the results of this work.

Rotational test – flow curves. The power law was fitted to experimental data, and all R^2 values were higher than 0.98 (dimensionless). The values for the flow behaviour index (n) and consistency index (k) obtained are shown in Table 1. All starches showed a non-Newtonian fluid behaviour ($n < 1$). At 5% solids and smaller granule size, the value of n was minor, but this trend was not observed at 10% solids content. CS had the lowest value of n, meaning a higher dependence on the shear rate and a more significant deviation from the Newtonian fluid behaviour.

Changes in n values could be due to granule size and solids content differences. At a high concentration, the

Table 1. Rheological parameters of the power law model (Ostwald-de Weale) describing the flow behaviour of amaranth, corn, and potato starches as a function of solids concentration

Sample	Solids concentration (%)	R^2	n	k (Pa·s^n)
AS	5	0.994 ± 0.000^d	0.259 ± 0.005^d	4.53 ± 0.42^a
	10	0.997 ± 0.000^a	0.214 ± 0.011^c	44.48 ± 5.93^c
CS	5	0.981 ± 0.001^b	0.305 ± 0.004^a	3.42 ± 0.22^a
	10	0.999 ± 0.000^e	0.148 ± 0.014^b	147.25 ± 7.95^e
PS	5	0.996 ± 0.000^a	0.378 ± 0.002^e	29.60 ± 2.76^b
	10	0.993 ± 0.000^c	0.296 ± 0.018^a	86.54 ± 12.26^d

$^{a–e}$ means within the same columns followed by different superscript letters are significantly different ($P < 0.05$); n – flow behaviour index; k – consistency index; AS – amaranth starch; CS – corn starch; PS – potato starch; R^2 – simple correlation coefficient

orientation of the granules in the direction of the shear force was more difficult, increasing the dependence on the shear rate. In addition to the concentration, the increasing polydispersity of the gelatinising granules and the changes in the contact area appeared to be important parameters to increase the shear thinning behaviour of starch dispersions. The dominating role of the dispersed phase in the rheological behaviour of starch dispersions was also demonstrated by the strong effect of the granular mean diameter (Okechukwu et al. 1996).

The consistency index (k) values increased with the solids concentration, indicating a higher granular interaction in the pastes. The lowest and the highest k values were observed in CS at 5 and 10% solids content, respectively, increasing by a factor of 45. The high consistency index value could be explained by the broader granule size distribution since the small granules affect the conformation of the paste, changing the average surface/volume granule ratio.

Viscoelastic properties – gel conformation. Starch gels are constituted by an 'infinite' tridimensional reinforced network (Tecante 2001). This network's elasticity increases with the granules' volume fraction (Matignon and Tecante 2017) as unswollen, or partially swollen granules are less deformable than fully swollen granules (Doublier et al. 1987).

The viscoelastic properties of gels are shown in Figure 3. All starches exhibited a solid-like be-

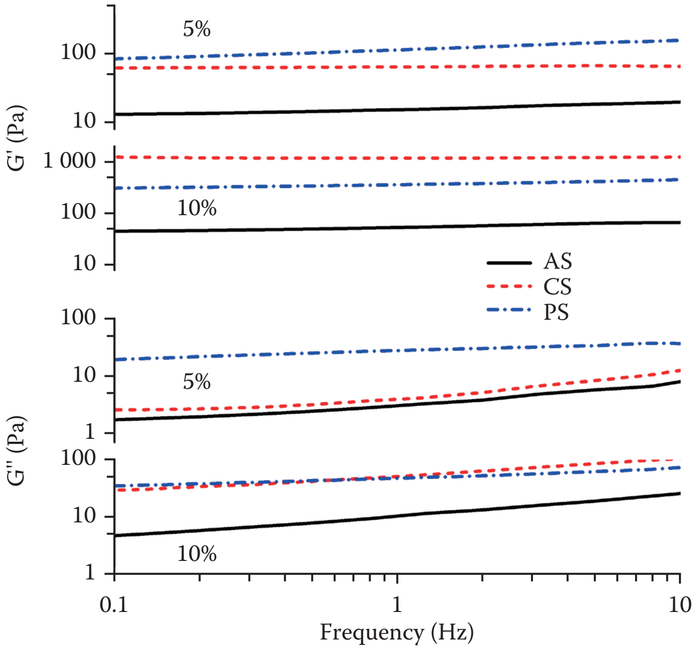

Figure 3. Frequency sweep of dynamic moduli of native starches, storage modulus G' (Pa) and loss modulus G'' (Pa) at 5 and 10% of total solids

AS – amaranth starch; CS – corn starch; PS – potato starch

Table 2. Viscoelastic parameters of native starch gels at 25 °C analysed by dynamic viscoelastic measurements at 0.5 Hz, for 5 and 10% solids percentages (*w*/*v*) for native starches

Sample	Total solids (%)	G' (Pa)	G'' (Pa)	Tan δ (–)
AS	5	14.46 ± 0.15^{a}	2.44 ± 0.14^{a}	0.169 ± 0.008^{c}
	10	50.53 ± 2.02^{a}	7.93 ± 0.21^{c}	0.157 ± 0.008^{b,c}
CS	5	63.86 ± 25.43^{a}	3.17 ± 0.69^{a}	0.052 ± 0.01^{a}
	10	1202.3 ± 206.5^{c}	42.2 ± 3.14^{b}	0.035 ± 0.004^{a}
PS	5	103.33 ± 4.16^{a}	25.4 ± 1.75^{d}	0.246 ± 0.026^{d}
	10	346.66 ± 10.78^{b}	43.33 ± 1.76^{b}	0.125 ± 0.005^{b}

$^{a-d}$ means within the same columns followed by different superscript letters are significantly different ($P < 0.05$); G' – storage modulus; G'' – loss modulus

haviour since G' > G''. Also, G'' showed a slight dependence on frequency. G' values were almost two orders of magnitude higher than G'' values confirming the gel-like behaviour of starch dispersions (Tecante 2001) and describing a network with an elastic response. CS samples exhibited a rather unexpected behaviour since their moduli were lower than those in PS at 5% solids concentration but higher at 10%. The values at 0.5 Hz are presented in Table 2 to compare the storage modulus at 5 and 10% solids concentrations. A value was almost 20 fold higher for CS than for AS and PS. In the case of PS, the large granules resulted in higher storage and loss moduli than those of the small granules of CS and AS, but only at 5% solids concentration. All starches formed gel structures, but the granule interaction was hindered to a greater extent in CS, probably associated with the granule size distribution, swelling power, and surface/volume granule ratio. The limitation in swelling conditions for bigger granules resulted in a gel with a more compact structure.

To explain this behaviour, the loss angle tangent or Tan δ (G''/G') was calculated during retrogradation as this parameter is useful to describe the viscoelastic behaviour of materials. Figure 4 shows a maximum value of around 1.5 for AS, having a significant effect on the viscous component, i.e. the starch-water dispersion began with a mainly liquid behaviour when the reversible swelling of granules took place (González-Reyes et al. 2003). The average values of PS and CS, 0.2 and 0.1, respectively, slightly decreased at both solids concentrations. Tan δ values near 0.1 indicate glassy crystalline polymers and gels, more structured systems (Steffe 1996). Values of Tan δ were lower than those of potato starch reported by Singh and Kaur (2004), who mentioned that a decrease in Tan δ during cooling indicated the formation of strong network structures and a more solid-like behaviour. In this study, the decrease in Tan δ values was

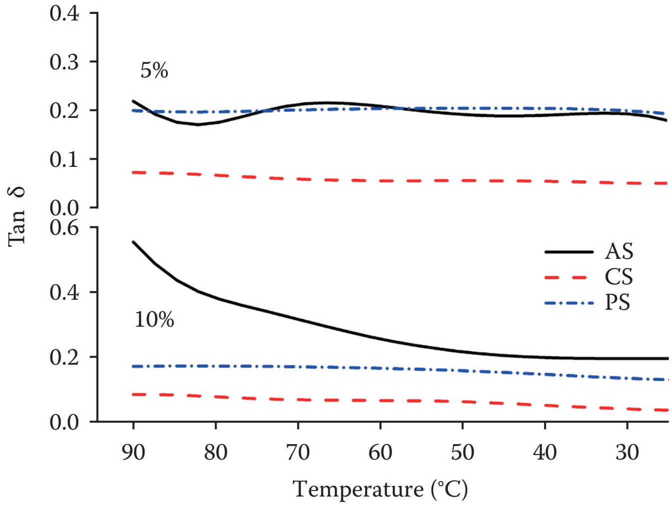

Figure 4. The behaviour of Tan δ at 5 and 10% of total solids for native starches

AS – amaranth starch; CS – corn starch; PS – potato starch

non-uniform for AS, probably because of the small size of the granules, which affected the granular interaction during the gel conformation as well as the lower amylose content (12% compared to 26.7% and 26.1% in PS and CS, respectively (Fonseca-Florido et al. 2016; Fonseca-Florido et al. 2017). The changes in Tan δ were more noticeable for CS at both solids concentrations, describing a stronger matrix.

Surface/volume granule ratio. It is important to mention that the granule size distribution and its associated surface and volume quantities can drive the behaviour of starch suspensions with a high dependence on the solids concentration. To show the importance of the surface/volume granule ratio, a theoretical analysis of the total surface and volume was carried out at 10% solids content, as the granular interactions affecting the properties of the starch suspensions were more important at high concentrations. Figure 5A shows the surface and volume of a sphere as a function of its diameter (Romes-

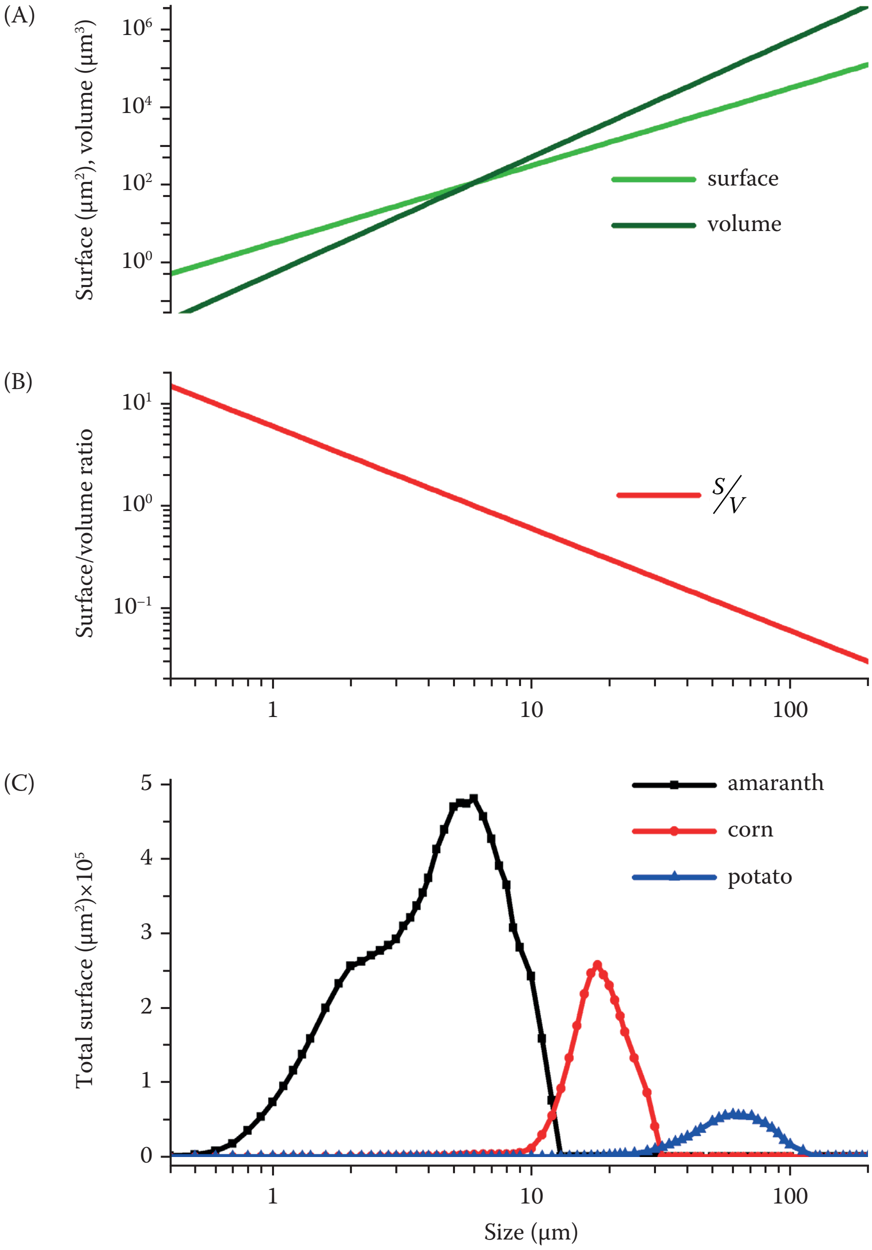

Figure 5. Surface and volume behaviour as a function of the size of a sphere – (A) surface and volume, (B) surface/volume ratio, (C) approximate total surface for the same amount (weight) of amaranth, corn, and potato starches, considering granules as spheres

burg 2011). Mathematically, the volume increases more rapidly compared with its surface, as demonstrated by the change in the surface/volume ratio vs size shown in Figure 5B. This means that bigger starch granules had larger volume and smaller surface than the small ones to keep in contact with water or other granules. Figure 5C shows the total surface calculated for AS, CS, and PS based on the granule size distribution of each type of starch (Figure 1). All total surface area and volume calculations were performed considering the same mass amount of each starch.

The total surface growth as a function of the temperature of the starch/water solution at different proportions can also be useful to explain the rheological behaviour. To compare the granule size of the three starch granules, a quantity called integrated total surface was calculated by integrating the total surface distribution (as shown in Figure 5C) based on the granulometry measurements. A high number of small granules in AS resulted in the highest total surface; meanwhile, a similar mass amount of big granules in PS resulted in the lowest total surface.

A higher increase in the total surface was observed for AS and PS when compared to CS. For a numerical comparison, ratios between total surface values (when the trace becomes horizontal) between 80 °C and room temperature (native starches) were calculated. The calculated ratio was 32.0, 30.3 and 7.8 for AS, PS and CS, respectively. Also, a similar ratio of maximum granule size was calculated between room temperature and 80 °C for the three starches (Figure 6). The calculated ratio for the maximum granule size was 5.8, 4.5, and 2.2 for AS, PS, and CS, respectively, confirming the more limited growth for CS as the contribution of the multimodal population affected the swelling capacity of granules in the matrix. Most probably, the small granules filled the spaces between the big granules, increasing the number of contact points. The interactions between small and big granules promoted a denser structure affecting the rheological properties because the swelling of the granules was more limited.

To further study the behaviour observed in CS, a theoretical analysis of the integrated surface was performed as a function of temperature. The integrated surface was calculated from the particle size distribution results of starch suspensions measured after thermal treatment in the range from room temperature to 90 °C (Figure 7). Before reaching the gelatinisation temperature, practically no change in size was observed; meanwhile, above the gelatinisation temperature, a sudden change in size occurred, although it was limited quickly, with no substantial change between 70 and 90 °C. This behaviour suggested a strong interaction between adjacent granules, which hindered the granule swelling.

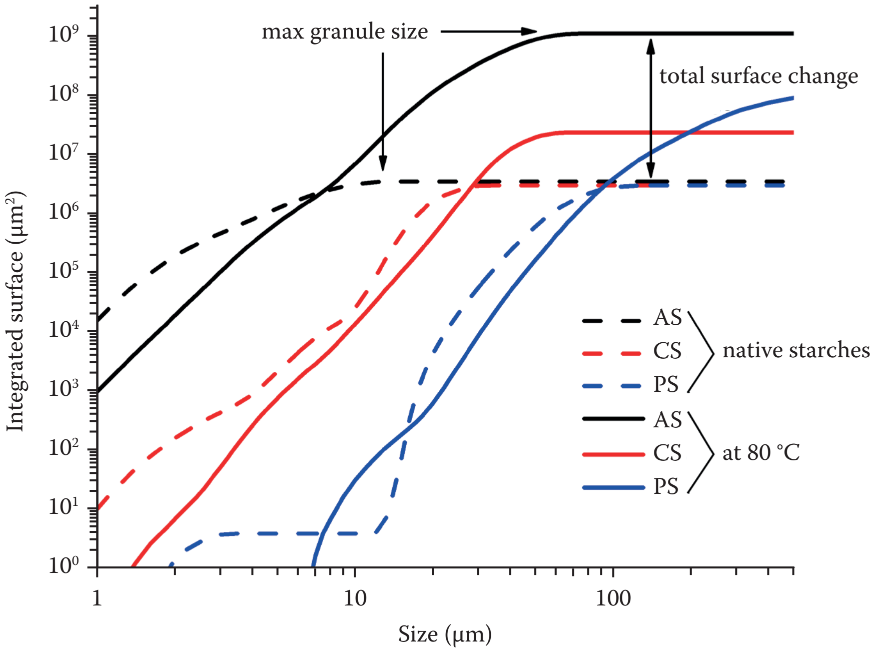

Figure 6. Comparison of the integrated surface as a function of the size between native and heated to 80 °C

AS – amaranth starch; CS – corn starch; PS – potato starch

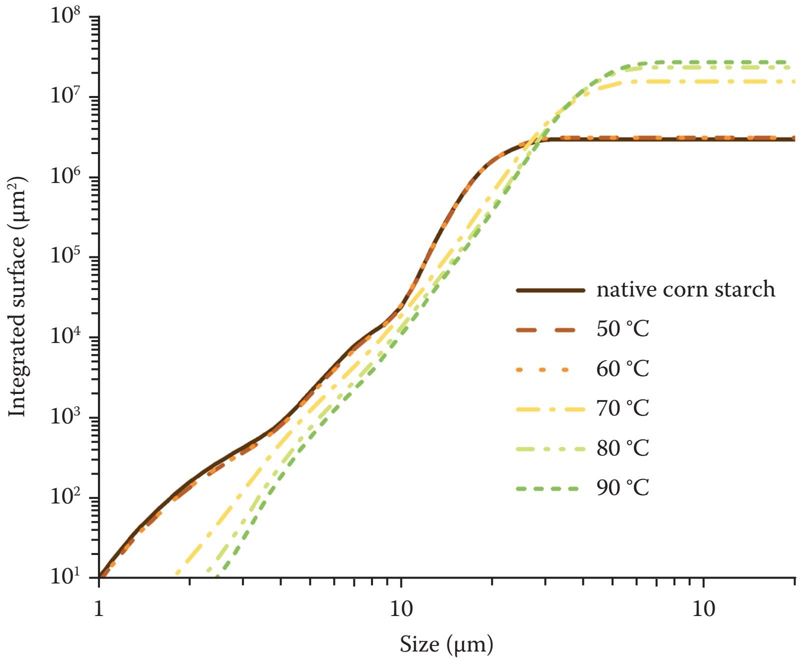

Figure 7. Evolution of the integrated surface as a function of the size for several temperatures from native corn starch to 90 °C at 10% of solids

CONCLUSION

Solids concentration and granule size distribution resulted in a different surface/volume granule ratio affecting the rheological properties of the starch dispersions. The results suggest that the properties of swollen granules drove the rheological behaviour during pasting profile and gel conformation.

CS had the lowest values of the flow behaviour index and the highest consistency index. This behaviour was related to the granule size distribution, where the small granules formed a more complex system with the big granules. The surface/volume granule ratio changed when the native starches were heated at 80 °C, and CS reached the lowest value (7.8) compared to 30.3 and 32.0 reached by PS and AS, respectively.

The gels obtained from all starches showed an essentially elastic response typical of authentic gels. For gel conformation, CS showed a high moduli dependence on the frequency, reflecting a more rigid system and a denser entanglement. This effect was related to the change in granule sizes which occurred at higher temperatures reducing the granule packing as reflected by the ratio for the maximum granule size reaching a value of 2.2 compared to 4.5 and 5.8 shown by AS and PS, respectively. This information would be useful in estimating the application of starch granules of specific sizes, size distributions, and swelling conditions as ingredients to offer different rheological characteristics in food systems.

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