Study of DIC hydrothermal treatment effect on rheological properties of standard maize (SMS), waxy maize (WMS), wheat (WTS) and potato (PTS) starches

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STUDY OF DIC HYDROTHERMAL TREATMENT EFFECT ON RHEOLOGICAL PROPERTIES OF STANDARD MAIZE (SMS), WAXY MAIZE (WMS), WHEAT (WTS) AND POTATO (PTS) STARCHES

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1. Abstract

Standard maize (SMS), waxy maize (WMS), wheat (WTS) and potato (PTS) starches were hydrothermally treated by Instantaneous Controlled Pressure Drop (DIC) process at different pressure levels (1, 2 and 3 bar) corresponding to the temperatures of 100, 122 and 136°C, respectively. The rheological properties and particle size of treated starches under various conditions were compared to the native ones. The results showed for all starches, except for WTS, a reduction of the consistency coefficient and the yield stress (τ_o) with increased intensity of the hydrothermal treatment conditions. Furthermore, τ_o vanished for severe treatment conditions. The DIC treatment yielded an increased fluidity and a loss of the conservative modulus of the pastes, as a result of partial gelatinization of starch granules. The extent of the observed effect depended on the botanical origin. Wheat starch exhibited a different behaviour: the consistency coefficient and the conservative modulus being higher for DIC treated starch except for the most severe conditions.

2. Introduction

Starch has many applications in food and non-food industries. As an ingredient, it is extracted from only a few species such as maize, wheat, potato, rice, tapioca, and sago. Pregelatinized starches have been widely used for many foods as a major ingredient to provide thickened textures at temperatures below the gelatinization temperature. They are obtained from native or modified starch, by drum drying (Vallous et al., 2002) or by extrusion cooking (Barron et al., 2000). Other processes of physical modification have been explored to improve qualities of the starch such as annealing (Tester et al., 2000; Jayakody and Hoover, 2008) and heat moisture treatment (HMT) (Kulp and Lorenz, 1981; Hoover and Manuel, 1996; Jacobs, et al., 1998; Collado and Corke, 1999; Tester et al., 2000; Gunaratne and Hoover, 2002; Vermeylen et al., 2006; Tukomane et al., 2007; Gunaratne and Corke, 2007, Chung et al., 2009). These two latter treatments differ in the water content, temperature and processing time used. Annealing occurs under large excess of water (50 to 60%) and relatively low temperatures (below the gelatinization temperature), while the HMT is conducted under restricted moisture content (10-30%) and higher temperatures (90-120 °C). Both treatments are applied over large periods of time (10-16h). The main effects of HMT are loss of birefringence, increased gelatinization temperature, broadened or unchanged gelatinization temperature range, change in X-ray diffraction patterns, reduced swelling volume and solubility, with consequent changes in functionality (Donovan et al., 1983; Collado and Corke, 1999; Gunaratne and Hoover, 2002). Annealing results in improved perfection of the crystallites within starch granules that narrows the gelatinization temperature interval; consequently, gelatinization temperatures shifted towards higher values (Hublin, 1994; Jacobs et al., 1998; Tester et al., 2000). The enthalpy of gelatinization remains unchanged or is moderately increased depending upon annealing conditions (moisture content and time) and the botanic origin (Karlsson and Eliasson, 2003; Lawal, 2005; Jayakody and Hoover, 2008) in contrast to HMT. The semi-crystalline structure

of starch granules is modified by these two usual physical treatments without disrupting the integrity of granule (Lim et al., 2001).

HMT starches have generally been performed at the laboratory scale and many authors have reported that such conditions produce inhomogeneous samples with lumps of gelatinized starch beside heat-moisture treated starch. For this reason, pressure is often required to ensure sufficient heating. To obtain a uniform heat distribution and rapid penetration of steam into the starch granules, Maruta et al. (1994) improved the conventional method by creating a reduced pressure in the vessel before the injection of live steam. This method was designated by these authors as the reduced-pressurized heat moisture treatment (RP-HMT).

The DIC treatment (Instantaneous Controlled Pressure Drop) has been developed at the laboratory as well as the pilot scale (Rezzoug et al., 2000), for drying and texturizing of food products such as pasta products (Maache-Rezzoug and Allaf, 2005). As for the RP-HMT process, an initial vacuum is applied before the treatment which is performed under high temperature/high steam pressure; the hydro-treatment step is then followed by an abrupt pressure drop towards vacuum pressure contrary to the RP-HMT treatment. This step induces a rapid modification of the thermodynamic equilibrium reached during the pressurisation (P_1,T_1) towards another equilibrium state (P_2,T_2). This new state induces a rapid cooling and the resulting temperature value depends on the vacuum pressure level (Zarguilli et al., 2009). The originality of the DIC process compared to other hydrothermal treatments is that starch is treated at an initial moisture content of 12.5% (wet basis), no hydration step being then used. During the treatment, the heating of starch is obtained by the absorption of latent heat of steam condensation which causes an increase in the moisture content as the processing time and pressure level increase (Zarguili et al., 2009).

The major effects observed after DIC treatment are almost similar to HMT treatment except that the gelatinization temperature range is narrowed as observed with annealing (Hublin, 1994;

Jayakody and Hoover, 2008). This result suggests that the treatment firstly induced the melting of crystallites of low cohesion (low stability) which required less energy to melt. Consequently, the residual structure after the DIC treatment contained crystallites with a greater stability (cohesion) (Maache-Rezzoug et al., 2008). Preliminary studies on standard and waxy maize starches (Loisel et al., 2006, Zarguili et al., 2006) showed that the thermal properties of DIC treated starch depend on the processing time and the steam pressure level. Increasing these two parameters induces an increase in the onset (T_{onset}) and in the peak ($T_{peak,}$) temperatures of gelatinization and a reduction in gelatinization enthalpy. The occurrence of a partial or total gelatinization was clearly attested by the decrease of enthalpy and a loss of birefringence under polarized light. The X-ray diffraction pattern confirmed the partial or total loss of the crystalline structure of native starch depending on the conditions of the DIC treatment: the relative crystallinity of hydrothermally treated maize starch decreased and the polymorphic type changed. The A-type crystalline pattern was progressively lost with the increase of processing pressure ($\geq$ 2 bar), and was substituted by the V_h-type X-ray diffraction pattern, corresponding to the formation of amylose-lipid complexes. At severe DIC conditions (pressure level of 3 bar), the typical peaks of A-type X-ray diffraction pattern were substituted completely by the ones of the V_h-type pattern (Maache-Rezzoug et al., 2008).

The objective of the present study was to describe the rheological properties of hydrothermally treated starches in the DIC process in relation to starch granules properties (size and size distribution, swelling behaviour). This study was based on rheological measurements (viscosity and viscoelasticity). These effects were investigated on starches of different origins: standard maize (SMS), waxy maize (WMS), wheat (WTS) and potato (PTS); identical treatments conditions (processing pressure and time) were applied, except for PTS. Lower pressure/time conditions were applied to potato starch due to its higher sensitivity to hydrothermal treatment.

3. Materials and methods

3.1. Materials

Standard maize starch (SMS), waxy maize starch (WMS, Waxilys 200), wheat starch (WTS) and potato starch (PTS) were supplied by Roquette Frères (Lestrem, France). The moisture content of these starches during the treatment was about 12% wet basis.

3.2. Methods

3.2.1. Moisture content

The starch moisture content was determined by air oven at 105 °C during 24 h, according to the A.F.N.O.R (NF V03-707, 2000) standard method and related to the wet basis (%, wb).

3.2.2. DIC hydrothermal treatment

The equipment and procedure of DIC hydrothermal treatment were largely described in previous studies (Zarguili et al., 2006). During the treatment, 22 g of starch (12.5%, wet basis) disposed in circular containers were placed in the processing vessel (12L) in a layer of 20 cm diameter and 0.5 cm height. An initial vacuum of 50 mbar was established. As demonstrated by Zarguili (2006), this initial vacuum allows the air resistance to be reduced and thus facilitates the diffusion of steam into the product, consequently a rapid heating is obtained. Saturated steam is introduced into the vessel at a fixed pressure level (1 to 3 bars) and maintained during a determined processing time. In this study the processing pressure was fixed at 1 bar (100 °C), 2 bar (122 °C) and 3 bar (135 °C). The pressurisation is followed by an abrupt decompression towards vacuum (50 mbar). After the vacuum phase, atmospheric air is injected to return to atmospheric pressure for sample recovery. During the treatment, starch is heated by the absorption of latent heat of vapour condensation and its moisture content is increased.

3.2.3. Pasting procedure using the Brabender Viscograph

The DIC treated starches were pasted with demineralised water using a Brabender Viscograph in order to obtain starch pastes under repeatable conditions. The starch concentrations were chosen to lie within the sensitivity range of the Viscograph, depending on the botanical origin of starches. The concentrations used were 6%, 4%, 7% and 2% for SMS, WMS, WTS and PTS, respectively. The suspension was heated at 1.5 °C/min from 50 to 95 °C, then kept for 20 min at the plateau temperature and subsequently cooled down to 70 °C at 1.5 °C/min before immediate characterisation. The starch concentration was checked by drying the suspensions as previously described (3.2.1.).

3.2.4. Granule size distribution

Granule size determination was carried out at room temperature using a Malvern Master Sizer (Malvern Instruments, Ltd) laser scattering analyser with a 300 mm Fourier cell (range 0.05-879 µm). The starch dispersion was first diluted (1/10) with demineralised water at 20°C before and immediately after the pasting procedure in the Brabender Viscograph, and then dispersed into the sample dispersion unit (1ml/100ml water). The measure was repeated three times. The volume distribution was obtained according to the Mie scattering theory (Loisel et al., 2006). From each distribution, the median volume diameter ($D_{v,0.5}$) was presented and the swelling ratio was defined as $(D/D_0)^3$, with D and D_0 the median diameters of treated and native starch, respectively (Nayouf et al., 2003); the size distribution was evaluated using the dispersion index referred to as the span, by the following equation:

$$Span = \frac{D(v,0.9) - D(v,0.1)}{D(v,0.5)} \qquad \text{(Eq.1)}$$

3.2.5. Rheological properties

Flow behaviour and viscoelastic properties of starch pastes were measured at 60 °C (to avoid retrogradation) using a controlled stress rheometer (TA Instrument AR1000) with the cone/plate geometry (6 cm/2°). The starch dispersions at 60 °C were poured onto the preheated plate of the rheometer, and then covered by a thin layer of paraffin oil on the edge of the sample to avoid evaporation. For flow measurements, an up-down shear scan from 0 to 660 s^{-1}(4 min) was linearly applied, followed by a logarithmic stepwise decrease from 660 to 0.01 s^{-1}, after equilibrium for each shear rate. The oscillatory tests at 60 °C were carried out on a new aliquot at 4% strain (linear viscoelastic range). The frequency range investigated was from 0.5 to 100 rad/s.

4. Results and discussion

4.1. Granule size distribution

The size distribution of starch granules was carried out before (Figure 1a) and after pasting (Figure 1b) in the Brabender Viscograph. Table 1 presents the variation of the median diameter $D_{V,0.5}$, the swelling ratio $(D/D_0)^3$ and the span, which measures the volume distribution width of starch granules, for SMS, WMS, WTS and PTS.

Before pasting the native starches exhibit a narrow size distribution of the granules, the median diameter lying between 12.9 for SMS, the smaller starch granules, to 44.6 µm for PTS the larger ones with also a minor peak at approximately one micron. These values correspond to the usual starch granule size distribution. For DIC treated starches, the size distribution curves before pasting were shifted towards higher sizes and broadened as increasing DIC conditions, with large differences depending on botanical origin: SMS being more prone to swell than WMS and WTS showing a dramatic increase at 3bar/5min. For PTS only slight modifications

were observed due to the low intensity of the DIC conditions. A progressive increase of the median diameter with the intensity of the treatment is attested for all starches. The span values increased for SMS, WMS and WTS, after DIC treatment reflecting a widening of the granule size distribution. This was particularly important for SMS (Table 1) and is probably due to the presence of a third peak at approximately 200 microns (Figure 1) corresponding to aggregates of starch granules. The different evolution of the swelling ratio $(D/D_0)^3$ clearly underlines the differences between starches: SMS and WTS present the highest swelling capacities at 3bar/5min of 105.7 and 327.6, respectively. Such a behaviour, particularly for WTS, reflects the cold swelling of DIC treated starch granules; it is not mentioned in the literature for HMT treated starches and has been ascribed to partial loss of cristallinity and subsequent enhanced capacity to hydration (Loisel et al., 2006).

After pasting, the size distribution curves (Figure 1) of all the native starches exhibit a shift towards larger sizes as expected. The 1bar/60min treated starches, SMS, WMS and WTS, present the same distribution curve as the native ones with smoothing of the minor peak for WMS. But the 2bar/60min and 3bar/5min treated SMS and WMS show the reverse tendency: a shift towards a lower starch granules size. For SMS and WMS the minor peak at one micron was converted into a larger one, at approximately 5 µm, which disappeared for the most severe conditions for WMS. This peak may be attributed to a population of small starch granules that swell upon pasting or to the disruption of larger ones. The median diameters $D_{V,0.5}$ (Table 1) increase for native starch after pasting as expected (from 12.9 to 41.1 µm, 14.3 to 32.5 µm, 19.8 to 36.6 µm and 44.6 to 112.3 µm) for SMS, WMS, WTS and PTS, respectively. The DIC treated SMS and WMS starches present similar sizes to the native ones for the lowest DIC conditions (1bar/60min). For medium and high DIC conditions a progressive decrease is observed: the swelling ratio reaches 0.4 for SMS and WMS at 3bar/5min. This modification can be ascribed to concomitant processes of swelling and disruption of starch granules

encountered during the pasting process. For DIC treated SMS and WMS the disruption phenomenon may prevail owing to the sensitivity of starch granules induced by the treatment. WTS exhibited an outstanding behaviour, by increasing the size of starch granules for the most severe condition (swelling ratio 1.93 for 3bar/5min). These modifications of the starch granules size distribution by DIC treatment will obviously affect the rheological properties.

4.2. Rheological properties

Figure 2 presents the rheograms of native and DIC treated starches of SMS, WMS, WTS and PTS. The flow curves are typical of shear-thinning fluids, except the ones of samples treated at 2bar/60min and 3bar/5min for SMS, 3bar/5min for WTS and at 1.5bar/5min for PTS. We also observe the persistence of the thixotropic behaviour of the starches, which may result from a disruption of starch granules aggregates under shearing. This phenomenon appeared less pronounced when the treatment conditions increased and disappeared for samples treated at 3bar/5min for SMS and 1.5bar/5min for PTS.

The Herschel-Bulkley equation (Eq.2) was applied satisfactorily from the equilibrium curves (not presented in Figure 2) according to Eq. (2):

$$\tau = \tau_0 + k\,\dot{\gamma}^n \qquad \text{(Eq.2)}$$

τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), τ_0 is the yield stress (Pa), K is the consistency index ($Pa.s^n$) and n is the flow behaviour index (dimensionless).

The values of the flow parameters (τ_0, K, n) and the apparent viscosity for $1s^{-1}$ are given in Table 2. We observe shear-thinning behaviour (n<1) with a yield stress for all the native and treated starches except at 2bar/60min and 3bar/5min for SMS, 3bar/5min for WTS, and 1.5bar/5min for PTS. For these DIC conditions, the rheological behaviour of treated starches tended towards a Newtonian one with increasing flow behaviour index.

For WMS and WTS the flow behaviour index remains almost constant for all the process conditions. For all starches the increase of the process conditions causes a decrease of the yield stress and of the apparent viscosity except for WTS.

In Table 2 are presented the values of the storage modulus (G'), and the loss modulus (G'') measured at 60 °C. The suspensions of native starches exhibited the behaviour of a weak gel with G'>G'' and G' almost independent of frequency (mechanical spectra, not shown).

The storage modulus G' sharply decreased for SMS as the process conditions became more intense, while the decrease was more progressive for WMS. This effect of the DIC process on the viscoelasticity of SMS and WMS starch suspensions agrees with previous results (Loisel et al., 2006). WTS is the only starch which behaved in a different way, exhibiting an increase of the storage modulus with the processing conditions.

Considering the effect of the treatment on the global properties of the starch suspensions, we can observe the same evolution between the rheological parameters (yield stress, viscosity, storage modulus, Table 2) and the size of the starch granules after pasting (Table 1): a tendency to a decrease for SMS, WMS and PTS and to an increase for WTS with the intensity of the treatment. But no direct relationship can be drawn between these two characteristics as the volume fraction of starch granules is not taken into account. It is well known that the rheological behaviour of a pasted starch suspension is the result of two main characteristics: the viscosity of the continuous phase and the volume fraction of the dispersed phase constituted by the starch granules (Doublier et al., 1987). The concentrations of the starch suspensions used in that study are very close to the packing concentration described by Steeneken (1989): i.e. the suspension can be assimilated to a packing of swollen starch granules that are responsible for the overall rheological properties of the suspensions, the contribution of the continuous phase being minored. In such a concentrated system, the storage modulus indirectly measures the rigidity of the swollen granules. The decrease of the storage modulus for SMS and WMS

resulting from the intensity of the treatment may reflect either a loss of rigidity of starch granules; or more probably a decrease of their volume fraction.

The DIC process presents some similarities with the HMT treatment as it is conducted at low moisture content between 15 and 20% for DIC (depending on the pressure) compared to the 30% usually used for HMT. All the authors state a decrease of the swelling factor (generally expressed as the volume of swollen granules to the volume of the dry starch) and of the Brabender viscosity after pasting and for SMS (Chung et al., 2009) and in a lesser extent for WMS (Hoover and Manuel, 1996; Gunaratne and Corke, 2007). PTS presents the greatest sensitivity to the HMT treatment according to these two characteristics (Gunaratne and Hoover, 2002) while WTS exhibits a decrease of the swelling factor but a minor viscosity loss (Gunaratne and Corke, 2007) or even a slight increase of the viscosity after pasting (Hoover and Vasanthan, 1994). The effect of the DIC treatment on the rheological properties of starch suspensions seems similar to a certain extent to the one of HMT treatment.

5. Conclusion

This study has shown that the rheological and morphological properties of starches from different botanical origins, standard maize (SMS), waxy maize (WMS), wheat (WTS) and potato (PTS), are influenced to a different extent by the processing conditions applied during the DIC treatment. The PTS is more sensitive to the treatment than other starches, whereas WMS and WTS are more resistant. When the processing treatments are intense the size distributions of the starch granules and the rheological properties of starch suspensions vary. We observed a reduction in swelling for SMS and WMS starch granules after pasting which was ascribed to their prevailing disruption. For WTS the reverse result was observed. The rheological behaviour tends to Newtonian behaviour (n=1), except for WMS, with a decrease of the yield stress and the apparent viscosity as the processing conditions increase. The

252 rheological properties are related to the evolution of the size of starch granules with the DIC
253 treatment.

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Figures Captions

Figure 1: Size distribution of native and DIC treated SMS, WMS, WTS, and PTS starches: (a) before pasting and (b) after pasting.

Figure 2: Flow curves of native and DIC treated starch dispersions measured at 60 °C after pasting, with starch concentration of 6%, 4%, 7% and 2% for SMS, WMS, WTS and PTS, respectively.

342 Figure 1

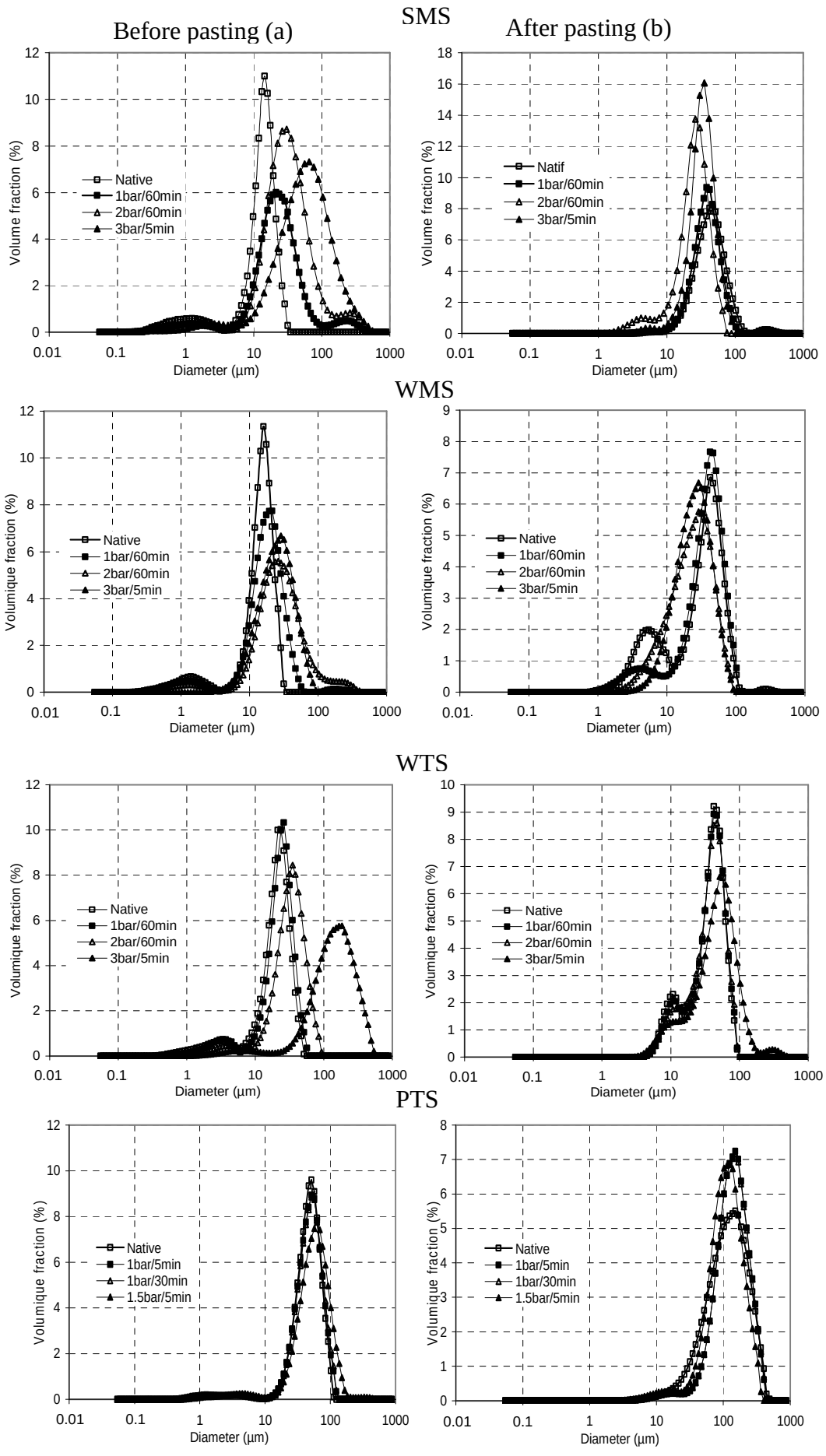
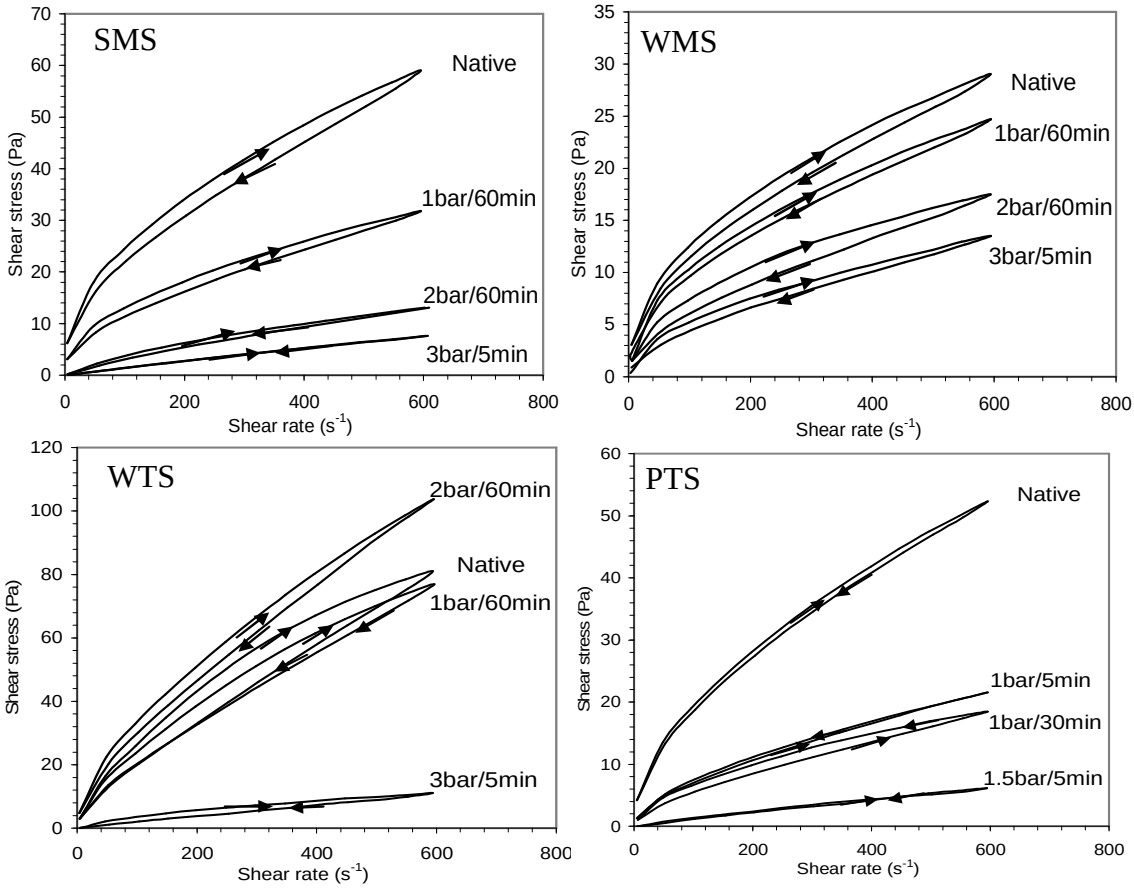


Figure 2



344 **Table 1**: Particle size characteristics of SMS, WMS, WTS and PTS after DIC treatment.

345 (standard deviation of $Dv,_{0.5}$ =0.3 µm)

346

	DIC conditions	Granulometry					
		Before pasting			After pasting		
Starch source	Pressure level/ processing time (bar/min)	$D_{V,0.5}$ (µm)	$(D/D_o)^3$	Span	$D_{V,0.5}$ (µm)	$(D/D_o)^3$	Span
SMS	native	12.9	1.0	1.4	41.1	1.0	1.3
	1/60	19.3	3.3	1.5	39.3	0.9	1.2
	2/60	27.7	9.9	2.4	23.8	0.2	1.3
	3/5	61.0	105.7	2.5	29.6	0.4	1.8
WMS	native	14.3	1.0	1.1	32.5	1.0	1.8
	1/60	17.1	1.2	1.4	36.1	1.4	1.7
	2/60	25.3	5.5	2.4	22.5	0.3	1.8
	3/5	39.5	21.1	1.8	23.7	0.4	1.5
WTS	native	19.8	1.0	1.2	36.6	1.0	1.3
	1/60	22.2	1.4	1.2	36.8	1.0	1.3
	2/60	30.5	3.6	1.4	36.7	1.0	1.4
	3/5	136.5	327.6	1.8	45.6	1.9	1.8
PTS	native	44.6	1.0	1.2	112.3	1.0	1.9
	1/5	45.8	1.1	1.2	114.8	1.1	1.5
	1/30	46.4	1.1	1.2	114.4	1.1	1.5
	1.5/5	54.1	1.8	1.4	104.9	0.8	1.5

347 **Table 2:** Herschel-Bulkley parameters and viscoelastic properties of SMS, WMS, WTS and

348 PTS pasted suspensions after DIC treatment.

	DIC conditions	Flow behaviour 60°C				Viscoelasticity (60°C ;6.3 rad/s)		
Starch source	Pressure level / processing time (bar / min)	τ_o (Pa)	K ($Pa.s^n$)	n	η_a (Pa.s)	G' (Pa)	G" (Pa)	Tan(δ)
SMS[a]	native	2.28	1.39	0.57	3.68	126.7	16.6	0.13
	1/60	0.68	0.72	0.58	1.41	115.4	13.4	0.12
	2/60	0.00	0.04	0.91	0.04	0.43	0.16	0.37
	3/5	0.00	0.01	1.00	0.01	3.12	0.91	0.29
WMS[b]	native	0.70	0.88	0.53	1.58	1.96	1.06	0.54
	1/60	0.71	0.81	0.52	1.52	1.69	0.93	0.55
	2/60	0.31	0.26	0.62	0.57	1.12	0.74	0.66
	3/5	0.36	0.58	0.54	0.95	1.20	0.90	0.75
WTS[c]	native	0.95	0.44	0.78	1.39	21.7	11.2	0.52
	1/60	0.87	0.75	0.70	1.63	30.1	12.8	0.45
	2/60	1.00	0.87	0.72	1.87	26.9	10.5	0.39
	3/5	0.00	0.03	0.88	0.03	63.5	12.4	0.19
PTS[d]	native	0.77	1.28	0.57	2.05	9.70	6.4	0.66
	1/5	0.06	0.47	0.60	0.53	3.90	2.5	0.64
	1/30	0.02	0.15	0.71	0.17	4.10	2.7	0.66
	1.5/5	0.00	0.02	0.89	0.02	0.34	0.04	0.11

349 [a] 6% (w/w) SMS suspension; [b] 4% (w/w) WMS suspension; [c] 7% (w/w) WTS suspension and [d] 2% (w/w)
350 PTS suspension; τ_0 :yield stress ; K : consistency index; n : flow behaviour index (τ_0, K and n were
351 determined from Herschel-Bulkley model); η_a:apparent viscosity measured at $1s^{-1}$.