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Starch pastes thinning during high-pressure homogenization

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ABSTRACT

High-pressure homogenization induced thinning of potato and cassava starch paste was investigated. The starch slurries at a concentration of 2.0 wt.% were heated at 90 °C for 1 h and then rapidly cooled in tap water. The cooled starch pastes were homogenized at various pressures ranging from 0 to 100 MPa using a lab-scale high-pressure homogenizer. The influence of homogenizing pressure on the temperature, apparent viscosity, electrical conductivity, and percent light transmittance of homogenized starch pastes were determined. Temperatures of homogenized starch pastes increased linearly with the increase of the applied pressure, and the rate was 0.177 °C/MPa and 0.186 °C/MPa for potato and cassava starch pastes, respectively. After high-pressure homogenization, the apparent viscosities of the starch pastes decreased, while the percent light transmittances of them increased. However, the electrical conductivities of starch pastes were not affected by homogenization.

1. Introduction

As one of the most abundant naturally occurring substances, starch is widely used in paper, textile, adhesive, and food industries (Jyothi, Sasikiran, Sajeev, Revamma, & Moorthy, 2005; Morikawa & Nishinari, 2000; Pareta & Edirisinghe, 2006; Roberts & Cameron, 2002). Native starch is known to be insoluble in cold water at atmospheric pressure (Nayouf, Loisel, & Doublier, 2003). When heated in the presence of excess water, above gelatinization temperature, starch granules lose their crystallinity, absorb large amounts of water, swell to larger sizes, and leach out amylose, which impart considerable viscosity to the starch/water system (Che, Li, Wang, Chen, & Mao, 2007a; Che et al., 2007b; Evans & Haisman, 1979; Kim, Lee, & Yoo, 2006). The magnitude of starch paste viscosity is very important since starch is generally used in the form of starch paste. Being a large polymer, starch often develops high paste viscosity which can cause practical problems in its application and further processing. For instance, starch pastes at high concentrations are hardly used to prepare pre-gelatinized starch by spray drying since they are difficult to be transported through pipes and atomized due to their high viscosities. Therefore, the development of an efficient process to lower starch paste viscosity without reducing starch concentration is of great interest. High-pressure homogenization is a promising alternative for reducing the viscosity of starch pastes.

High-pressure homogenization, where high pressure is experienced over very short time, is different from static high-pressure system, because pressure induced phenomena of cavitation, shear, and turbulence are involved simultaneously (Paquin, 1999). Cavitation is the formation of cavities filled with gas or vapor as pressure decreases, and their collapse as soon as the pressure increases again (Freudig, Tesch, & Schubert, 2003). The sudden collapse of cavitation bubbles induces high pressure gradients and high local velocities of liquid layers in their vicinity. These in turn cause shear forces that are capable of breaking the chains of polymers by disrupting covalent bonds, provided the chains are longer than a critical value. This is the mechanochemical action of homogenization on polymers. Water is partially decomposed into OH radicals and H atoms in the collapsing cavitation bubbles. Some of these radicals diffuse out of the cavities to the surrounding liquid and then react with solute molecules causing polymer degradation. This mechanism is called radical attack. Moreover, the pressure changes so fast that the implosive collapse of a bubble is an adiabatic process that leads to a rapid, momentary temperature increase up to 5000 K in the gas phase of the collapsing bubble and above 1000 K in the thin layer of liquid adjacent to the cavity. Polymers in the hot interfacial region between the bubble and the surrounding liquid may also be pyrolyzed (Czechowska-Biskup, Rokita, Lotfy, Ulanski, & Rosiak, 2005).

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Nomenclature

A	constant, mPa s
C	first-order reaction rate constant, MPa s
K	consistency index, mPa s^n
K_∞	the limiting value of consistency index, mPa s
n	flow behavior index (dimensionless)
P	homogenizing pressure, MPa
R^2	coefficient of determination
t	processing time, s
$\dot{\gamma}$	shear rate, s^{-1}
η_{app}	apparent viscosity, mPa s
τ	shear stress, mPa

With the specific energy output, high-pressure homogenization was reported to introduce novel changes to the treated samples. The degradation or disruption of polymers caused by high-pressure homogenization was studied by several researchers (Modig, Nilsson, Bergenståhl, & Wahlund, 2006). Lagoueyte and Paquin (1998) demonstrated that the high shear, turbulence forces, and cavitation involved in microfluidization process produced degradation of xanthan, as a result, the thickening and stabilizing properties of xanthan were reduced. The extent of reduction increased with the increase of applied pressure and number of passes. Floury, Desrumaux, Axelos, and Legrand (2002) investigated the degradation of methylcellulose during high-pressure homogenization. They found that the molecular weight of methylcellulose was reduced by homogenization at pressures up to 350 MPa resulting in marked decrease in apparent viscosity. Kasaai, Charlet, Paquin, and Arul (2003) reported that microfluidization was useful for partial depolymerization of large food polymers in order to reduce their solution viscosities. Modig et al. (2006) studied the degradation of octenyl succinate anhydride starch resulted from high-pressure homogenization using flow field-flow fractionation and multi-angle light scattering. The results showed that high-pressure homogenization disrupted the modified starch and the extent of disruption depended on the energy dissipation rate during processing.

As a frequently used emulsification process, high-pressure homogenization was reported to reduce the apparent viscosities of sunflower oil-in-water emulsions by Floury, Desrumaux, and Lardières (2000). They showed that the apparent viscosities of the emulsions decreased with the increasing of homogenizing pressure until a limiting value was achieved, and the emulsions changed from shear-thinning to Newtonian fluids as the pressure was increased from 20 to 300 MPa. More recently, Floury, Desrumaux, Axelos, and Legrand (2003) evaluated the effect of homogenizing pressure on emulsifying and stabilizing properties of methylcellulose, which is extensively used as stabilizer in emulsions. They found that homogenized methylcellulose solutions showed weaker thickening and stabilizing properties compared to the one of native methylcellulose.

Starch is often high-pressure homogenized with other food components to make more stable and homogeneous products. The intense mechanical treatment is expected to strongly influence the structure and properties of starch and then the quality of final products. The aim of this work was to investigate the high-pressure homogenization induced thinning of starch pastes. In order to find out the influences of homogenization pressure, the temperatures, apparent viscosities, electrical conductivities, and percent light transmittances of homogenized starch pastes were measured as a function of the homogenizing pressure.

2. Materials and methods

2.1. Materials

Potato and cassava starches were purchased from Beijing Aoboxing Bio-Tech Co., Ltd. and Beijing Quanfeng Starch Company, respectively. Potato and cassava starches were chosen because of their high paste viscosities.

2.2. Preparation of starch pastes

Starch slurry at a concentration of 2.0 wt.% was prepared by adding required amount of air-oven dried potato or cassava starch into deionized water (electrical conductivity 2.1×10^{-4} S/m) at room temperature (about 25 °C). Well mixed slurries in beakers were heated in water bath at 90 °C for 1 h to gelatinize the starch and then cooled in tap water for 1 h. The beakers were covered with preservative films to minimize moisture loss during heating and cooling.

2.3. High-pressure homogenization of starch pastes

The pre-gelatinized and cooled starch pastes were homogenized using a lab-scale high-pressure homogenizer (NS1001L-PANDA 2K, Niro Soavi S.p.A., Parma, Italy) at 0, 20, 40, 60, 80, and 100 MPa for one pass. Approximately 100 mL starch paste was obtained at each pressure level. NS1001L-PANDA 2K was a two-stage homogenizer, and only the first high-pressure valve was used (Che et al., 2007b). The temperatures of starch pastes were monitored just before and immediately after homogenization with an immersion thermometer. After the temperature measurements, the homogenized starch pastes were cooled to room temperature for further characterizations.

2.4. Microscopy of homogenized starch pastes

The starch pastes were observed under optical microscope (CX31 Biological Microscope, Olympus Corporation, Japan) in dark field. The microscope was equipped with a CCD camera module to take pictures.

2.5. Rheological properties of homogenized starch pastes

Steady shear experiments were carried out using a stress-controlled rheometer (AR2000ex, TA Instruments Ltd., Crawley, UK) with a concentric cylinder geometry at 25 °C. For each test, 19.6 mL freshly prepared starch paste was used. After loading into the concentric cylinder geometry, the sample was allowed to equilibrate for 2 min prior to the experiment. The shear stress was then measured as a function of shear rate which was increased from 1 to 100 s^{-1} logarithmically. Ten points were collected per decade. The viscosities of non-homogenized starch pastes (NH) were also measured as control samples. The power law model ($\tau = K\dot{\gamma}^n$) was used to describe the flow behavior (shear stress against shear rate) of starch pastes. Where τ is shear stress (MPa), $\dot{\gamma}$ is shear rate (s^{-1}), K (which corresponds to viscosity when the fluid is Newtonian) is consistency index (MPa s^n), and n is flow behavior index (Floury et al., 2000).

2.6. Measurements of electrical conductivities of homogenized starch pastes

The electrical conductivities of homogenized starch pastes were measured using a conductivity meter (DDS-320, Shanghai Kangyi Co., Ltd., Shanghai, China) at 25 °C.

2.7. Determination of starch paste clarity

Starch paste clarity was investigated by measuring the percent light transmittances (%) of starch pastes. The homogenized starch pastes were stored at room temperature for 2 h, subsequently the percent light transmittances of these homogenized starch pastes were determined at 620 nm (1 cm path length) against a water blank using an ultraviolet visible spectrophotometer (TU-1810, Beijing Purkinje General Instrument Co., Ltd., Beijing, China).

2.8. Statistical analysis

All the experiments were carried out in triplicate. The results reported are means ± S. D. of three determinations excluding the flow curves.

3. Results and discussion

3.1. Effect of high-pressure homogenization on the temperatures of starch pastes

The temperatures of homogenized starch pastes are shown in Fig. 1. As can be seen from Fig. 1, linear relationships with high coefficients of determination ($R^2 > 0.99$) are suitable for describing the relationship between the homogenizing pressure and the temperatures of homogenized starch pastes. The temperatures of homogenized starch pastes increased linearly with the increasing of homogenizing pressure. The slopes of the lines indicating the dependence of temperature on the homogenizing pressure were 0.177 °C/MPa and 0.186 °C/MPa for potato and cassava starch pastes, respectively.

The linear increase in temperature has been widely reported with different values for the extent of temperature rise with homogenizing pressure (Brookman, 1974; Che et al., 2007b; Desrumaux & Marcand, 2002; Floury et al., 2000; Hayes & Kelly, 2003; Thiebaud, Dumay, Picart, Guiraud, & Cheftel, 2003). The strong warming up of the fluid is due to viscous stress caused by high velocity of the fluid flow, friction between the fluid and the valve, and cavitations. The energy input is mostly dissipated in the fluid as heat resulting in temperature rise (Floury et al., 2000).

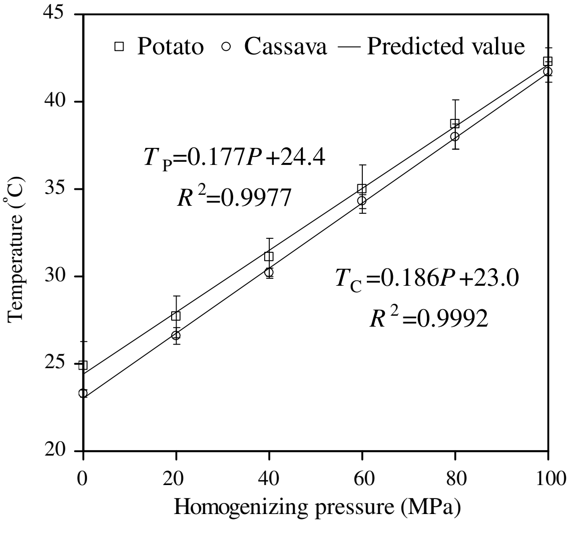

Fig. 1. The influence of the homogenizing pressure on the temperatures of starch pastes. T_P and T_C (°C) are the temperatures of homogenized potato and cassava starch pastes, respectively; and p (MPa) is the homogenizing pressure.

3.2. Microscopy of homogenized starch pastes

Micrographs of starch pastes are shown in Fig. 2. When heated in excess of water at 90 °C, starch granules absorbed large amounts of water and swelled to larger sizes. The starch granules lost their native figures after gelatinization (Fig. 2 A and C). The starch concentration in this work (2 wt.%) is higher than the close packing concentration (0.25 wt.% for potato starch and 1.7 wt.% for cassava starch (Evans & Haisman, 1979)), so the swollen starch granules aggregated in the flocculated pastes. Most of the swollen starch granules were disintegrated during homogenization at 0 MPa (Fig. 2 B and D). After homogenizing the starch pastes at 20 MPa, the swollen starch granules were all disintegrated in the process (the micrographs are not illustrated).

3.3. Effect of high-pressure homogenization on the flow behaviors of starch pastes

The flow curves (apparent viscosity ($\eta_{app}=\tau/\dot{\gamma}$) versus shear rate) of potato and cassava starch pastes are shown in Figs. 3 and 4, respectively. And the parameters of power law model obtained from regression analysis are listed in Table 1.

As can be observed from Figs. 3 and 4, the apparent viscosities of non-homogenized starch pastes decrease with the increase of the shear rate suggesting that these starch pastes exhibit shear-thinning or pseudoplastic behavior. The flow behavior indices of the non-homogenized potato and cassava starch pastes are less than unity (Table 1). The flow behavior indices of the starch pastes homogenized at 0 MPa are greater than those of non-homogenized starch pastes, but are still less than unity. It means that these starch pastes are also shear-thinning fluids, but the shear-thinning behaviors get weaker. As the homogenizing pressure is increased beyond 20 MPa, the flow behavior indices approach unity indicating that these starch pastes could be regarded as Newtonian fluids. The apparent viscosities of these starch pastes were independent of the shear rate in the tested shear rate range. The apparent viscosities (or consistency indices) of the starch pastes were reduced with the increase of the homogenizing pressure from 0 to 100 MPa (see Figs. 3 and 4, and Table 1).

Non-homogenized starch pastes are composites in which swollen starch granules are embedded in an amylose gel matrix (Miles, Morris, Orford, & Ring, 1985). The flow behaviors of starch pastes are thus affected by the rheological properties of swollen starch granules and continuous phase composed of water and amylose (Lagarrigue & Alvarez, 2001). The strong interaction between aggregated granules restricts the flow of starch-water system resulting in high apparent viscosity. Furthermore, the swollen starch granules are soft and deformable to shear force resulting in typical shear-thinning behavior (Rao & Tattiyakul, 1999). The shear-thinning behaviors of potato and cassava starch pastes have been well established (Evans & Haisman, 1979; Mishra & Rai, 2006; Rao & Tattiyakul, 1999).

During homogenization at 0 MPa, the involved moderate shear force partly disintegrated the swollen starch granules. Consequently, the apparent viscosities of starch pastes decreased and the shear-thinning behaviors became less marked. After homogenization at 20 MPa, the swollen starch granules were all disintegrated by the intense mechanical forces involved in the process leading to remarkable decreases in apparent viscosities and transformations from shear-thinning to Newtonian behavior for these starch pastes. Further increase of the homogenizing pressure provided more intense mechanical forces which possibly fragmented some long chain molecules of starches. The starch paste rheology will depend upon the molecular weight of starch. Thus, the degradation of starches arose from high-pressure homogenization

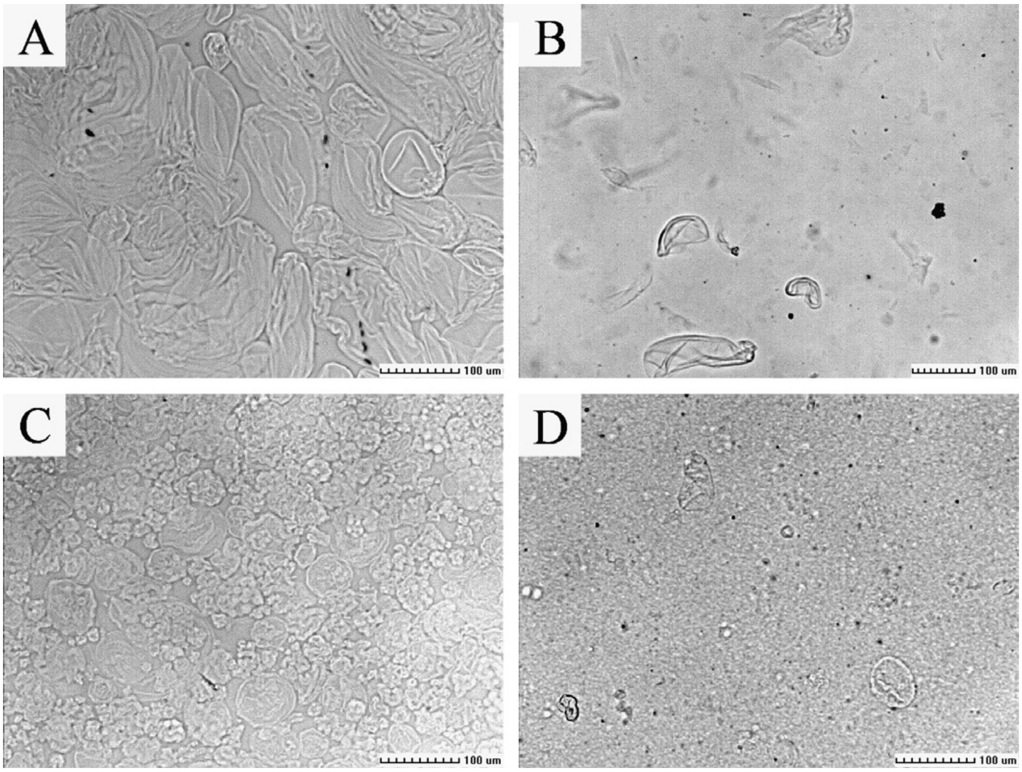

Fig. 2. Micrographs of starch pastes: (A) non-homogenized potato starch paste; (B) potato starch paste homogenized at 0 MPa; (C) non-homogenized cassava starch paste; (D) cassava starch paste homogenized at 0 MPa.

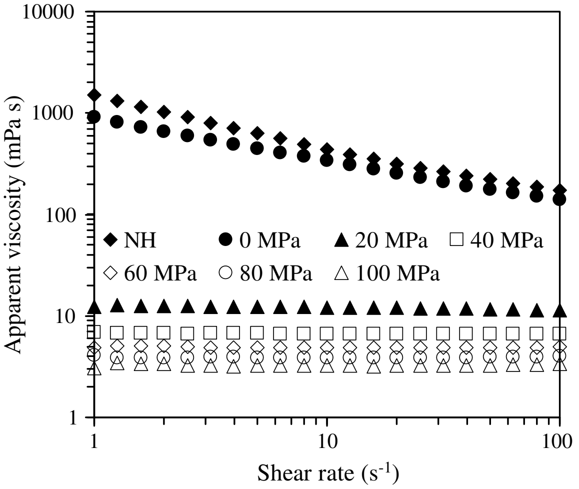

Fig. 3. Typical flow curves (apparent viscosity against shear rate) of potato starch pastes treated under different conditions at 25 °C.

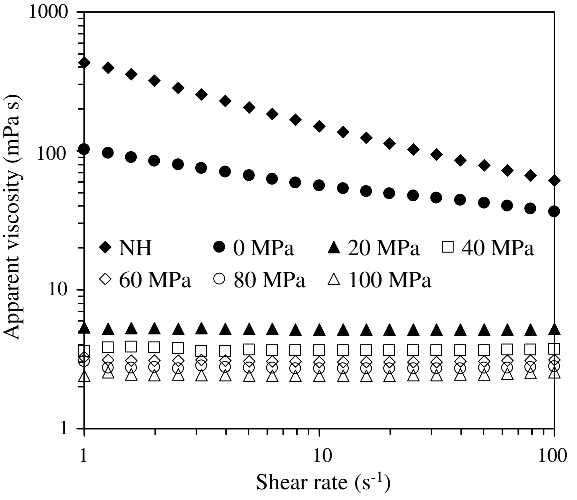

Fig. 4. Typical flow curves (apparent viscosity against shear rate) of cassava starch pastes treated under different conditions at 25 °C.

possibly contributed to the considerable reductions in the apparent viscosities of homogenized starches.

The homogenization induced degradation of starch might be mainly attributed to the violent shear force encountered by starch molecule in the homogenizing valve. Czechowska-Biskup et al. (2005) postulated that a shear induced degradation occurs in a non-random manner (breakage near the mid-point of the chain is preferred), and there is a definite minimum chain length limiting the degradation process. When it is reached, no further chain disruption is observed. It is reasonable to assume that there is a definite minimum apparent viscosity corresponding to the minimum chain length. The starch molecule chains get shorter and shorter with increasing homogenizing pressure and progressively approach the minimum chain length at a given condition. Pyrolysis is not expected to play a dominant role in starch degradation since it is a local phenomenon limited to the surface of cavitation bubbles (Czechowska-Biskup et al., 2005).

As discussed above, the flow behavior of starch pastes homogenized at high pressures (>20 MPa) are probably dominated by the disrupted starch molecules. The flow behavior indices of these starch pastes are all close to unity, which enables the direct comparison between the consistency indices. Thus, the consistency indices of the starch pastes homogenized at higher pressures were further regressively analyzed to assess the effect of homogenizing

Table 1
Power law model parameters for starch pastes treated under different conditions

Starch source	Pressure (MPa)	K (mPa s^n)	n	R^2
Potato	NH	1786.03 ± 408.61	0.56 ± 0.03	0.9897 ± 0.0048
	0	1285.25 ± 595.11	0.56 ± 0.04	0.9984 ± 0.0013
	20	11.60 ± 1.26	0.98 ± 0.00	1.0000 ± 0.0001
	40	6.15 ± 0.54	0.99 ± 0.00	0.9998 ± 0.0002
	60	4.35 ± 0.52	1.00 ± 0.00	0.9998 ± 0.0001
	80	3.52 ± 0.33	1.00 ± 0.01	0.9998 ± 0.0001
	100	2.90 ± 0.32	1.00 ± 0.02	0.9991 ± 0.0007
Cassava	NH	379.97 ± 36.45	0.5934 ± 0.0222	0.9991 ± 0.0002
	0	75.08 ± 20.49	0.7893 ± 0.0076	0.9996 ± 0.0004
	20	5.25 ± 0.09	0.9915 ± 0.0034	0.9999 ± 0.0001
	40	3.50 ± 0.21	0.9964 ± 0.0041	0.9997 ± 0.0001
	60	2.91 ± 0.18	0.9946 ± 0.0009	0.9997 ± 0.0002
	80	2.53 ± 0.23	1.0042 ± 0.0091	0.9996 ± 0.0002
	100	2.29 ± 0.11	1.0085 ± 0.0080	0.9997 ± 0.0003

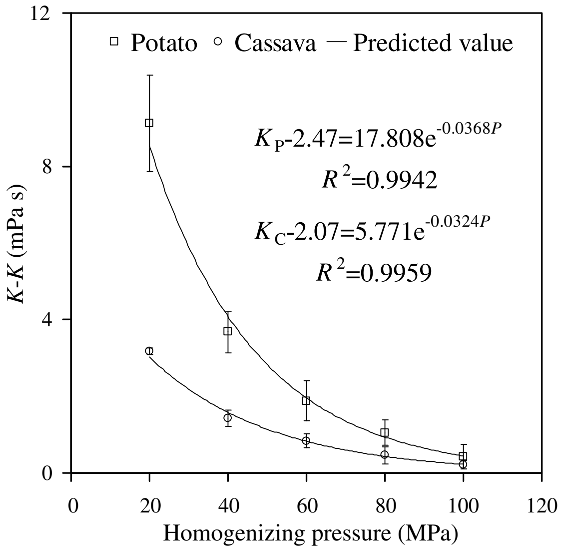

Fig. 5. The relationship between $(K - K_\infty)$ and the homogenizing pressure ($P > 20$ MPa). Where K_P and K_C are the consistency indices of homogenized potato and cassava starch pastes, respectively.

pressure on the starch paste rheology which revealed the change of starch molecule structure (Fig. 5). There seems to be a limiting value (K_∞) that the consistency index will reach when very high homogenizing pressures are applied. The limited viscosity of homogenized oil-in-water emulsion has already been noted by Floury et al. (2000). As can be seen from Fig. 5, after the assumed limiting value of consistency indices (K_∞, 2.47 MPa s for potato starch pastes and 2.07 MPa s for cassava starch pastes) had been removed, two quasi first-order models, which satisfactorily describe the relationships between $(K - K_\infty)$ and homogenizing pressure (P, MPa), were obtained. A modified first-order model which empirically describes the change of consistency index along with processing time (t, s) and homogenizing pressure is obtained after the processing time has been taken into consideration:

$$K - K_\infty = A\exp(-Pt/C)$$

Where, A (MPa s) is constant; C (MPa s) is a first-order reaction rate constant. It shows that the reduction of starch paste viscosity can be achieved by increasing processing time and/or homogenizing pressure. If high temperature rise is avoided by cooling the paste at each pass, more number of passes is possible. In addition, high homogenizing pressure can reduce the processing time significantly.

Table 2
Electrical conductivities and percent light transmittances of homogenized starch pastes

Pressure (MPa)	Electrical conductivity(10^{-3} S/m)		Percent light transmittance (%)	
	Potato starch paste	Cassava starch paste	Potato starch paste	Cassava starch paste
0	3.38 ± 0.21	4.45 ± 0.08	57.1 ± 10.0[a]	55.1 ± 5.7[a]
20	3.29 ± 0.18	4.39 ± 0.11	90.9 ± 1.0[a]	81.1 ± 1.3[a]
40	3.34 ± 0.09	4.45 ± 0.08	92.0 ± 3.6[a]	82.9 ± 1.0[a]
60	3.37 ± 0.05	4.54 ± 0.05	92.6 ± 2.9[a]	83.1 ± 1.1[a]
80	3.40 ± 0.05	4.51 ± 0.05	94.2 ± 2.1[a]	82.6 ± 2.0[a]
100	3.47 ± 0.02	4.64 ± 0.10	93.4 ± 0.7[a]	82.8 ± 1.2[a]

[a] Values followed by the same letters in a column do not differ significantly at $p < 0.01$ level.

Table 3
Analysis of variance for electrical conductivities of homogenized starch pastes

Source of variations	SS[a]	DF[a]	MS[a]	F-ratio	Probability
Starch source	11.22	1	11.22	1055.68	0.00
Pressure	0.15	5	0.03	2.91	0.03
Interaction	0.01	5	0.00	0.24	0.94
Residual error	0.26	24	0.01		
Total	11.65	35			

[a] SS, sum of squares; DF, degrees of freedom; MS, mean square.

3.4. Electrical conductivities of homogenized starch pastes

The electrical conductivities of homogenized starch pastes are listed in Table 2. The electrical conductivities of homogenized potato starch pastes are less than those of homogenized cassava starch pastes. Two-factor analysis of variance with repetition was utilized to assess the effects of homogenizing pressure and starch source on the electrical conductivity of starch pastes and the results were summarized in Table 3. The results suggest that the electrical conductivity is not affected by homogenizing pressure but starch source ($p < 0.01$).

Electric current is carried by the movement of ions (Shirsat, Lyng, Brunton, & McKenna, 2004). So the electrical conductivity of liquid depends on the amount of ions that can move freely in the liquid. It is recognized that starches contain phosphates which are bound to amylopectin through ester binding. These ions are released from starch granules during gelatinization resulting in significant increase in electrical conductivity (Chaiwanichsiri, Ohnishi, Suzuki, Takai, & Miyawaki, 2001). The difference in electrical conductivity between potato and cassava starch paste might be ascribed to the various amounts of phosphates in native starches. The total amount of ions was not changed during homogenization since the starch concentration was kept constant. No gel network that could restrict the movement of released ions formed in the starch paste after gelatinization due to the low concentration of starch. So the electrical conductivities were not increased after homogenization even though the apparent viscosities were reduced sharply.

3.5. Light transmittances of homogenized starch pastes

Paste clarity is a quality characteristic of starch because it gives shine and opacity to product color (De la Torre-Gutiérrez, Chel-Guerrero, & Betancur-Ancona, 2008). Light transmittance provides the information on the behavior of starch paste when the light passes through it (Sandhu, Singh, & Lim, 2007). Higher light transmittance implies a more transparent paste. The percent light transmittances of homogenized starch pastes are presented in Table 2. The percent light transmittances of starch pastes increased dramatically after homogenization at 20 MPa, however, further increases of homogenizing pressure had no more effect on them ($p < 0.01$).

The light transmittance of starch paste is a function of the amount of swollen starch granules in the paste which refract light (Singh, Inouchi, & Nishinari, 2006). The more swollen starch granules there are, the lower light transmittance is. Generally, starch with large granules tends to leave fewer swollen starch granules after gelatinization (Singh, McCarthy, Singh, & Moughan, 2008), since large granules are less resistant to hydrothermal and mechanical treatments. Potato starch granules are larger than those of cassava starch (Jobling, 2004). There were fewer swollen starch granules in potato starch pastes resulting in higher percent light transmittances. The swollen starch granules in non-homogenized starch pastes were all disintegrated during homogenization at 20 MPa promoting paste clarity. Consequently, further increases of homogenizing pressure could not develop more transparent pastes any longer. Singh et al. (2008) claimed that the repulsion between adjacent starch molecules caused by the negatively charged phosphate groups could reduce interchain associations, which increased starch paste transparency. However, the effect of phosphates on starch paste clarity is not clear in this study, because cassava starch with higher phosphate content (see section 3.3) gives lower light transmittance.

4. Conclusions

Potato and cassava starch pastes at the concentration of 2 wt.% were homogenized at pressures ranging from 0 to 100 MPa to evaluate the effect of high-pressure homogenization on the properties of starch pastes. During high-pressure homogenization, the energy input is mostly dissipated in starch paste as heat, as a result the temperature of the homogenized starch paste increased linearly with the increasing of homogenizing pressure. High-pressure homogenization first disintegrates the swollen starch granules in the starch paste causing noteworthy decrease in apparent viscosity, increase in paste clarity, and a change from shear-thinning to Newtonian fluid. Homogenization involved shear force then possibly breaks starch molecule chains causing further decrease in apparent viscosity. The apparent viscosity of homogenized starch paste decreases with the increase of homogenizing pressure until a limiting value is achieved. However, the electrical conductivity of starch paste is not affected by high-pressure homogenization.

High-pressure homogenized starch paste is easier to flow and more transparent than non-homogenized starch paste at the same concentration. These homogenized starch pastes may find new applications in industries. Future research may focus on the complex effects of homogenizing conditions (valve geometry, homogenizing pressure and temperature, etc.) on the structure and properties of starches.

Acknowledgments

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