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Effects of high-pressure homogenization on the properties of starch-plasticizer dispersions and their films

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ABSTRACT

The properties of starch-plasticizer (glycerol:xylitol = 1:1) dispersions obtained with and without high-pressure homogenization and their corresponding films were investigated. The fully gelatinized dispersions with or without homogenization were subsequently converted into films using solution casting. The apparent viscosity of the dispersions with or without homogenization was determined. The water vapor permeability, opacity, crystalline/amorphous nature, and mechanical properties of these starch-based films were determined. The high-pressure homogenization greatly reduced the apparent viscosity of the dispersions and altered the flow behavior from shear-thinning into Newtonian one. The films obtained from high-pressure homogenized dispersions had better moisture barrier property, better film transparency and higher tensile strength but lower elongation.

1. Introduction

Starch is one of the most abundantly occurring biopolymers in nature. It has currently attracted considerable attention as a raw material for biodegradable thermoplastic polymer or biodegradable particulate filler (Angellier, Boisseau, Dole, & Dufresne, 2006). There are several ways to convert starch materials into film or coating, including solutions casting, extrusion, thermomolding, injection, sheeting and blowing. For the success of most of these methods the starch–water (or solvent) dispersions have to be prepared in such a way that they result in good and uniform films. In order to form a homogenous, smooth and strong film, it is important that the starch gets fully dissolved in water or other compatible solvents (Shamekh, Myllärinen, Poutanen, & Forssell, 2002). Due to large molecular size and strong tendency of hydrogen bonding, gelatinized starch solution is usually composed of swollen starch granules, which is not beneficial in forming homogenous and excellent films (Cheng, Chen, Liu, Ye, & Ke, 2010).

To reduce the size of starch granules and enable them to fully dissolve in solvents, there are two convenient techniques for a controlled reduction in the size of polysaccharides, namely, sonication and high-pressure homogenization (Floury, Desrumaux, Axelos, & Legrand, 2002; Lorimer, Mason, Cuthbert, & Brookfield, 1995; Paquin, 1999; Tang, Huang, & Lim, 2003). Sonication induces high pressure gradients and high local velocities in liquid layers due to very rapid formation and collapse of cavities. Hence, sonication can be conveniently used for dissolving the biopolymers in compatible solvents (Czechowska-Biskup, Rokita, Lotfy, Ulanski, & Rosiak, 2005). High-pressure homogenization is a novel technique which is extensively used in recent years to emulsify and disperse various products in industries such as chemical, pharmaceutical, specialty biotechnological and foods (Floury et al., 2002). High-pressure homogenization reduces the particle size of polysaccharides through simultaneous cavitation, turbulence and shear (Paquin, 1999).

These treatments are expected to greatly influence the properties of starch or starch-based dispersion and the quality of final products. A large body of work by various researchers in this aspect has already been reported (Che et al., 2009; Cheng et al., 2010; Guraya & James, 2002). Kang and Min (2010) studied the potato peel-based biopolymer film using high-pressure homogenization (138 MPa with 2, 5 and 10 passes), irradiation and ultrasound. It was found that the moisture barrier, tensile and color properties of the films produced from high-pressure homogenized dispersions were better than those obtained by ultrasound or irradiation.

However, there is lack of details regarding the influence of homogenization pressure and the number of passes on starch-plasticizer dispersions and the characteristics of resultant films. In this context, this work was aimed at characterization of starch-plasticizer dispersions and also the resultant films in terms of apparent viscosity, water vapor permeability, opacity, crys-

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talline/amorphous nature and mechanical properties as a function of the homogenization pressure and the number of passes through a high-pressure homogenizer.

2. Materials and methods

2.1. Materials

Corn starch was obtained from Hebei Zhangjiakou Yujing Food Co. Ltd. (Hebei, China). Glycerol (analytical grade) was purchased from Beijing Chemical Reagent Ltd. (Beijing, China). Xylitol (food grade) was purchased from Tianjin Jinguigu Science & Technology Development Co. Ltd. Moisture content of the starch and glycerol was determined and compensated for while making solutions. The moisture content of the crystalline xylitol was taken to be zero. All of these materials were used as received.

2.2. Preparation of dispersion

Starch-plasticizer dispersions at a solid concentration of 5.0 wt.% were prepared by adding 7.0 g of pre-dried corn starch and 3.0 g of plasticizers (glycerol and xylitol) into deionized water at room temperature. The glycerol:xylitol ratio of 1:1 was maintained throughout. The dispersion batches were thoroughly stirred at 300 rpm (in beakers) for 1 h using a thermostated water bath in boiling condition. Evaporation was minimized by covering the beakers with six layers of preservative film. A high-pressure homogenizer (AH100D, ATS Engineering Inc., Italy) was used to homogenize the previously fully gelatinized dispersions. Initially, the homogenizer was maintained at 50 °C with the use of a circulating water bath. Finally, the fully gelatinized dispersions were homogenized at 10, 20, 30 and 50 MPa with 1, 2, and 3 passes.

2.3. Microscopy of starch film solutions

The starch-plasticizer dispersions obtained with and without high-pressure homogenization 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.

2.4. Rheological properties of starch film solutions

Rheological measurements were carried out using a stress-controlled rheometer (AR2000ex, TA Instruments Ltd., Crawley, UK), fitted with an aluminum parallel plate (40 mm diameter and 1 mm gap) for steady shear measurements. For each test, 20 ml of freshly prepared dispersion (Section 2.2) was used. A thin layer of silicone oil (0.378 Pa s, 40 °C) was applied on the surface of the samples in order to prevent evaporation. The sample was allowed to equilibrate for 2 min on the Peltier system before the tests were carried out. The steady shear tests were performed at 40 °C and the shear rate was increased from 1 to 100 s^{-1}. The power law model given by Eq. (1) below was used to describe the flow behavior of these starch-based dispersions.

$$\sigma = K\gamma^n \quad (1)$$

where σ is shear stress (Pa), γ is shear rate (s^{-1}), K (which corresponds to viscosity when the fluid is Newtonian) is consistency index (Pa s^n), and n is flow behavior index (Floury, Desrumaux, & Lardières, 2000).

2.5. Preparation of the film

Films were cast by syringing 13 mL of the above mentioned dispersions into 9 cm diameter polycarbonate petri dishes. Films were dried for over 8 h at 45 °C.

2.6. Water vapor permeability (WVP)

WVP tests were conducted using ASTM method E96-80 with some modifications (ATSM, 1989). Each film sample was sealed over a circular opening of 0.000568 m^2 in a permeation cell that was conditioned at 25 °C in a desiccator. To maintain a 75% RH gradient across the film, anhydrous calcium chloride (0% RH) was placed inside the cell and a sodium chloride saturated solution (75% RH) was placed in the desiccator. The extent of water vapor transport was determined from the weight gain of the permeation cell. Cell weight was measured every 24 h over a week. Changes in the weight of the cell were recorded and plotted as a function of time. The slope of each line was calculated by linear regression and the water vapor transmission rate (WVTR) was calculated from the slope of the straight line (g/s) divided by the cell area (m^2). After the permeation tests, film thickness was measured and the WVP (g Pa^{-1} s^{-1} m^{-1}) was calculated. WVTR and WVP are determined using Eqs. (2) and (3) as given below, respectively.

$$\mathrm{WVTR} = \frac{1}{A}\left(\frac{\Delta m}{t}\right) \quad (2)$$

$$\mathrm{WVP} = \frac{\mathrm{WVTR} \times e}{S \times (R_1 - R_2)} \quad (3)$$

where Δm is the mass change of the cell test (g), t is the time (s), A is the test area (m^2), S is the saturation vapor pressure of water (3.169×10^3 Pa) at the test temperature (25 °C), R_1 and R_2 are the relative humidity (RH) values in the desiccators and the permeation cell, respectively. Similarly, e is the film thickness (m). All of these tests were carried out in triplicate.

2.7. Opacity

Opacity of the films was determined using a modified BSI standard procedure (1968) (Mali, Grossmann, García, Martino, & Zaritzky, 2004a) on an UV–vis spectrophotometer (TU-1810, Beijing Purkinje General Instrument Co., Ltd., Beijing, China). Film samples were placed in the internal side of a spectrophotometer cell to record the absorbance spectrum between 400 and 800 nm. Opacity of the films was defined as the area under the curve and expressed as absorbance unit × nanometers (AU nm). All the tests were conducted in triplicate.

2.8. X-ray diffractometry (XRD)

X-ray diffraction tests were carried out with a XD-2 X-ray diffractormeter (Beijing Purkinje General Instrument Co., Ltd., China) under the following conditions: Nickel filtered CuKα radiation (λ = 0.15406 nm) at a voltage of 36 kV and current of 20 mA. Samples were scanned from 10° to 40° (2θ) with a scanning rate of 0.25° min^{-1} and sampling interval of 0.02°.

2.9. Mechanical properties

The mechanical properties including the tensile strength (TS) and percent elongation at break (%E) were measured using a material testing machine (Instron 4411, Britain) in accordance with ASTM-D D412-98a (ASTM, 1998). Seven specimens were cut from each of the starch films with a size of 70 mm × 15 mm. The thickness of the samples was measured using a micrometer before test.

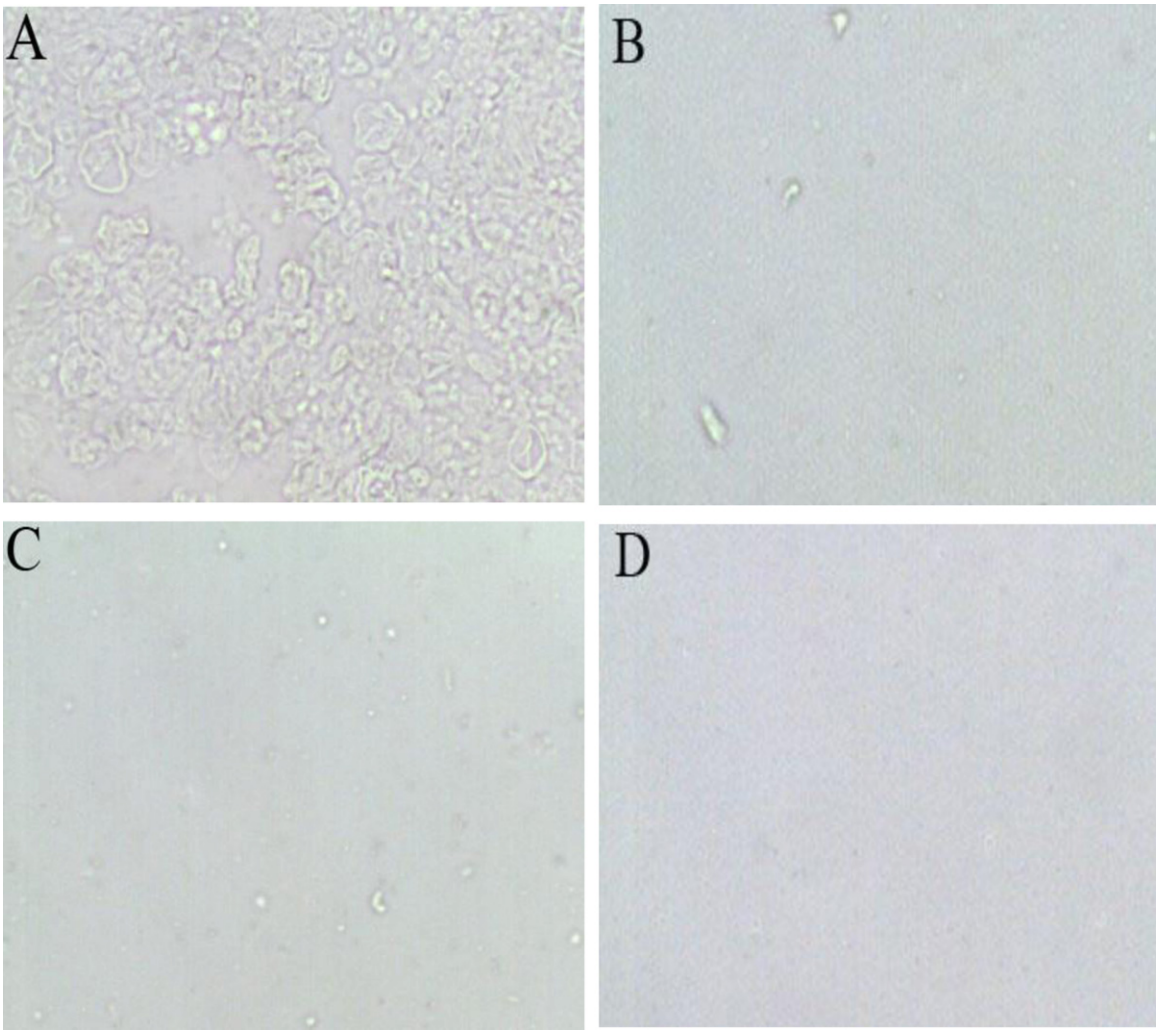

Fig. 1. Micrographs of starch-plasticizer dispersions (40×): (A) 0 MPa 0 pass; (B) 10 MPa 1 pass; (C) 10 MPa 3 passes; (D) 20 MPa 3 passes.

Measurements were taken at five different positions for each sample and the average value of these readings is reported here. In all of these tests, initial grip separation was 37 mm and cross-head speed was 10 mm/min. The tensile strength was calculated by dividing the maximum force exerted on the film during fracture by the cross-sectional areas. Percent elongation at break was expressed as percentage of change of the original length of a specimen between grips at break.

2.10. Statistical analysis

All experiments were carried out in at least triplicate and results are reported as the mean and standard derivation of these measurements. Analysis of variance (ANOVA) was carried out in order to test the significance. The SPSS statistical package (LEAD Technologies, US) was used for this purpose.

3. Results and discussion

3.1. Microscopic images of the dispersions

Micrographs of starch-plasticizer dispersions are shown in Fig. 1. As can be seen in this figure, when heated in boiling water, starch granules absorbed large amounts of water and swelled (Fig. 1A). When the gelatinized dispersions were homogenized (10 MPa with 1 pass) the swollen starch granules were ruptured (Fig. 1B). As the number of passes increased, the size of the granules was reduced (Fig. 1C). Finally, the swollen starch granules were completely ruptured or disintegrated when processed at 20 MPa with 3 passes (Fig. 1D).

3.2. Rheological properties of starch film solutions

The flow curves (apparent viscosity versus shear rate) of the dispersions are presented in Fig. 2. The parameters of the power law model obtained through regression analysis are listed in Table 1. The power law model was also used to predict the apparent viscosity of the dispersion without homogenization. It fitted the testing apparent viscosity very well (Fig. 2).

Table 1
Power law model parameters for starch-plasticizer dispersions treated by high-pressure homogenization.

Samples		K (mPa s^n)	n	R^2
Pressure (MPa)	Passes			
0	0	1349.90 ± 19.12	0.47 ± 0.009	0.99
10	1	39.66 ± 0.58	0.89 ± 0.010	0.99
10	3	25.04 ± 0.94	0.90 ± 0.025	0.99
30	3	23.12 ± 0.27	0.90 ± 0.008	1
50	3	12.44 ± 0.37	0.91 ± 0.017	0.99

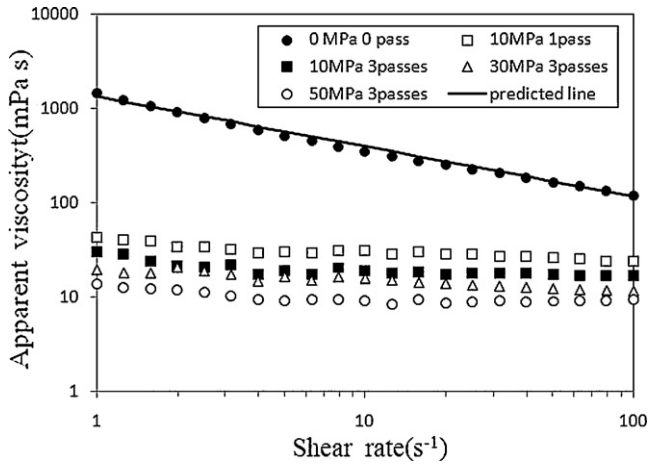

Fig. 2. Typical flow curves (apparent viscosity verse shear rate) of starch-plasticizer dispersions treated by high-pressure homogenization.

As can be seen from Fig. 2, the apparent viscosity of non-homogenized dispersion decreases with increase in shear rate suggesting that the dispersion is exhibiting a shear-thinning or pseudoplastic behavior. The flow behavior index of the non-homogenized dispersion is 0.47, well below unity (Table 1). As the homogenization pressure and the number of passes are increased, the apparent viscosity of the dispersions become independent of the shear rate in the range of shear rate studied. This indicates that the flow behavior of the homogenized dispersions becomes Newtonian. The apparent viscosity values (or consistency induces) of these dispersions decreased when the native starch dispersions were homogenized at different pressures. When the dispersions were homogenized at 10 MPa with passes from 1 to 3, the consistency induces was decreased from 39.66 ± 0.58 to 25.04 ± 0.94 mPa s^n (Table 1). However, there is no significant difference on the apparent viscosity values (or consistency induces) of starch dispersions between homogenization pressure at 10 and 30 MPa with 3 passes (see Table 1 and Fig. 2).

Non-homogenized starch-based dispersions used for preparation of films are composite in which swollen starch granules are embedded in an amylose gel matrix (Miles, Morris, Orford, & Ring, 1985). The flow behavior of these dispersions is thus affected by the rheological properties of swollen starch granules (Lagarrigue & Alvarez, 2001). High apparent viscosity in this system is because of the strong interaction between swollen granules which restricts the flow of starch–water system. Furthermore, the swollen starch granules are soft and deformable to shear force resulting into typical shear-thinning behavior (Rao & Tattiyakul, 1999). After the dispersions were homogenized, the swollen starch granules were disintegrated by the intense mechanical forces involved in the process. Thus, the apparent viscosity of the homogenized starch-plasticizer dispersions is remarkably decreased, and the shear-thinning behavior transformed into Newtonian one.

Modig, Nilsson, Bergenståhl, and Wahlund (2006) studied the homogenization-induced degradation of modified starch using asymmetrical flow field-flow fractionation and multi-angle light scattering. The results showed that the homogenization at 15 MPa caused a significant decrease in both molar mass and root mean square radius of three different octenyl succinate anhydride starch samples originating from potato. The starch paste rheology also depends upon the molecular weight of starch. The degradation induced by homogenization might be mainly attributed to the violent shear force encountered by starch molecule in the homogenizing valve. However, the degradation is not efficient at low power (Czechowska-Biskup et al., 2005). So the flow behavior of dispersions homogenized at low pressures (<30 MPa) are probably dominated by the rupture of swollen starch granules. As the dispersions homogenized at pressure changed from 30 MPa to 50 MPa with 3 passes, the degradation of starch contributed to the lowering of the apparent viscosity of the homogenized dispersions.

3.3. Opacity

Opacity is a property of prime importance if the film is to be used in food coating or in food packaging (Gontard, Guilbert, & Cuq, 1992). Film opacity is an effective index for providing information on the size of dispersed particles in the starch matrix. Particle sizes or aggregate domains larger than visible wavelength obstruct the light and give rise to translucent or opaque films (Piyaporn, Duangdao, Duanghathai, & Kawee, 2007). Opacity is affected by the thickness of the film (Wu et al., 2009). Because of this reason the film thickness was rigorously controlled in this study so that the uncertainty in film opacity due to variation in film thickness is avoided. The opacity values of the film samples are presented in Table 2, where lower relative opacity values indicate that the films are more transparent. In the case of the films obtained from homogenized dispersions, the opacity decreased from 83.69 ± 8.02 to 22.85 ± 6.52 AUnm. As the homogenization pressure and the number of passes increased, the films became more transparent. In the case of the dispersions which were not homogenized, when heated in boiling water, starch granules absorbed large amounts of water and swelled into larger granules. The swollen starch granules subsequently aggregated when they were dried into the films. These relatively large particle sizes of the aggregates obstructed the light passing through the films because of which the transparency of the film became relatively low. When the dispersions were homogenized, the swollen starch granules were ruptured and starch molecules became greatly stretched. The films obtained from the homogenized dispersions allowed better transmittance of the light which resulted in greatly transparent films. Similar observations (Bertuzzi, Armada, & Gottifredi, 2007) were made with high amylose corn starch based films. Alkaline treatment allowed sufficient unfolding and dispersion of the starch molecules in the dispersion thereby resulting into homogeneous and transparent starch films. However, there is no significant difference on the opacity of the films at high pressure of 50 MPa with different passes. It is probably because there is no more starch aggregates that can be ruptured at high pressure. Only some starch molecules were degraded. Even so, there is a definite minimum chain length limiting the degradation process at a given condition (Czechowska-Biskup et al., 2005).

3.4. Water vapor permeability (WVP)

An important function of food packaging is to avoid or at least minimize the moisture transfer between the surrounding atmosphere and the food. Due to this reason, lower water vapor permeability is a desired attribute of the packaging films (Mali, Karam, Ramos, & Grossmann, 2004b).

In the case of films formulated without homogenization the WVP values were close to 0.51 g m^{-1} h^{-1} Pa^{-1}. As shown in Table 2, the WVP values decreased when the dispersions were homogenized. The rate of water vapor transfer through a starch film includes the rates of adsorption, diffusion and desorption of water molecules. In this process, the water molecules dissolve in one side of the film, move through the porous space of the film and then desorb from the other side (Miller & Krochta, 1997). Chemical structure, amylose to amylopectin ratio, polymer packing, crystallinity, amount of plasticizer used and the environmental humidity can have profound effect on the water vapor permeability of starch films (Chung & Lai, 2007). It is believed that structural modification

Table 2
Effect of high-pressure homogenization on water vapor permeability (WVP), opacity and mechanical properties of starch films.

Samples		WVP ($\times 10^{-1}$ g m^{-1} h^{-1} Pa^{-1})	Opacity (AU nm)	Mechanical properties	
Pass	Pressure (MPa)			TS (MPa)	%E
0	0	5.10 ± 0.09^{a}	83.69 ± 8.02^{a}	2.56 ± 0.18^{a}	62.94 ± 8.35^{a}
1	10	4.25 ± 0.09^{b}	53.32 ± 7.46^{b}	2.75 ± 0.26^{a}	56.14 ± 6.44^{ab}
1	20	4.09 ± 0.39^{b}	36.64 ± 3.84^{cd}	2.98 ± 0.24^{ab}	53.29 ± 4.11^{ab}
1	30	3.97 ± 0.20^{b}	30.36 ± 3.95^{de}	3.24 ± 0.54^{bc}	51.87 ± 6.41^{b}
1	50	3.90 ± 0.19^{b}	27.25 ± 1.09^{ef}	3.51 ± 0.35^{c}	48.79 ± 10.81^{b}
2	10	4.04 ± 0.74^{b}	43.51 ± 2.22^{c}	2.94 ± 0.26^{ab}	55.20 ± 15.09^{ab}
2	20	3.92 ± 0.43^{b}	32.81 ± 3.01^{de}	3.30 ± 0.29^{bc}	51.61 ± 1.59^{b}
2	30	3.92 ± 0.33^{b}	26.68 ± 3.92^{ef}	3.47 ± 0.22^{c}	49.37 ± 5.65^{b}
2	50	3.83 ± 0.25^{b}	24.81 ± 3.67^{ef}	3.59 ± 0.24^{c}	48.11 ± 9.72^{b}
3	10	3.91 ± 0.50^{b}	37.50 ± 1.90^{cd}	3.23 ± 0.34^{bc}	51.77 ± 6.36^{b}
3	20	3.80 ± 0.50^{b}	30.25 ± 3.98^{de}	3.48 ± 0.37^{c}	49.70 ± 2.33^{b}
3	30	3.76 ± 0.13^{b}	24.23 ± 4.05^{f}	3.61 ± 0.42^{c}	49.04 ± 14.01^{b}
3	50	3.73 ± 0.11^{b}	22.85 ± 6.52^{f}	3.64 ± 0.68^{c}	47.64 ± 6.60^{b}

Values are mean ± standard deviation (n = 3, 3 and 7 for WVP, opacity and mechanical properties, respectively). Different letters within the same column differ significantly (P < 0.05).

of the starch network must have occurred when the dispersions were homogenized. We observed some extent of aggregation in gelatinized starch dispersions. When the films were prepared without homogenization, greater extent of pores or cracks were observed in those films which facilitated the water vapor permeation. After the homogenization the aggregates were broken down and the uniformity of the dispersion was greatly enhanced. As a consequence, the formation of pores or cracks was minimized and a more compact structure was formed. The greater compactness in the structure of the film offered greater resistance to the diffusion of the water molecules and ultimately resulted into lower WVP values (Phan, Debeaufort, Voilley, & Luu, 2009). Once the non-homogenized starch dispersions were treated with high-pressure homogenization, the WVP values of the resulting films were decreased significantly (Table 2). The apparent viscosity of non-homogenized starch dispersion was higher than the homogenized ones. It was easier to hold small bubbles in the non-homogenized dispersions. Furthermore, there were a lot of giant swollen starch granules in the non-homogenized dispersions. Pores or cracks could be formed easily in those films and the transfer of water vapor through the starch film was preferred. However, there was no significant difference among the treated films (Table 2). This is probably because all the homogenized dispersions had low apparent viscosity and the starch granules were ruptured into small pieces. Bubbles and swollen granules were minimized which resisted the diffusion of the water vapor.

3.5. X-ray diffractometry

XRD measurements were carried out for starch films obtained using starch-plasticizer dispersions homogenized at different pressures and the number of passes. The intensity curves showed strong peaks at scattering angles of 16.7° and 19.6° and a weaker peak at scattering angle of 21.5° (Fig. 3). The peaks observed at these scattering angles are typical B-type and V-type crystal structures. V-type diffraction pattern indicates the single-helical structures formed by complexes between amylose and glycerol or lipids. As can be seen in Fig. 3, the peak intensity (2θ = 16.7°) of homogenized starch films is higher than that of the non-homogenized one.

The degree of crystallinity in a polymer sample depends on the mobility of the molecule chains during the crystallization process (Rindlav-Westling, Stading, Hermansson, & Gatenholm, 1998). After high-pressure homogenization, some of the long chains of starch molecules were disrupted and the viscosity of the starch-plasticizer dispersions became lower which further facilitated the mobility and the rearrangement of starch molecules. Thus, the development of B-type crystallinity was favored in the starch film obtained from the homogenized dispersions compared to one obtained from non-homogenized dispersion.

3.6. Mechanical properties

Tensile strength of the test films is presented in Table 2. There are remarkable differences between homogenized and non-homogenized starch films. As shown in this table, as the homogenization pressure and the number of passes increased the tensile strength of starch films augments. In the absence of the homogenization, the tensile strength of starch-plasticizer films is about 2.56 MPa. When the same dispersion was homogenized at 50 MPa with 3 passes, the tensile strength increased to about 3.64 MPa. After dispersions were treated by homogenization, the swollen starch granules were disintegrated and amylose was released fully in casting dispersions. During the drying process of the dispersions, a large proportion of water evaporates which facilitates the formation of starch network. During this stage, the polymer chain density per unit area increases which further facilitates amylose chains to come together (Alves, Mali, Beléia, & Grossmann, 2007). It is known that amylose network forms denser films than amylopectin (Rindlav-Westling et al., 1998) which may be the main reason behind the denser films obtained from homogenized dispersions. But the tensile strength was not always improved significantly as the homogenization pressure and the number of passes increased. No significant effects of passes were observed on the tensile strength of films at high pressure

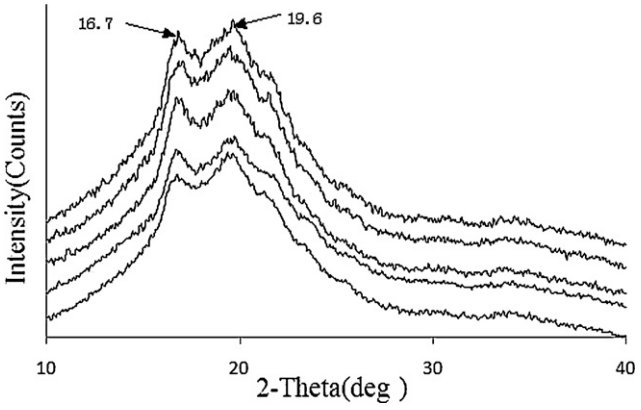

Fig. 3. X-ray diffraction patterns of starch films formed from dispersions with high-pressure homogenization (0 MPa 0 pass, 10 MPa 3passes, 20 MPa 3passes, 30 MPa 3passes and 50 MPa 3passes from bottom to top).

of 50 MPa. This probably occurred because a low content of amylase was released in casting dispersions as no more pieces of starch granules could be ruptured.

However, as a result of more compact structure, the films obtained from homogenized dispersions were more fragile than the one obtained from non-homogenized dispersion. As can be seen from Table 2, in the case of the films obtained from homogenized dispersions, elongation at break is about 47.64% compared to 62.94% of the film from non-homogenized dispersion. These results are supported by the X-ray diffraction data which show that the homogenized starch films indeed had higher crystallinity. It is established that the elongation property decreases with the increase in the crystallinity in films (Lafargue, Pontoire, Buléon, Doublier, & Lourdin, 2007). It was strangely that the elongation at break did not change further among the films obtained with high-pressure homogenization. This phenomenon may be affected by a lot of reasons. On the one hand, pores or cracks minimized by homogenization increased the continuity of films which facilitated elongation. On the other hand, increased crystallinity made films more rigid which decreased the elongation of films.

4. Conclusions

In this study, starch-plasticizer (glycerol:xylitol = 1:1) dispersions at 5 wt.% solid concentration were homogenized at pressures ranging from 10 to 50 MPa with 1–3 passes. This extent of homogenization was found to disrupt the swollen starch granules and reduce the chain length of the starch molecules to some extent. The homogenization altered the shear-thinning behavior of the dispersions into Newtonian and greatly reduced the dispersion viscosity. The homogenized dispersions were found to improve the properties of the films obtained from solution casting. The transparency, moisture barrier properties and tensile strength of the films obtained from homogenized dispersions were much better than those obtained from the non-homogenized samples. However, the films obtained from high-pressure homogenized dispersions had poorer elongation property.

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