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Impact of ultrasonic treatment on properties of starch film-forming dispersion and the resulting films

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ABSTRACT

In this study, power high intensity ultrasound was used to treat the gelatinized maize starch dispersions. To investigate the effects of ultrasound treatment on the properties of the film-forming dispersions and their resulting films, the apparent viscosity, solubility, morphology of the film-forming dispersions and the opacity, water vapor permeability, mechanical properties, X-ray diffraction of the films formed from these dispersions were tested. It was found that gelatinized maize starch dispersions treated by ultrasound for a certain period of time has a drastic decrease in viscosity, and a distinct increase in solubility. Microscopic observation shows distinctly rapture of ghost fragments in the dispersion with prolonged treatment time. It was also found that ultrasound treatment produces an excellent film with good transparency, improved moisture resistance and stronger structure.

1. Introduction

The development of biodegradable packaging alternatives has been the subject of much research and development in recent times, particularly with regard to renewable alternatives to traditional oil-derived plastics (Debeaufort, Quezada-Gallo, & Voilley, 1998; Mali, Grossmann, García, Martino, & Zaritzky, 2005). Starch is considered as one of the most promising alternative because it is an abundant, inexpensive, biodegradable and edible material with good film formation ability (Mali, Grossmann, García, Martino, & Zaritzky, 2002). There are a few techniques than can be used to convert starch materials into film or coating, including extruding and followed by blowing process based on its thermoplastic properties (Matzinos, Tserki, Kontoyiannis, & Panayioto, 2002; Otey, Westhof, & Doane, 1987; Thunwall, Boldizar, & Rigdahl, 2006); casting, dipping or spraying after it is dissolved in a solvent (usually water) and followed by a drying process. However, unlike films produced from casting or spraying, film blowing processing is restricted by poor tenacity of the thermo-melted starch after extruding (Thunwall, Kuthanová, Boldizar, & Rigdahl, 2008). Dissolving sarch in water then casting, dipping or spraying on a specific surface is still the main method to produce the edible starch film or coating (Galdeano et al., 2009). A lot of work has been done by pioneers in this field since 1970s (Mali et al., 2005). In order to form a homogenous, smooth and tight film, it is important that the starch should be fully dissolved in water or other solvents (Shamekh, Myllärinen, Poutanen, & Forssell, 2002). Although native starch is of high hydrophilic polymer, it is difficult to dissolve in water because of their large molecular size and strong hydrogen bonding. To attain maximum dissolution, as a rule, starch dispersion should be performed at low starch concentrations and at temperatures above 100 °C (Lagarrigue, Alvarez, Cuvelier, & Flick, 2008). These conditions however are not very economical. To attain a more feasible and economical dissolution method, starch has been dissolved in water after being partially degraded by acid or enzymatic treatment (Chung & Lai, 2007; Shamekh et al., 2002).

The ultrasonic treatment of starch has been performed as early as 1933 (Kardos & Luche, 2001). From that point on, more and more researchers have shown that ultrasonic treatment has a great effect on the behaviors of gelatinized starch dispersions. After being treated by ultrasound, the starch dispersion showed a decrease in viscosity, and an increase in solubility and clarity due to the increase in numbers of free mobile macromolecules, rather than breakage of starch molecules (Jenny, Kai, Raymond, Sandra, & Muthupandian, 2009; Iida, Tuziuti, Yasui, Towata, & Kozuka, 2008). These studies suggest that ultrasound treatment facilitates the disintegration of starch granules and the formation of homogenous starch solution. When compared to conventional dissolution methods for starch,

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ultrasonic treatment is relatively inexpensive and efficient (Liu, Du, & John, 2007). This study focuses on the behavior of ultrasound treated maize starch dispersion and properties of the film formed from those dispersions since few investigations have been made in this field.

2. Materials and methods

2.1. Materials

Normal maize starch was purchased from Kuihua Group Co., Ltd. (Shan Dong, China). On a dry weight basis, the chemical composition was: carbohydrates 99.05%, proteins 0.24%, fat 0.45%, ash 0.12%, and amylose 28%. All other reagents were of analytical grade.

2.2. Characterization of gelatinized maize starch dispersion treated by ultrasound

Maize starch was mixed with distilled water in a beaker. The maize starch suspensions with concentrations of 3%, 5%, 7%, 10% (w/w) were heated in a water bath to 90°C and then held at that temperature for 30 min with regular stirring at speed of 100 rpm. The gelatinized maize starch dispersions (GMSDs) thus prepared were cooled down to 60 °C. Approximately 100 g of starch dispersions with different concentrations were transferred from the vessel to a 250 ml beaker and treated by ultrasound for 0, 2.5, 5, 10, 15 and 30 min respectively at 60 ± 3 °C, using a 20 KHz ultrasound generator (Model scientz-IID, Xinzhi Biotechnology Corp., Ningbo, China) equipped with a tapered horn tip (0.6 cm end diameter). The ultrasound power density was set to 74 W/cm^2. The samples were held in a temperature controlled water bath to prevent the temperature from increasing.

Apparent viscosity of the samples was measured using a RVDV-I+ viscometer (Brookfield Engineering Laboratories, USA) at 50 ± 1 °C. Temperature of the samples was controlled by a thermostat water bath. The viscometer was a spindle type with a spring of 0.72 mN-m in torque and a spindle speed of 100 rpm. Viscosity determinations were performed in triplicate.

Starch solubility was determined as described by Schoch with slight modification (Schoch, 1964). The ultrasound treated samples were diluted with hot distilled water until the starch concentration reached 2% (w/w). Then 200 g of the 2% (w/w) GMSDs were transferred into a pre-weighed 250 ml centrifugal bottle and capped and shaken thoroughly. The centrifugal bottle was then dried and centrifuged for 15 min at 1000 × g. After centrifugation, 50 g of the supernatant from the centrifugal bottle was transferred into an Petri dish and dried overnight in a forced air oven at 105 °C. The dried residue was then cooled in a desiccator and weighed for soluble starch. Solubility was calculated with the following equation: (%, w/w) = grams of dried residue/(grams of dry starch) × 100. The determination was triplicated.

All ultrasound treated GMSDs were diluted to 1% (w/w), and stained with I_2/KI solution. Morphological observation of the samples was performed with an Olympus DP12 microscope (Tokyo, Japan) and photographed using a digital camera.

2.3. Film preparation

To investigate the properties of the films formed from GMSDs with relatively low and high concentrations, 5% and 10% (w/w) ultrasound treated GMSDs prepared according to the procedure described above was poured into pre-warmed plastic moulds respectively and dried at 60 °C for approximately 6 h, using a ventilated oven. The fresh films were stored for one week at 50% RH and 25 °C before testing. All films' thicknesses were controlled to 0.070 ± 0.005 mm.

2.4. Film opacity

The film opacity was measured by a spectrophotometer (UV-2550PC, Shimadzu, Kyoto, Japan) and defined as the area under the absorbance spectrum between 400 and 800 nm according to ASTM D 1003-00 method (ASTM, 2000a). The film samples were cut into a rectangular piece (1 cm × 2.5 cm), fixed on the inner side of a 1 cm spectrophotometer cell and the absorbance spectrum recorded. The film opacity determinations were repeated three times.

2.5. Water vapor permeability

The water vapor permeability (WVP) test was based on a modified ASTM E96-00 method (ASTM, 2000b). The film sample (5.4 cm in diameter) was equilibrated at 25 °C and 50% RH for one week, and then placed and sealed on the top of a plastic cell (5 cm in diameter), containing 15 ml distilled water (100% RH). Vaseline was used to prevent the leakage of moisture through seals. The test cells were placed into a desiccator containing 1000 g silica gel at 25 °C. The test cell was weighed at time intervals of 3 h over 24 h. Water vapor permeability (WVP) were calculated as WVP ($\mathrm{g\,m^{-1}\,s^{-1}\,Pa^{-1}}$) = $(\mathrm{d}W \times L)/\mathrm{d}t \times \mathrm{d}P \times A$; where d$W$ (g) is the weight loss of the test cell, L is the film thickness (m), A is the exposed area (m^2) and dt (s) is the time under the partial water vapor pressure gradient (dP = 2533 Pa). The WVP determinations were performed in triplicate.

2.6. Film mechanical properties

Tensile stress and strain at break as evaluated in a tensile test was performed using a texture analyzer TMS-2000 (Food Tech Corp, Virginia, USA) according to ASTM D882-00 (ASTM, 2000c). Before testing, the films were cut into strips (25.4 mm × 80 mm) and conditioned at 50% RH for one week. Force and distance were recorded during extension of the strips at 48 mm/min up to break. Tensile stress and strain at break were calculated. A minimum of five replications of each test sample was run.

2.7. X-ray diffraction

X-ray diffraction patterns were recorded using a D/max-rA Diffractometer (Regaku, Japan) operating at 60 kV and 200 mA with CuKα radiation (λ = 0.1542 nm), in an angular range 3–35° at a speed of 2°/min with a step width of 0.02°. The starch films for test were equilibrated at 25 °C and 50% RH for one week previous to test. The relative crystallinity was calculated as described by Bertuzzi, Armada, and Gottifredi, 2007.

3. Results and discussion

3.1. Characterization of ultrasound treated corn starch dispersion

Fig. 1 shows the effect of ultrasound on the viscosity of GMSD samples with 3%, 5%, 7% and 10% (w/w) GMSDs were treated respectively by power ultrasound (74 W/cm^2) for 0–30 min at 60 °C. The results show that there is a drastic decrease in apparent viscosity of the GMSDs with different concentrations after 15 min, but only a slow decrease or no decrease for the remainder of the time.

It is well known that when a starch based film is produced by casting or spraying, the starch dispersion should be heated first. When starch is heated in the presence of excess water, the starch granules undergo an order to disorder transition, called gelatinization (Lionetto, Maffezzoli, Ottenhof, Farhat, & Mitchell,

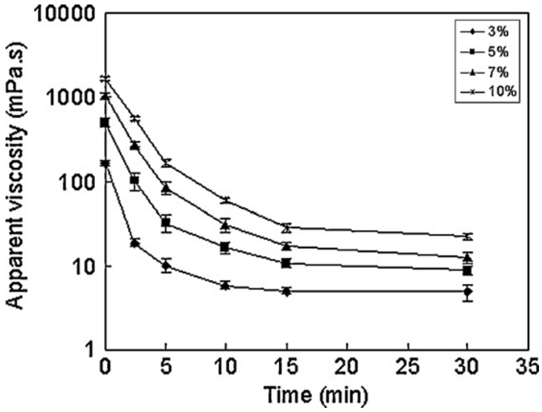

Fig. 1. Effect of power ultrasonic treatment on depression of viscosity of 3%, 5%, 7% and 10% (w/w) GMSDs. Ultrasound treatment condition: power intensity 74 W/cm^2, temperature 60°C.

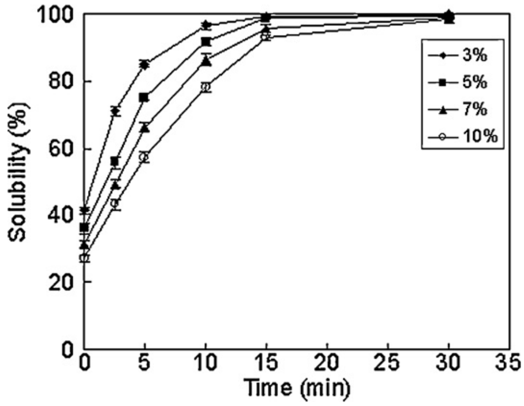

Fig. 3. Effect of power ultrasonic treatment on solubility of 3%, 5%, 7% and 10% (w/w) GMSDs. Ultrasound treatment condition: power intensity 74 W/cm^2, temperature 60°C.

2006). During this procedure, the starch granules adsorb water and begin to swell. After a critical temperature is reached, the water molecules disrupt hydrogen bonding and penetrate the granules, and the starch granules swell. As a result of the irreversible swelling of starch granules, melting of crystallites and leaching of amylose from granules, the starch dispersion undergoes a drastic increase in viscosity (Lagarrigue & Alvarez, 2001; Rao & Tattiyakul, 1999). In practice, for a specific starch at the same gelatinization condition, if the concentration of the film forming starch dispersion is too high, such as above 6 wt%, it is difficult to form a good quality film by casting or spraying. This defect at high starch dispersion concentrations results from drastically elevated viscosities after gelatinization and large number of air bubbles which are hard to expel from the sticky dispersion should be attributed to high concentration of the starch dispersion. In addition, starch dispersions with low concentration require more energy and time for drying and is not economical. This investigation shows that ultrasonicaiton can produce high concentration starch dispersion with low viscosities for film preparation by casting or spraying.

Ultrasound treatment is one of the methods used to depolymerise various biopolymers. Depolymerization of macromolecules occurs due to the high pressure associated with the collapse of the cavitation bubbles which lead to intense heating in residual bubbles and induces drastic mechanical effects. This phenomenon should increase the splitting of the less mobile polymers into smaller fragments (Mason & Lorimer, 1988; Kardos & Luche, 2001). As seen in Fig. 2, for initial GMSD, the non-solubilized portion of the gelatinized granules which are frequently termed “ghosts” is intact, but when treated by power ultrasound, the ghosts are disintegrated gradually. After 15 min treatment, all the ghosts in the experimental maize starch dispersion have disappeared. This behavior could be attributed to the fact that intense mechanical effect and heat induced by power ultrasound break the cross-link between polymers (mainly amylose/amylopectin) which primarily result in robust ghosts (Martine & Michael, 2007). Similar results have been revealed that gelatinized waxy maize starch (Iida et al., 2008) and wheat starch (Seguchi, Higasa, & Mori, 1994) treated by ultrasound can disintegrate robust granular ghosts.

Because of the disintegration of the ghosts in the GMSDs under power ultrasound, polymers responsible for ghost formation should be fully hydrated and solubilized. The following solubility test supports this speculation. Fig. 3 shows that with prolonged ultrasound treatment, the solubility of GMSDs increase quickly due to the rupture of ghosts and increase of free mobile polymer strands, after 15 min, the starch solubility reaches nearly 100%.

3.2. Film opacity

Opacity is a property of prime importance if the film is to be used as a food coating or as a food packaging (Mali, Grossmann, & García, 2004). Fig. 4 shows the opacity of the native and ultrasound treated maize starch films. With prolonged ultrasound treatment, the opacity value of the resulting films formed from 5% to 10% (w/w) GMSDs decrease from 106.6–21.6 AU nm to 116.9–22.3 AU nm respectively. This result indicates that the transmission of light through the resulting films formed from ultrasound treated GMSDs is much better than through native corn starch film. Especially after 15 min ultrasound treatment, the opacity of the films formed from 5% to 10% (w/w) GMSDs is similar to 0.05 mm PE film. This result could be explained by the thorough rupture of starch ghosts and elimination of air bubbles in film-forming dispersions.

3.3. Film water vapor permeability

The rate of water vapor transporting through a starch film is composed of the rates of adsorption, diffusion, and desorption,

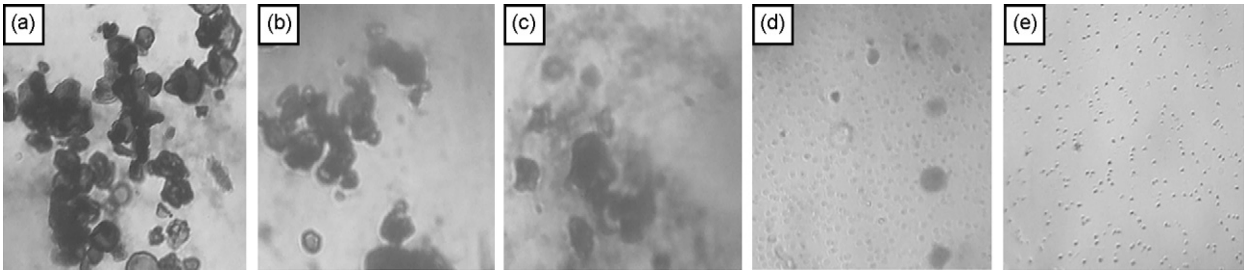

Fig. 2. Morphologies of starch granular structure in dispersion after treated by ultrasound (power intensity 74 W/cm^2, temperature 60°C) for 0 min (a), 2.5 min (b), 5 min (c), 10 min (d), and 15 min (e), observed under a light microscope (100×).

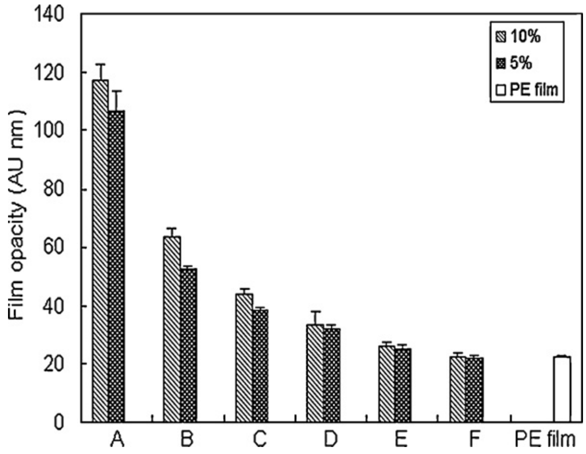

Fig. 4. Opacity of the film formed from 5% to 10% (w/w) GMSDS treated by ultrasound for 0 min (A), 2.5 min (B), 5 min (C), 10 min (D), 15 min (E) and 30 min (F), compared with PE film.

where the water molecules dissolve in one side of the film, move within the void space among polymer segments and then desorb from the polymer surface on the other side of the film. Chemical structure, ratio of amylose to amylopectin, polymer packing, crystallinity, plasticizer, and environmental humidity are all factors related to the permeability of starch films (Chung & Lai, 2007). Generally, the starch film has poor moisture resistance due to its hydrophilic characterization (Petersson & Stading, 2005). However, as seen in Fig. 5, the resulting films formed from ultrasound treated GMSDs has lower WVP. The WVP of the native starch films formed from 5% to 10% maize starch dispersion reach 26.2 and $28.6 \times 10^{-11}\,\mathrm{g\,m^{-1}\,s^{-1}\,Pa^{-1}}$ respectively. Higher concentration of the film-forming dispersion results in higher WVP value ($p < 0.05$). Suggestion for this result is that the film formed from 10% (w/w) starch dispersion is rough due to its film forming difficulty and air bubbles existence. It is hard to spread this dispersion evenly on the mould and to expel the air bubbles in dispersion during film preparation. However, after the maize starch dispersion has been treated by ultrasound for 15 min, WVP value of the films formed from 5% to 10% GMSDs decrease to about $20 \times 10^{-11}\,\mathrm{g\,m^{-1}\,s^{-1}\,Pa^{-1}}$, and concentration of GMSDs has not significant impact on WVP of the resulting films ($p > 0.05$). It seems to imply that the rearrangement of free mobile chains of polymer matrix (mainly amylose and amylopectin) during drying process is favor to form more compact and homogeneous film structure, and previous study has shown more compact structure with less free volume can lower WVP in the case of synthetic polymer films (Miller & Krochta, 1997).

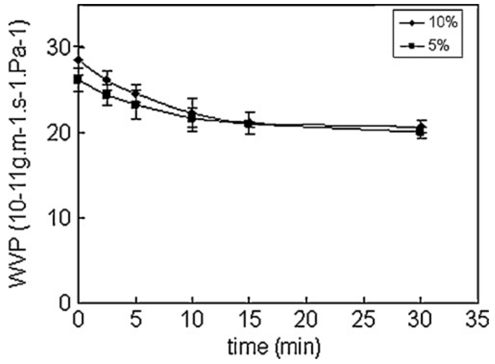

Fig. 5. Water vapor permeability of the film formed from 5% to 10% (w/w) GMSDS treated by ultrasound from 0 min to 30 min.

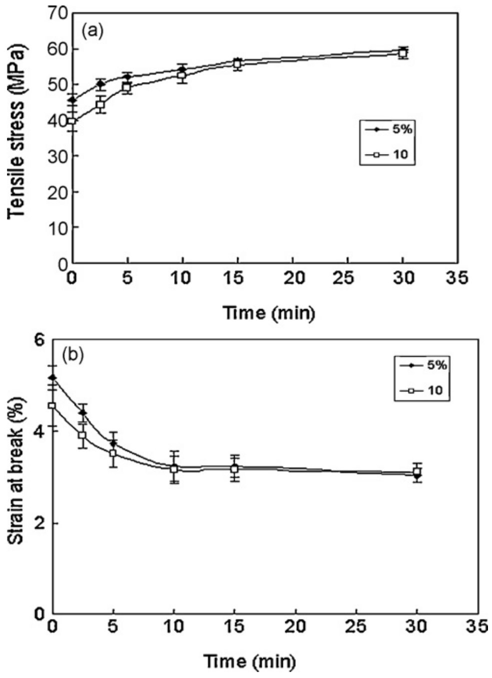

Fig. 6. Tensile stress (a) and strain at break (b) of film formed from 5% to 10% (w/w) GMSDs after treated by ultrasound for 0 min, 2.5 min, 5 min, 10 min, 15 min and 30 min.

3.4. Mechanical properties of starch film formed from ultrasound treated gelatinized maize starch dispersions

To compare the effect of ultrasound treatment on the mechanical properties of the resulting films to untreated ones, in this study, plasticizers were not added. It can be seen in Fig. 6(a), tensile stress of maize starch based films is affected remarkably by ultrasound treatment procedure previous to casting. With prolonged ultrasound treatment, tensile stress of the films increases. After 30 min ultrasound treatment, the tensile stress achieves about 59.5 MPa for both films made of 5% and 10% (w/w) GMDs. There are about 30.7% and 47.7% enhancement in tensile stress respectively compared with the films formed from 5% to 10% (w/w) GMSDs without ultrasound treatment. It has been discussed above that ultrasound treatment seems to be favorable to construct more compact and tight film and thus improve its moisture resistance. This improved film structure also is probably an important factor for tensile stress enhancement. Another factor for tensile stress enhancement may be attributed to the destruction of the ghost and thus releasing of amylose after ultrasound treatment, because increasing amylose content in casting starch solution gives stronger films (Tharanathan, 2003).

However, as a result of more compact structure due to high intermolecular hydrogen bond between polymers, the film formed by ultrasound treatment before casting is more brittle than the original film. As can be seen in Fig. 6(b), after 30 min ultrasound treatment, strain at break decrease from the initial 5.2% and 4.6% to about 3.0% respectively for film formed from 5% to 10% (w/w) GMDs.

3.5. X-ray diffraction

Diffraction patterns recorded can be explained by the superposition of different kinds of crystals: crystallites ‘A’ and ‘B’ formed by an aggregation of amylose chains with different double helical conformations and ‘V’ crystals formed by amylose–lipid complexes

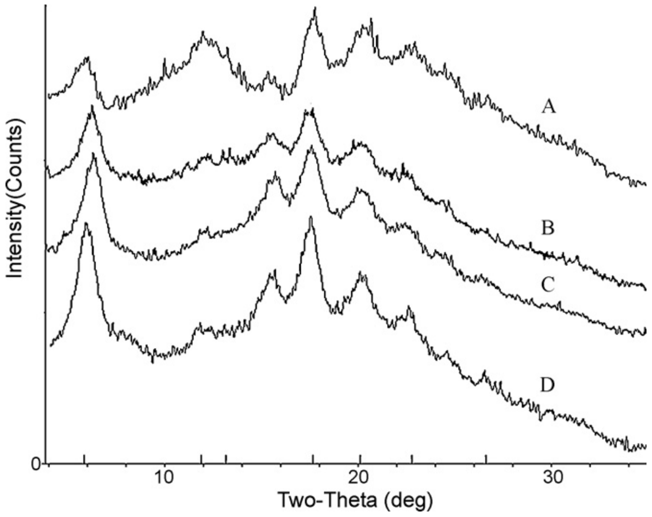

Fig. 7. X-ray diffraction patterns of starch based films formed from native and ultrasonic treated GMDs (10%, w/w). Letters: A–D represent ultrasound treatment time: 0 min, 5 min, 15 min and 30 min respectively.

units. Starch based film crystallinity depends on grade of amylose dissolution and amount of hydrogen bond formed within the film (Chen & Jane, 1994; Liu & Han, 2005).

As can be seen in Fig. 7, ultrasound treatment does not change the crystallites type of the films, all experimental films with different ultrasound treatment times (0, 10, 15, and 30 min) are formed “B” type crystalline structure, but an distinct increase in peak intensity and a slight decrease in peak width with prolonged ultrasound treatment can be observed at $2\theta = 5.6^\circ$, and a slight increase in peak intensity also can be observed at $2\theta = 15^\circ$. A tendency to increase crystallinity as ultrasound treatment time increases is shown in this study. Previous investigation has revealed that film crystallinity is highly related to chain mobility. Preparation procedures that enhance starch solubilization and chain mobility will improve resulting film properties (Bertuzzi et al., 2007). It suggests that high intensity ultrasound treatment on GMDs is in favor of forming films with excellent properties.

4. Conclusions

In this study, power high intensity ultrasound was used to treat GMSDs. The results show this treatment can effectively rupture the swelling starch granules and ghosts in the dispersions, increase solubilized amylose amounts and enhance free mobility of polymers in the starch dispersions, resulting in a drastic decrease in apparent viscosity and increase in solubility even for 10% starch dispersion. These behaviors of the ultrasound treated film-forming dispersions are in favor of film casting or spraying even in high level of concentration. Ultrasound treated gelatinized maize starch dispersions can achieve desirable resulting films with good transparency, improved moisture resistance, and stonger structure. The results also show the resulting films with ultrasound treatment lead to a decrease in strain at break, this defect may be overcome by the addition of some plasticizers such as glycerol. More further investigations are still need to be made in this field.

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