Original Article

Effect of ultrasonication duration of polyvinyl alcohol (PVA) gel on characterizations of PVA film

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Many works reported a PVA based film prepared using ultrasonication, however, information on the effect of this treatment on changes of PVA’s properties is still limited. The main objective of this work was to study the effect of ultrasonication duration on the properties of PVA film. The PVA gel was sonicated using a 360 W ultrasonic probe for 2.5, 5, 7.5 and 10 min. Ultrasonication duration for 7.5 min results in a significant effect (p ≤ 0.05) on an increase in tensile strength, but not on tensile modulus and strain at the break of the film. After this vibration duration, tensile strength increased by almost 29 %, strain at break decreased by 30 %, opacity decreased by 22 %, and water vapor permeability decreased by 11 %. On the other hand, moisture resistance decreased significantly (almost 12 %). The sonicated film underwent larger lattice strain and had higher crystal structure compared to non-sonicated film. This work informs that ultrasonication on PVA gel is a potential method to fulfil some properties of PVA film for food packaging material.

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1. Introduction

Poly-vinyl alcohol (PVA) is an attractive material for bioplastics due to its water-solubility and biodegradability [1,2]. PVA is a common bioplastic with excellent characterizations including high oil and chemical resistance, and stable in almost all organic solvents. Also, PVA is suitable for paper adhesives material, and packaging application [3]. However, this material has high moisture absorption due to the presence of many hydroxyl groups [3]. Attempt to reduce this weakness is the mixing PVA with filler more hydrophobic than PVA. Last studies have reported that PVA based composite displays decreased moisture absorption after adding pineapple leaf fiber [4], and bacterial cellulose nanofiber [5] to pure PVA. Moreover, better homogeneity of this filler in PVA matrix leads to improve the moisture resistance of the composites [6]. This result can be obtained using ultrasonication method. Ultrasonic equipment uses sound energy to create acoustic

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capitation of air bubbles in a gelatinized PVA [7]. On collapsing, the bubbles produce microjets and shock waves, which can disperse filler in the PVA matrix [8,9]. Many researchers have introduced better dispersion of the filler in PVA based composite film prepared via ultrasonication, however, their findings did not characterize the mechanical and physical properties of pure PVA film [5,10,11]. According to last studies, the ultrasonication disrupted the polymer structure in the starch leading to change in its characterization including tensile, and thermal properties [12–15]. Although many studies have reported the use of ultrasonication for preparation of PVA based film, however, no comprehensive information was given regarding the changes in the structure and characterizations of the pure PVA after ultrasonication [3,6,10,16]. Therefore, it becomes more important to know how much effect of the ultrasonic treatment on changes in neat PVA properties. This study addresses this lack by measuring the effect of ultrasonication of the PVA gel on is to further study the properties of the PVA film resulting from ultrasonication of the PVA gel on opacity, TGA/DTG, DSC, XRD, FTIR, tensile properties, water vapor permeability, and moisture absorption. Also, SEM photographs are carried out to observe the fracture surface of tensile samples.

2. Materials and methods

2.1. Materials

PVA (type 2488, degree of polymerization 2400, and the minimum degree of alcoholization 86 %) was purchased from products Jiangxi Jiang Wei Hi-Tech Co. Ltd (China). PVA was vacuum-dried at 50 °C for 24 h before use.

2.2. Preparation of PVA films and viscosity measurement

10 g PVA powder was dissolved in 100 g distilled water using a hot plate magnetic stirrer (Scilogex MS-H280-Pro) at 80 °C and 500 rpm for 2 h until gelatinized. The PVA gel was treated ultrasonically at 360 W for 2.5, 5, 7.5 and 10 min using an SJIA 1200 W ultrasonic probe (20 mm diameter) dipped it in the gel for 10 mm. Part of the non- and sonicated PVA gel was separated to measure its viscosity using a NDJ-8S Digital Rotary Viscometer at 60 rpm for 12 s. Another portion of this gel was poured onto a petri dish (145 mm diameter) that was dried in a vacuum drying oven (DZF-6000 Vacuum Dryer) at 50 °C for 20 h. Each PVA film is labelled with ultrasonic duration. For example, PVA-0 and PVA-2.5 are a film without and with sonication for 2.5 min.

2.3. Characterization

2.3.1. Scanning electron microscopy (SEM)
The fracture surface of films from tensile samples was observed with a Hitachi 3400 N series SEM with an accelerating current of 10 kV and probe current of 8 mA to optimize observation of the surface morphology of the sample. The sheet was coated with carbon followed by gold for 60 s.

Fig. 1 – Schematic description of the crystallinity index estimation.

2.3.2. Opacity of film

A spectrophotometer (Shimadzu UV 1800, Japan) was used for determining the opacity of the film. All films were cut into 10 cm × 25 mm rectangles and placed in a spectrophotometer. ASTM D 1003-00 [Standard test method for haze and luminous transmittance of transparent plastics] was used as a testing standard. The area under the absorbance spectrum between 400 nm and 800 nm was determined as the opacity value of films. The process was repeated three times for each film.

2.3.3. Fourier transform infrared (FTIR)

An FTIR spectrometer (Frontier, Perkin Elmer, USA) was used to characterize FTIR spectra of films. The scanning was performed within the wavenumber range of 4000–600 cm−1 with 4 cm−1 resolution.

2.3.4. X-ray diffraction (XRD)

X-ray diffraction patterns of PVA films were recorded using a PANanalytical Xpert Pro diffractometer with Cu Kα radiation at 40 kV and 30 mA. Samples were scanned between 2θ = 10° to 100°. The crystallinity index (CI) percentage was measured using Eq. 1:

\[
CI \% = \left( \frac{I_T - I_A}{I_T} \right) \times 100
\]

where \( I_T \) is the intensity of the main peak at about 19.5° and 40.8° of a lattice plane, and \( I_A \) is the lowest intensity at either side of a crystallinity peak as shown simply in Fig. 1.

2.3.5. Thermogravimetric (TG) and Differential Thermogravimetric (DTG) analyzing, and differential scanning calorimetry (DSC)

This method is similar to the one reported in more detail in a previous study [17]. The thermal properties of the samples including TGA and DTG were measured using a DTG-60 instrument, Shimadzu (Japan), from room temperature up to 575 °C. The heating rate was 20 °C/min with 50 mL/min nitrogen flow. DSC of all studied samples was carried out using TA
2.3.6. Tensile test
The procedure for tensile testing was similar to a previous study [18]. Tensile testing was performed using a Com-Ten testing machine 95 T with tensile speed for 3 mm/min at room temperature to measure tensile strength (TS), tensile modulus (TM) and elongation at break (EB). The films were cut into 10 cm × 1.5 cm rectangles according to the ASTM D882-12 standard [19]. Before the test, samples were conditioned for 48 h under 50 ± 5 % relative humidity at 25 °C. Tensile tests were repeated five times for each sample.

2.3.7. Moisture Absorption and water vapor permeability
Moisture absorption (MA) was measured in a similar way to that in a previous study [20]. PVA films were cut into 1 cm × 1.5 cm rectangular pieces and dried in a ventilated oven (Universal Oven Memmert UN-55) until constant weight. Saturated NaCl solution was placed in a closed chamber to obtain 75 % relative humidity (RH). The dried samples were placed in the closed chamber at 25 °C for 8 h and weighed using a precision balance to the nearest 0.1 mg (Kenko) every 30 min. Moisture absorption was calculated using Eq. 2:

\[
\text{Moisture absorption (\%)} = \frac{w_t - w_0}{w_0} \times 100
\]

where \(w_0\) is initial weight and \(w_t\) is final weight of the sample.
Water vapor permeability (WVP) method was similar to the method described by previous work with modifications [21]. PVA film was cut to 4.5 cm diameter and conditioned in desiccators at RH 50 ± 5% and 24.5 °C for 1 week. Film samples were placed over the top of a plastic bottle (diameter 4.5 cm) filled with 50 mL of distilled water (RH 100%). To prevent leakage of the moisture around the edge of the sample, the edges of the sample were sealed to the top of the bottle with a tight-fitting plastic lid with the centre removed. All samples were placed in desiccators with 1000 g silica gel (RH 12%) at room temperature. The samples were weighed every 3 h over 24 h. WVP was calculated by following Eq. 3.

\[
WVP = \frac{dW \times L}{dt \times A} \quad (3)
\]

where \(dW\) = weight loss of the test cell (g), \(L\) = thickness of film (m), \(A\) = exposed area (m²), \(dt\) = time (s) under partial water vapor gradient (dP = 2533 Pa). WVP determination was repeated 3 times for each film.

2.3.8. Statistical analysis
IBM SPSS Statistics 25.0 (IBM Corporation, Chicago, USA) was used for analyzing the experimental data. The effect of various ultrasonication treatments on properties of the films was identified using analysis of variance (ANOVA) and P-test. Detection of differences amongst the mean values of the film properties was based on Tukey’s multiple range test using a 95% confidence level \((p \leq 0.05)\). The measurements were replicated at least three times for each sample tested.

3. Results and discussion

3.1. SEM morphology
SEM morphology of fracture surface from the tensile testing sample of non-sonicated and sonicated PVA film is displayed in Fig. 2. PVA-0 has smoother fracture surface (Fig. 2a) than 2.5 min sonicated PVA (Fig. 2b). This is because a relatively unobstructed crack propagation passing through PVA-0 film is present. As PVA gel was sonicated the polymer structure became more compact [15]. Consequently, this result leads to crooked cracks through the weaker parts of the film. This longer tortuous path of the crack due to the more compact polymer structure yields beach marks (marked with the red arrow in Fig. 2c, d) on the fracture surface. This result is in agreement with a previous study showing more fractions of these beach marks on the surface of PVA based film after addition of nanocellulose [22].

3.2. Viscosity of PVA gel
The viscosity of PVA gel after ultrasonication is lower than that before ultrasonication as shown in Table 1. A significant drop of the viscosity is present after 5 min ultrasonication. In this case, each step of this sonicated treatment for 2.5, and 5 min drops a significant viscosity \((p < 0.05)\) for 39, 51% respectively. Further sonicated treatment decreased in slight viscosity. The decrease in this viscosity is because due to depolymerisation and decreased the molecular weight of PVA chains [23]. The shorter PVA chains are free more mobile that can restructure their chains themselves during the drying process, so resulting in more compact and homogenous polymer structure after ultrasonication [21]. The depolymerized PVA chains due to ultrasonication also have a higher amount of free hydroxyl groups [24]. A similar finding was also reported by last works that viscosity of PVA decreased after ultrasonication [25].

3.3. Opacity of the film
Fig. 3 displays opacity for the non-sonicated and sonicated film. Each film had various opacity value with various ultrasonic durations (Table 1). Ultrasonication results in decreases

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**Table 1 – The crystallinity index (CI) of the film calculated from XRD curve (Fig. 5), Opacity value (Fig. 3), Transmittance (T) ratio 1141/1095 cm⁻¹ from FTIR curve (Fig. 4b), and viscosity value.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>CI (%) at 2θ</th>
<th>Opacity (AU/mm)</th>
<th>T ratio of 1141/1095 (%)</th>
<th>Viscosity (mPa.s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>19.5°</td>
<td>42°</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PVA-0</td>
<td>61.4</td>
<td>26.7</td>
<td>22.36 ± 0.0</td>
<td>413.4 ± 7.2</td>
</tr>
<tr>
<td>PVA-2.5</td>
<td>60.7</td>
<td>28.7</td>
<td>21.73 ± 0.0</td>
<td>253.9 ± 7.5</td>
</tr>
<tr>
<td>PVA-5</td>
<td>64.3</td>
<td>27.1</td>
<td>26.09 ± 0.0</td>
<td>201.9 ± 8.1</td>
</tr>
<tr>
<td>PVA-7.5</td>
<td>62.0</td>
<td>29.6</td>
<td>17.44 ± 0.0</td>
<td>191.7 ± 3.2</td>
</tr>
<tr>
<td>PVA-10</td>
<td>63.2</td>
<td>27.1</td>
<td>22.87 ± 0.0</td>
<td>185.3 ± 4.3</td>
</tr>
</tbody>
</table>

*a Different subscripts \(^{a,b,c,d,e}\) in the same column indicate significant differences at \(p < 0.05\).
and increases in the opacity which can be attributed to the various refractive indexes of the film resulting from varied crystal orientation [26]. 5 min ultrasonication of PVA gel led to the lowest PVA transparency. The lowest opacity (17.44 AUmm) was obtained on PVA-7.5 with 22 % lower than PVA-0. This is attributable to the fraction of the crystals for PVA-7.5 was higher than in PVA-0 and the crystalline segments probably better aligned with respect to each other in comparison to PVA-0. The more aligned crystalline parts made the light easier to pass through thus the increase in the transparency of the film [27]. Sonication may result in a polycrystalline structure consisting of many crystalline segments that are randomly oriented in each other. The increasing number of crystalline segments will obstruct light passing through the film by scattering and reflections at the crystal boundaries, resulting in a reduction in transparency.

### 3.4. FTIR curves

Fig. 4 displays FTIR functional groups of the non-sonicated and sonicated films. All samples show a similar pattern. Ultrasonication did not cause the appearance of new functional groups but changed to peak position and intensity of PVA functional groups. Bands at about 3300, 2926, 1721, 1373, 1252, 1082, 838 cm$^{-1}$ correspond to O–H stretching, C–H stretching, C–O bonds, the C–H bending, the O–H in-plane bending, C–O stretching vibrations, deformation vibrations of C–H bonds respectively [4,28,29]. All the peak intensity of these bands after ultrasonication became stronger (lower (transmittance (T) value). The most significant increase was measured on the 2.5 min sonicated film. The T value at the peak of the O–H stretching for PVA-0 was 6.58 % shifted to 3.70 % in this time (Fig. 4a). This 30 % lower value for T corresponds to more hydrophilic film as a result of an increase in the number of hydroxyl groups [30,31]. This is in agreement with the highest moisture absorption of the PVA-2.5 as shown in Fig. 8a. The shorter chains after ultrasonication have more free hydroxyl groups than the long chains before de-polymerization hence the increase in the intensity of O–H stretching. For further ultrasonic treatments, the vibration intensity of all functional groups became weaker. The T value of O–H stretching for PVA-7.5 was 19.5 % higher than for PVA-2.5 (3.7 %) resulting from decreasing the number of free hydroxyl groups after the interconnection of the polymer chains via the hydrogen bonds [5,24]. This bonding was observed on a shift of the peak position from 3295 (PVA-2.5) to 3302 cm$^{-1}$ (PVA-7.5) [17,32]. This is consistent with PVA-7.5 being less moisture absorbent than PVA-2.5 (Fig. 8a) and PVA-7.5 which has a higher crystallinity index than PVA-2.5 (Table 1). The change in the crystalline structure of the PVA after various ultrasonic treatments can be also observed from the transmittance ratio of peaks at 1141/1095 which corresponds to the ratio between the crystalline and amorphous fraction in PVA [5]. Obvious shifts in both these transmittance peaks can be seen in Fig. 4b which shows the change in the FTIR pattern for various durations of ultrasonication. The crystalline/amorphous ratio increased by 43 % in the first 2.5 min of ultrasonication (Table 1). But, further sonication caused much smaller increases in the crystalline fraction: the ratio shifted 3 % from 22.6 % (PVA-2.5) to 23.3 % (PVA-7.5). This is in agreement with the result in Fig. 5.

### 3.5. XRD pattern

Fig. 5 shows X-ray diffraction patterns without and with ultrasonic treatments. All samples show a similar pattern. The main peak of the non-sonicated film was observed at 2θ = 19.5° corresponding to the (101) crystal plane which infers its semi-crystalline nature [33]. This is characteristic of pure PVA [5,32]. The minor peak appeared at 40.8° pertaining to (111) crystal plane [34,35]. The less intense peak was present at 23.1° which corresponds to (200) lattice planes [36]. After ultrasonication, the PVA film shows the higher crystalline structure. The crystallinity index (CI) for PVA-0 was 26.7 % which increased to 29.6
% for PVA-7.5 as a result of the intermolecular hydrogen bonding between polymers resulting in a more orderly and tightly packed structure. This result is consistent with T 1141/1095 ratio in the FTIR curve showing higher T ratio in the 7.5 sonicated film (23.3 %) than that in the non-sonicated film (15.8 %). Ultrasound treatment caused the deformation of the (101) lattice plane as shown by shifting the peak position (see inset) from $2\theta = 19.5^\circ$ (PVA-0) to $2\theta = 19.7^\circ$ (PVA-7.5) [37]. This phenomenon was also observed on the (111) lattice plane with peak position which shifts from $2\theta = 40.5^\circ$ (PVA-0) to 40.9$^\circ$ (PVA-10). Diffraction pattern at this (111) plane became sharper after ultrasonication resulting from an increase in the crystalline structure. A shift of the peak position toward the higher $2\theta$ value was believed as the presence of the compressive residual stress [38,39]. On collapsing, the bubbles from ultrasonication produce microjets and shock waves which can compress and reduce the interlayer spacing of the molecular chains, consequently increasing the lattice strain. The PVA new crystalline fractions may be randomly oriented centre aligned with respect to each other via ultrasonication. For longer ultrasonication durations, CI at the (111) lattice plane for the film shows a more consistent increase in comparison to that at the (101) lattice plane ($2\theta = 19.5^\circ$) as shown in Table 1. CI at diffraction of $2\theta = 40.8^\circ$ for PVA-2.5 increased by 7.49 % compared to PVA-0, but CI at $2\theta = 19.5^\circ$ for PVA-0 was 61.4 %, higher than for PVA-2.5 (60.7 %). This more consistent increase may be attributed to the amount of the chain fraction in the (111) lattice plane for recrystallization being less than that in the (101) lattice plane. Thus the polymer chains at (111) lattice plane are probably more quickly ordered through packing together than at (101) lattice plane. This result is in good agreement with our previous study of the CI of a PVA/starch blend without and with ultrasonication [5].

3.6. Thermal analyzing

Fig. 6 shows TG, DTG, and DSC curves of non-sonicated and sonicated PVA films. All the samples show similar patterns with three steps of weight loss as a function of the increasing temperature as shown in Fig. 6(a). The first step, a slight decrease of the weight loss at 75–160$^\circ$ C corresponds to evaporation of absorbed water [40,41]. The second step (260–480$^\circ$ C), the large weight loss is attributed to the volatile decomposition of the PVA film [14]. A third weight loss represents a final decomposition to ash [42]. Fig. 6(b) shows that ultrasound reduces the thermal stability of the PVA film. This is evidenced by the maximum rate of weight loss (0.56 mg/°C at 390 °C) for PVA-0 being lower than that for PVA-2.5 (0.61 mg/°C at 367 °C). The decrease in this thermal stability in the sonicated PVA is attributed to the higher number of free OH groups and the increased number of short PVA chains as media for heat for transfer [5]. Short PVA chains are more mobile than long chains [24]. These media are increasingly vibrated with increasing temperature leading to weakening the hydrogen bonding among intermolecular chains, thus decreasing in thermal stability of the sonicated PVA chains. This result is consistent with FTIR curve for sonicated PVA film which shows higher O–H stretching (Fig. 4a) than the non-sonicated
one. Fig. 6c shows the DSC curves of the films as a function of temperature. The onset of the melting temperature ($T_o$) and the melting point ($T_m$) of the film increase after ultrasonication. This is evidenced by $T_o$ and $T_m$ for PVA-0 film are 194 and 196 °C respectively which are lower than that for PVA-7.5 film (199, and 199 °C). These shifts are probably due to more compact structure and increasing the crystal structure of the film [17]. This result is also confirmed by PVA-7.5 film’s higher crystallinity index (Fig. 5).

### 3.7 Tensile properties

Fig. 7 shows the average values of the tensile strength (TS), tensile modulus (TM), and elongation at break (EB) of the non-sonicated and sonicated films. After ultrasonication for 2.5 min, TS was 22.44 MPa higher than PVA-0 (19.80 MPa), an increase of 13.13 %. The increase in TS for PVA-2.5 is because of the increase in the crystallinity fraction of the PVA (see Table 1 at $\theta = 42^\circ$) and the lattice strain. The higher CI led to the decrease in the mobility of the PVA chains due to strong hydrogen bonding of the intermolecular interaction of the PVA chains. After 7.5 min ultrasonication, TS reaches a maximum value of 25.50 MPa, 28.79 % higher than TS for PVA-0. This
highest value corresponds to the highest CI of PVA-7.5 (29.6 \% at 29 \textdegree C). The presence of the compressive residual stress (Fig. 5) contributes to improving the tensile properties [38]. The external tensile stress must overcome the compressive residual (negative tensile) stress before the crack tips experience sufficient tensile stress to propagate. Meanwhile, EB increases after ultrasonication despite increasing tensile strength. A possible reason for this is the increase in the slippage between crystalline planes due to the applied shear stress breaking the weaker intermolecular hydrogen bonding between different crystalline segments and aligning the disordered polymer chains.

3.8. Moisture absorption and water vapor permeability

Fig. 8 displays average MA (a) and WVP (b) values of the non-sonicated and sonicated films after 8 h at 75 \% RH at room temperature. PVA-0 had the lowest MA (5.97 \%) most certainly due to the fewest number of the free O–H groups. This is in good agreement with this sample having the lowest O–H stretching (highest T value) as shown in Fig. 4. After ultrasonication of shorter durations (2.5 min), moisture resistance of the film decreased. This is consistent with lowering T values at the O–H stretching peak. The MA for PVA-2.5 (8.49 \%) was higher than PVA-0 (5.97 \%). After longer ultrasonic treatment, MA decreased again just as the T value associated with the hydrophilic functional groups did. After 8 h in moisture chamber, MA for PVA-7.5 was 6.75 \%, absorbing 20.55 \% less moisture than PVA-2.5. As has been shown, there are fewer free hydroxyl groups in PVA-7.5 probably due to the strong intermolecular interaction of the PVA chains via intermolecular hydrogen bonding [43]. Water is less able to diffuse through this less hydrophilic film. This result is in agreement with previous studies which show decreasing the moisture absorption of the biocomposite film after ultrasonic treatment [14,44]. Furthermore, WVP of both sonicated and non-sonicated films are shown in Fig. 8b. As expected, ultrasonication decreases water vapor permeability. WVP of the PVA-0 film is 30.9 \% lower than that of PVA-2.5 film after 24 h. The decrease in WVP is because of more compact polymer structure of sonicated PVA film yielding greater resistance for water molecule diffusion through the film. The non-sonicated film having long polymer chains and high amorphous fraction could provide more spaces for gas to pass through in comparison to the sonicated film. In contrast, after ultrasonication, this space is decreased resulting from restructuring the short-chain via intermolecular interaction, and increased structural compatibility resulting in more barriers for gas to diffuse. This result is consistent with higher crystallinity index (Fig. 5), and higher melting temperature (Fig. 6c) of the film after ultrasonication. A similar phenomenon is also in agreement with previous studies that reported that more homogeneous and compact structure of the starch film reduces WVP [15,45].

4. Conclusion

This present study demonstrates that ultrasonication of PVA gel changes significantly the structure and characterizations of the PVA film. The results show that ultrasonication changed the degree of the transparency of the PVA film due to the change in the molecular chain orientation. This mechanical treatment depolymerized the PVA chains. After ultrasonication PVA chains underwent the larger lattice strain and had higher compressive residual stress. Ultrasonication increased the PVA crystallinity index of the film and compatibility of the polymer chain structure. The sonicated PVA film had higher tensile strength and better gas barrier property than non-sonicated film. Even the sonicated film has still high toughness, despite increasing tensile strength. However, this treatment decreases moisture resistance due to the presence of many free OH groups. The weakness of this lower moisture resistance could be reduced using more hydrophobic filler in PVA film prepared with ultrasonication. This work suggests that ultrasonication of PVA gel contributes to meet some necessary properties for food packaging material.

Conflict of interest

The authors have declared no conflict of interest.
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