

Mechanical and Water Vapor Barrier Properties of Bagasse Hemicellulose-based Films

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The film-forming ability of bagasse hemicellulose and its potential for packaging were investigated in terms of its mechanical and water vapor barrier properties. The films were prepared under various hemicellulose concentrations, chitosan/glycerol amounts (based on the amount of hemicellulose), and temperatures. These were subsequently evaluated by measurement of their mechanical and water vapor barrier properties. Bagasse hemicellulose-based films with higher tensile strengths were obtained at higher hemicellulose concentrations. Scanning electron microscope images showed that the bagasse hemicellulose-based films did not have pores less than one micron in size, suggesting compatibility between the hemicelluloses and the other components present in the films. Moreover, the tensile strength, elongation, and water vapor barrier of the film increased by approximately 124%, 115%, and 48%, respectively, when the drying temperature increased from 25 to 55 °C. These results indicate that the bagasse hemicellulose can be used as part of the raw material for films with good barrier and mechanical properties.

Keywords: Bagasse hemicellulose; Film; Chitosan; Tensile strength; Water vapor permeability

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INTRODUCTION

The use of synthetic polymers and plastics in packaging materials can have negative environmental consequences, particularly in densely populated countries such as China. Therefore, there is a strong market demand for the development of safe, eco-friendly materials to replace synthetic polymers (Azahari and Othman 2012; Schmidt *et al.* 2013), especially using renewable agricultural feedstocks. Currently, food packaging films made from renewable materials are receiving a lot of research attention (Farris *et al.* 2014). Several studies have described film products made from materials such as soy protein (Pan *et al.* 2014), sodium alginate (Liu *et al.* 2013), starch (Maran *et al.* 2013), and their derivatives.

Hemicellulose, the second most abundant natural polysaccharide, primarily consists of C5 and C6 sugar monomers, such as D-xylose, D-galactose, D-arabinose, D-glucose, D-mannose, and traces of uronic acids (Scheller and Ulvskov 2010). A number of hemicellulose sources exist, including the pre-hydrolysis liquor of kraft-based dissolving pulp production processes (Liu *et al.* 2011). In the literature, many studies have been devoted to the development of relevant technologies for the utilization of hemicellulose. For example, xylan from hardwood (Hansen *et al.* 2012) has been used in the preparation of packaging films or as a potential film constituent.

However, previous studies have shown that films prepared using purified hemicellulose demonstrated bad film-forming properties or worse barrier properties (Peng

et al. 2011). Therefore, a highly functional hemicellulose-based film could be achieved by mixing it with chitosan. Because chitosan contains hydroxyl and amine groups, it is potentially miscible with hemicellulose because of the formation of hydrogen bonds (Dhall 2013). The objective of this study is to characterize the properties of bagasse hemicellulose films prepared from renewable agriculture waste by co-blending them with a second component of chitosan. Cross-linked hemicellulose-chitosan networks can be established *via* hydrogen bonding when both bagasse hemicellulose and chitosan are used, improving the strength and water vapor barrier properties of the resulting films. Glycerol was added as a plasticizer to obtain flexible films that could be folded and manipulated without breakage. The effects of the hemicellulose concentration and the drying temperature on the mechanical properties and water vapor permeability (WVP) of the films were studied. The morphology of the hemicellulose-based films was investigated by scanning electron microscopy (SEM). This work provides a basis for the preparation of bagasse hemicellulose films as a biodegradable food packaging material.

EXPERIMENTAL

Materials

Bagasse was provided by a factory in Yunnan province China. An alkali extraction method was used with some modifications to extract hemicelluloses from sugarcane bagasse. Extraction was done with 1:5 ratio bagasse to solvent of NaOH. After filtration, the supernatant were adjusted with HCl to pH 5.5. The suspension was filtered, and then the supernatant was added with 4 volumes of ethanol obtain the pellet. Later, the pellets were dried at 50.0 °C (Liu *et al.* 2012).

Hemicellulose analysis showed the following sugar composition (molar percent): 68.73% xylose, 4.93% arabinose, 11.27% glucose, and 14.88% galactose. The weight-average (Mw) of the bagasse hemicellulose dissolved in distillate water, and was determined by Gel permeation chromatography (GPC) in a GPC Jasco LC-Net II/ADC equipped with a photodiode array detector and refractive index detector. It showed that hemicellulose of bagasse had a weight-average molecular weight (MW) of 6087 g/mol. Chitosan (molecular weight of 900,000 g/mol, with a degree of deacetylation of 90.20%), glycerol (98% purity), sodium hydroxide, and anhydrous calcium chloride were purchased from Aladdin Sci., Ltd. (Shanghai, China).

Methods

Hemicellulose-based film preparation

All films were prepared by casting from their film-forming solutions. Various concentrations of hemicellulose (1.6, 2.4, 3.2, 4.0, and 4.8 wt.%) were first prepared in deionized water. Chitosan solutions in 1% acetic acid were also prepared. The hemicellulose and chitosan solutions (chitosan amounts of 20, 25, 30, 35, and 40%, based on the amount of hemicellulose) were then mixed for 15 min at 45 °C using a magnetic stirrer.

The specified amount of glycerol was added to the above solution. The prepared solutions were filtered, and 50 g of each solution was cast onto 150 × 100 mm acrylic plates and dried at the specified temperatures. The dried films were peeled from the casting plates and stored in desiccators. The compositions of all film samples are listed in Table 1.

Table 1. Reaction Conditions for Hemicellulose-Based Films

Samples	Hemicelluloses (wt.%)	Chitosan (%)	Glycerol (%)	Temperature (°C)
A	1.6 to 4.8	35	20	55
B	3.2	20 to 40	20	55
C	3.2	35	0 to 40	55
D	3.2	35	20	25 to 65

Mechanical properties of hemicellulose-based films (tensile strength and elongation)

The mechanical properties of the films were determined according to ASTM standard method D882-97 (ASTM 1999). The film samples were cut into strips 15 mm wide and 80 mm long using a sharp razor blade. Before testing their mechanical properties, the film samples were conditioned at 23 ± 2 °C and $60 \pm 5\%$ relative humidity (RH) in a climate-controlled chamber for 24 h. The tensile strength (TS) was calculated using Eq. 1,

$$TS = F/A \quad (1)$$

where *TS* is the tensile strength in MPa, *F* is the force (N) at the maximum load (F_{\max}), and *A* is the initial cross-sectional area (m^2) of the film specimen. The elongation at break (*E*) was calculated as the ratio of the increase in length relative to the original length, expressed as a percentage.

Water vapor permeability (WVP) of hemicellulose-based films

The water vapor permeability (WVP) was measured according to the modified ASTM E96-95 standard method (ASTM 1995). After 3 to 5 g of dried anhydrous $CaCl_2$ was added to the test cup, the film samples were sealed over the rim of the cup with molten paraffin. The samples were placed in a constant temperature and humidity box at 23 ± 2 °C and $60 \pm 5\%$ relative humidity (RH). The cups were weighed every 12 h for one week until the moisture change of the sample from the previous weighing was less than 5 mg. For each sample, three replicates were tested and the average is reported.

The water vapor transmission rate (WVTR) and water vapor permeability (WVP) of the film were calculated using Eqs. 2 and 3 (Rafieian *et al.* 2014):

$$WVTR = \Delta m / \Delta t \times A \quad (2)$$

$$WVP = WVTR \times L / \Delta p \quad (3)$$

where *WVTR* is expressed in $g/h \cdot m^2$ and *WVP* is expressed in $g \cdot cm/cm^2 \cdot s \cdot Pa$; $\Delta m/\Delta t$ is the rate of water gain in g/h; *A* is the exposed area of the film in m^2 ; *L* is the mean thickness of the film sample in mm; and Δp is the difference between the partial water vapor pressure on the two sides of the film in Pa.

Transparency (TR) of hemicellulose-based films

The film transparency was determined according to the ASTM standard method D1746-96 (ASTM 1997), with some modifications. The film samples were cut into rectangular shapes (15×50 mm), and the transparency was measured using a spectrophotometer (Shimadzu UV-1800, Kyoto, Japan) at 700 nm. Ten replicates of each film were tested, and the average result is reported. The percent transparency was calculated using Eq. 4,

$$TR (\%) = I_t / I_0 \times 100 \quad (4)$$

where I_t is the light intensity with the sample and I_0 is the light intensity without the sample.

Scanning electron microscopy (SEM)

The surfaces and cross-sections of the samples were studied using a scanning electron microscope (FEI Quanta 200, USA) at an acceleration voltage of 20 kV. Prior to visualization, the film samples were sputter-coated with gold in a Polaron SC7620 Mini Sputter Coater. Cross-sectional observations were done on samples previously fractured in liquid nitrogen.

RESULTS AND DISCUSSION

Mechanical Properties of Hemicellulose-based Films

The tensile strength and elongation of the film samples increased with increases in the hemicellulose concentration from 1.6 to 4.0 wt.%, as shown in Fig. 1(a). A further increase in the hemicellulose concentration beyond 4.0 wt.% led to decreases in these mechanical properties. The elongation decreased from 64% to 55% when the hemicellulose concentration was increased from 4.0 to 4.8 wt.%. The decrease in tensile strength may have been due to the decreased interaction between the molecular chains of the film. Pelissari *et al.* (2013) also reported that the composition and interaction components of the film structures are responsible for different densities. The hemicellulose-based films obtained in this study exhibited tensile strengths of 6 to 22 MPa and elongations in the range of 20% to 65%, both of which are moderate according to the classifications of Krochta and de Mulder-Johnston (1997).

Chitosan is known to possess a good film-forming capability and has high molecular weight (Dang and Yoksan 2015). Chitosan can closely associate with hemicellulose through hydrogen bonding, resulting in improved tensile strength and elongation in the studied films, as shown in Fig. 1(b). The tensile strength increased sharply when the chitosan amount increased from 20% to 35%, and the maximum tensile strength (14.37 MPa) was achieved at the chitosan amount of 35%. The presence of the chitosan contributes to the formation of a stronger polymer network (Bonilla *et al.* 2014). When studying the mechanical properties of cellulose films, Shih *et al.* (2009) also found that the addition of chitosan improved the mechanical properties of the resulting cellulose/chitosan films.

It has been reported that the addition of glycerol, propylene glycol, or sorbitol to a film-forming solution can improve its flexibility (Aydinli and Tutas 2000). In this paper, glycerol was added to improve the mechanical properties of hemicellulose-based films. As shown in Fig. 1(c), the elongation of the hemicellulose-based films increased from 22% to 67% when the glycerol amount was increased from 0% to 30%. This can be explained by the formation of polymer-plastic hydrogen bonds because of the presence of polar groups (-OH) in glycerol, which replace some of the polymer-polymer interactions in the films (Brandenburg *et al.* 1993). However, the high hygroscopicity of glycerol resulted in a tensile strength of only 12.26 MPa, which was achieved at the optimal glycerol amount of 30%.

Regarding the mechanical properties of the hemicellulose-based films, it was noted that the drying temperature had a noticeable effect on the tensile strength and elongation of the films, as shown in Fig. 1(d). The maximum tensile strength of 14.26 MPa and elongation of 56% were achieved *via* drying at 55 °C. At higher drying temperatures, more

thermally induced cross-linking reactions can occur. Similar results have been reported by Srinivasa *et al.* (2004) for the drying kinetics of chitosan films. The drying temperature was found to have a noticeable effect on the rates of moisture reduction of the samples and the mechanical properties of the films.

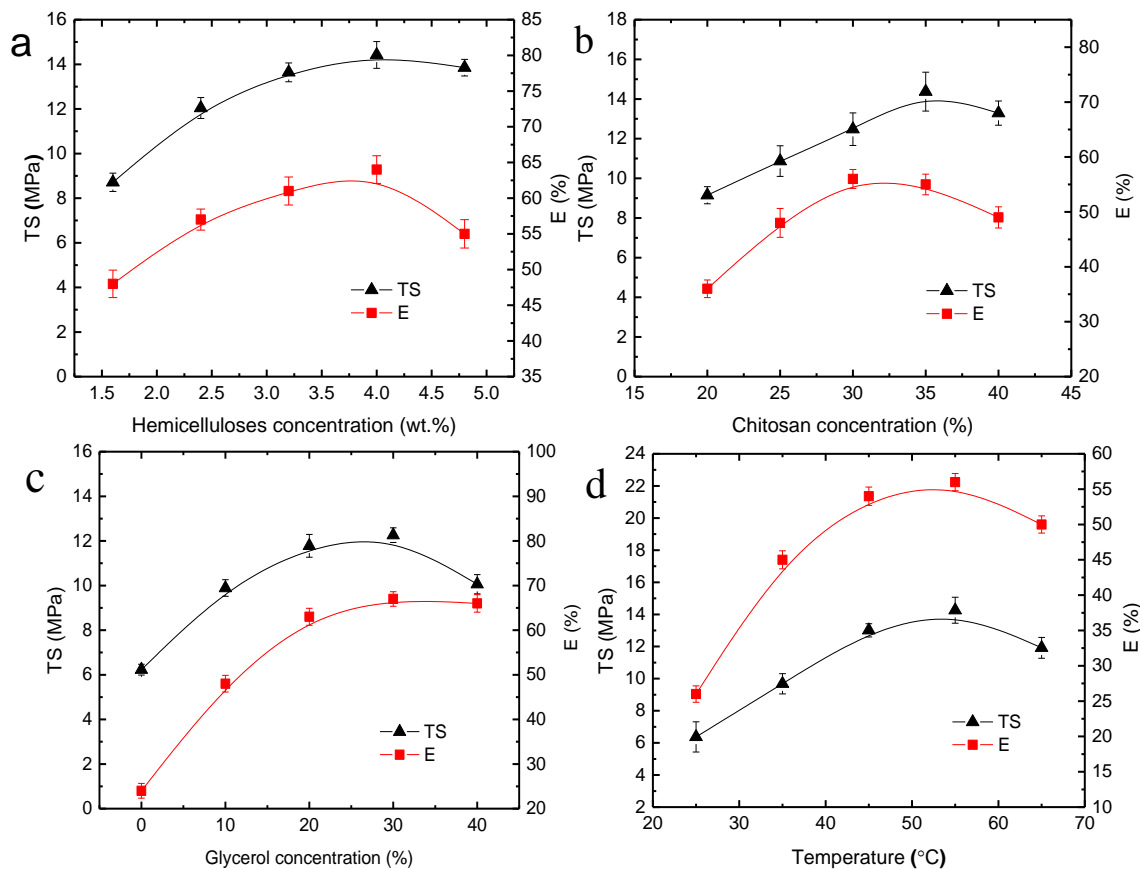


Fig. 1. Tensile strength and elongation as functions of: (a) hemicellulose content, (b) chitosan content, (c) glycerol content, and (d) drying temperature

Water Vapor Permeability (WVP) of Hemicellulose-based Edible Films

As shown in Fig. 2(a), the WVP of the films increased greatly with increasing hemicellulose concentration, reaching $2.08 \text{ g}\cdot\text{cm}/\text{cm}^2\cdot\text{s}\cdot\text{Pa}$ at 4.8% hemicellulose concentration. Higher chitosan amounts also led to a higher WVP, as expressed in Fig. 2(b). These results are related to the hygroscopic nature of hemicellulose and chitosan, which led to a more open matrix. Therefore, the transfer of water molecules through the film network was enhanced and a higher WVP was obtained. Jongjareonrak *et al.* (2005) reported that a higher WVP was obtained for gelatin films containing more protein. Thus, films prepared using higher hemicellulose or chitosan concentrations were more hygroscopic than those containing lower concentrations.

Glycerol molecules are highly hydrophilic in nature. The addition of glycerol as a plasticizer not only increases water absorption into the film matrix but also modifies the structure of the film network by increasing the free volume, thus increasing the diffusion coefficient. As a consequence, increasing the glycerol amount from 10% to 40% increased the WVP of the hemicellulose-based films from 1.45 to $2.62 \text{ g}\cdot\text{cm}/\text{cm}^2\cdot\text{s}\cdot\text{Pa}$, as shown in

Fig. 2(c). In another study, Mali *et al.* (2005) reported that in cassava starch films, the addition of more plasticizer increased the equilibrium moisture content.

As shown in Fig. 2(d), the effect of the drying temperature on the WVP of the hemicellulose-based films was studied across a temperature range of 25 to 65 °C. The WVP of the films decreased from 1.71 to 0.89 g·cm/cm²·s·Pa when the drying temperature was increased from 25 to 55 °C. The results from this study were much lower than those reported by Zhang and Han (2006) for starch films (0.83 to 3.79 g·cm/cm²·s·Pa). It was concluded that an increase in the drying temperature during the preparation of the hemicellulose-based films led to lower WVPs in the films. This was caused by improved polymer arrangement and cohesion within the film matrix, consequently improving the interactions between the polymer chains and facilitating a more compact structure (Miller and Krochta 1997; Orliac *et al.* 2003).

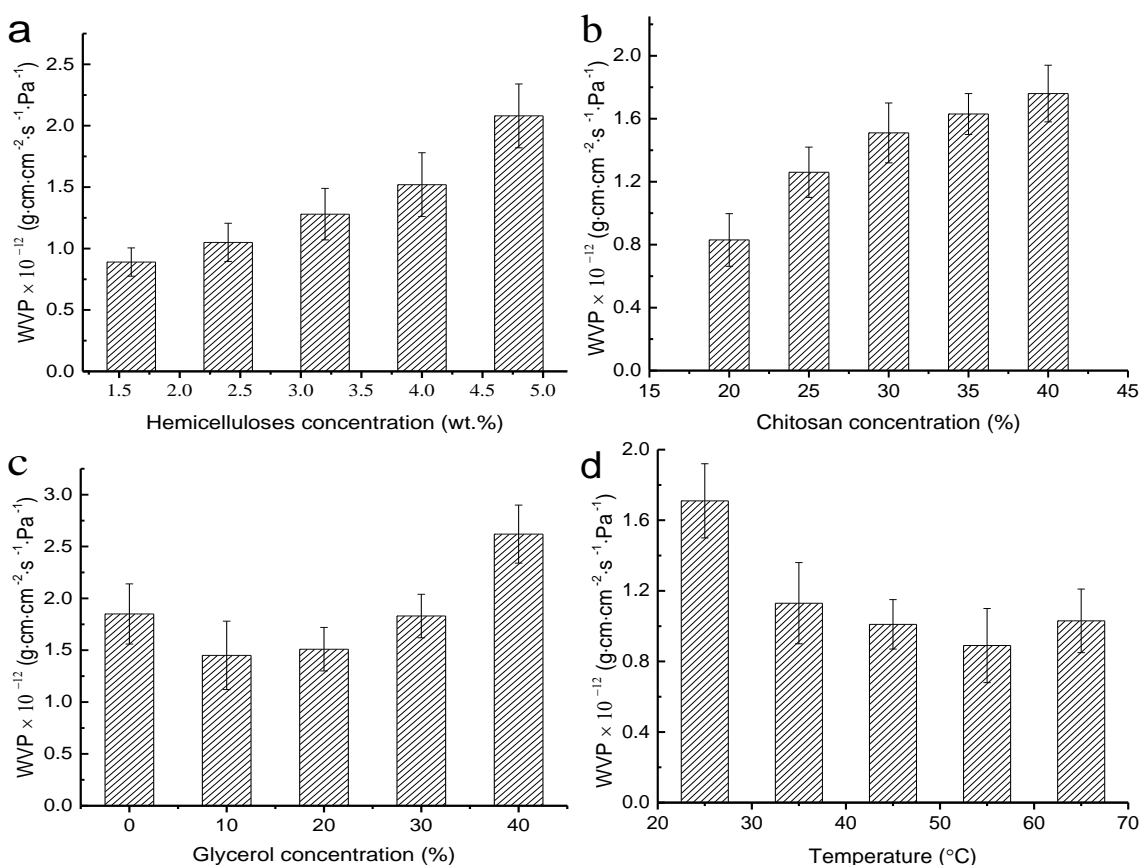


Fig. 2. Water vapor permeability as functions of: (a) hemicellulose content, (b) chitosan content, (c) glycerol content, and (d) drying temperature

Transparency (TR) of Hemicellulose-based Films

The effects of the hemicellulose, chitosan, and glycerol concentrations on the transparency of the composite films were determined, as shown in Fig. 3. The hemicellulose-based films exhibited good transmission (50% to 70%) in the visible light region. Higher transparency is an indicator of good compatibility and interaction among the constituent components (Cerqueira *et al.* 2012). Figure 3(d) shows a higher variation in the transparency (56% to 73%) when the drying temperature was increased from 25 to 55 °C compared with that resulting from changes in the other parameters. This was because

the drying temperature had a greater influence on the structure of the films on account of the formation of more hydrogen bonds or cross-links (Chiou *et al.* 2008), leading to smoother, denser-textured hemicellulose-based films. The above results suggest that hemicellulose-based films with more hydrogen bonds or of greater cross-linking degree are more transparent.

The decrease in film transparency observed in this study with increasing glycerol concentration, seen in Fig. 3(c), was assumed to be due to its plasticizing effect. Small, hydrophilic glycerol molecules readily interact with chitosan chains and hemicelluloses, thus decreasing interactions between these polymer chains (Janjarasskul and Krochta 2010). As a consequence, a less dense, more disorganized polymer matrix is formed at higher glycerol concentrations. In addition, differences in the refractive indices of glycerol and the other film constituents can also lead to decreased film transparency at higher glycerol concentrations. These results are in agreement with those of Limpisophon *et al.* (2009), who reported that gelatin films from blue shark skin at glycerol contents of 25% were more transparent than those prepared at 50% glycerol content.

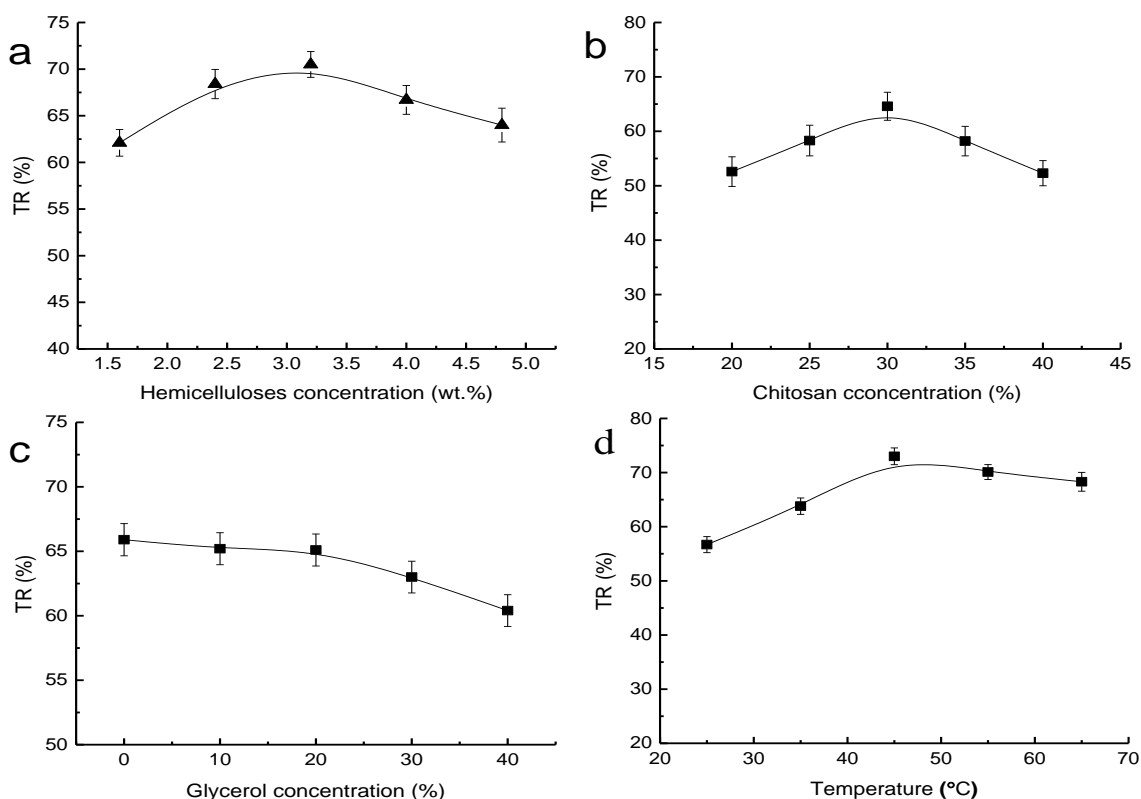


Fig. 3. Transparency of hemicellulose-based films as functions of: (a) hemicellulose content, (b) chitosan content, (c) glycerol content, and (d) drying temperature

SEM External Surface Images of Hemicellulose-based Films

The microscopic and cross-sectional structures of the hemicellulose-based films were examined using SEM. As shown in Fig. 4, the hemicellulose-based film was nonporous and the texture was plain and without any evidence of pores, seen in Figs. 4(a) and (b). In fact, there was no visible evidence of pores larger than 1 μm in size in these films. These results support the conclusion that the hemicellulose and chitosan present in

these films were compatible, thus imparting good mechanical and barrier properties to these films. A study by Wang *et al.* (2014) also showed that soy protein films exhibited a rather homogenous structure.

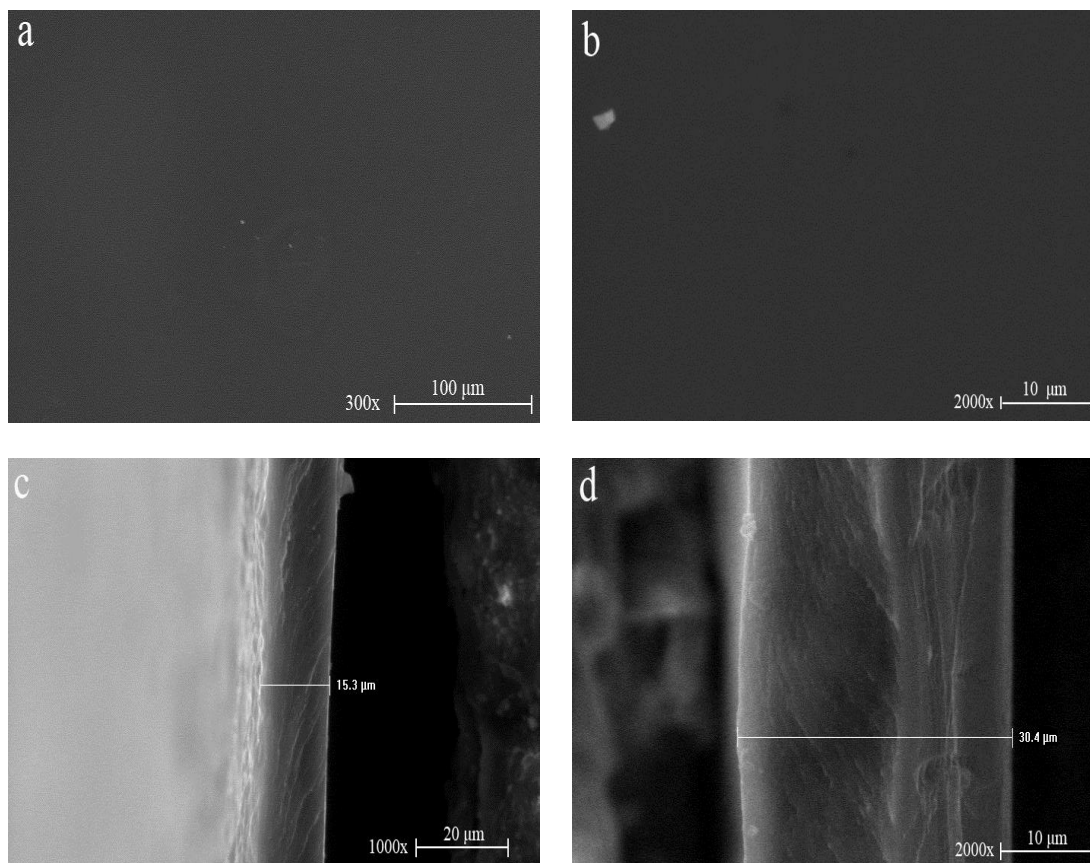


Fig. 4. SEM images of hemicellulose-based films: (a) 4.0wt.% hemicellulose+ 30% chitosan and dried at 55 °C, surface area; (b) 3.2wt.% hemicellulose+ 35% chitosan and dried at 55 °C, surface area; (c) 4.0wt.% hemicellulose+ 30% chitosan and dried at 55 °C, cross-sectional area; (d) 4.0wt.% hemicelluloses+ 35% chitosan and dried at 25 °C, cross-sectional area

CONCLUSIONS

1. Hemicellulose-based films were successfully prepared using hemicellulose from bagasse, together with chitosan and glycerol (as a plasticizer), based on the casting approach, with adequate mechanical and barrier properties.
2. The drying temperature strongly influenced the properties of the resulting films. The tensile strength reached 14.26 MPa and the WVP was decreased by 48% when the drying temperature was 55 °C.
3. The properties of hemicellulose-based films were influenced by chitosan and glycerol concentrations. In particular, the tensile strength and elongation of films were positively influenced by appropriately chitosan and glycerol separately. For WVP, the glycerol concentration had adverse effect influence. With increasing glycerol concentration, a slight decrease in the barrier properties of the films was observed.

4. The SEM images showed that the hemicellulose-based films possessed smooth surfaces and were absent of pores larger than 1 μm in size.

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