

## Water Sorption and Physicomechanical Properties of Corn Starch-Based Films

Kuorwel K. Kuorwel,<sup>1</sup> Marlene J. Cran,<sup>2</sup> Kees Sonneveld,<sup>3</sup> Joseph Miltz,<sup>4</sup> Stephen W. Bigger<sup>1</sup>

<sup>1</sup>School of Engineering and Science, Victoria University, Melbourne, Victoria 8001, Australia

<sup>2</sup>Institute for Sustainability and Innovation, Victoria University, Melbourne, Victoria 8001, Australia

<sup>3</sup>KS PackExpert & Associates, Mansfield, Victoria 3724, Australia

<sup>4</sup>Department of Biotechnology and Food Engineering, Technion-Israel Institute of Technology, Haifa 32000, Israel

Correspondence to: M Cran (E-mail: marlene.cran@vu.edu.au)

**ABSTRACT:** The mechanical performance of thermoplastic starch (TPS) films and films made of TPS blended with an aliphatic polyester (APTPS) was investigated after exposure to water mixtures of various compositions and different levels of relative humidity (RH). Conditioning of TPS films at various RH levels resulted in a noticeable absorption of moisture, particularly at high RH levels, and their mechanical properties decreased significantly. High magnification imaging of the films revealed the presence of significant microcracking on the surface of the TPS films which could potentially result in the increased water absorption and consequent reduction in mechanical integrity. The APTPS film was free of any visible microscopic defects and was also less susceptible to water ingress.

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### INTRODUCTION

Packaging materials manufactured from synthetic, oil-based polymers such as polyolefins do not degrade readily in the environment.<sup>1–3</sup> Conversely, polymers that are chemically synthesized from natural, renewable resources such as starch and others [e.g., poly(lactic acid)] are generally biodegradable or compostable and are more readily decomposed in the environment.<sup>4–6</sup> Bio-based polymers that have the potential to be used for the preparation of packaging films or coatings for food packaging applications include polysaccharides such as starch, alginate, cellulose, chitosan, carageenan, and proteins such as whey protein, corn zein, and their derivatives.<sup>7–10</sup> Starch is a relatively inexpensive, renewable, and natural polysaccharide obtained primarily from cereal grains, potatoes, tapioca, and arrowroot.<sup>11</sup> Starch consists of amylose and amylopectin molecules present at different molecular ratios. Amylose is a linear molecule consisting of glucose units connected by 1,4-glucosidic linkages, whereas amylopectin is a highly branched molecule consisting of short 1,4-glucose chains connected by 1,6-glucosidic linkages.<sup>12</sup> Starch alone cannot be processed into a useful packaging film due to its inherent brittleness and hydrophilic nature, unless it is modified mechanically, physically, or chemically and/or combined with plasticizers.<sup>13,14</sup>

Modified starch-based materials can be manufactured into suitable packaging films by conventional plastic conversion proc-

esses such as compression molding, extrusion, and thermoforming.<sup>15,16</sup> Starch-based materials have the potential to be applied in packaging applications for food products with relatively low water activities ( $a_w$ ) such as biscuits, snacks, and cereals.<sup>11,17,18</sup> Recently, Plantic Technologies (Melbourne, Australia), has developed commercial starch-based materials for packaging applications.<sup>19</sup> Furthermore, Novamont (Nagoya, Japan) and Bioenvelope (Québec, Canada) have manufactured EverCorn™ and Bio-P™, respectively from starch-based materials that are purportedly tolerant to high  $a_w$  levels.<sup>20</sup>

The objective of this study was to investigate the physicomechanical properties of starch-based films as a function of water content and/or relative humidity (RH) to identify the maximum level at which the films can be used practicably for packaging applications. This, in turn, will enable assessment of the suitability of these materials for the packaging of foodstuffs with a given water activity.

### EXPERIMENTAL

#### Materials

The materials used in this study were thermoplastic starch (TPS) which is a chemically modified high amylose corn starch (Gelose 939) supplied by Penford (Sydney, Australia) and a TPS blended with an aliphatic polyester (APTPS), which is a

commercial starch-based film (Biograde-F) supplied by Biograde (Melbourne, Australia). The TPS material has been specifically designed for the production of extruded or thermoformed packages. The APTPS is a commercial biodegradable film used primarily for bags and liners. Analytical reagent grade glycerol was purchased from Merck (Melbourne, Australia).

### Preparation of TPS Film

The preparation of the starch-based film was achieved by heat pressing under compression. Master batches were prepared by gradually adding the starch-based material to a plasticizer made of a mixture of water and glycerol. The final composition of the formulation was 65% (w/w) starch-based material, 10% (w/w) water, and 25% (w/w) glycerol. A sample weighing *ca.* 15 g of the resultant mixture was placed between Mylar™ films that were then positioned between a set of aluminium plates and pressed in a laboratory press (IDM Instruments, Melbourne, Australia, Model No. L0003). The temperature of the upper and lower platens of the press was maintained at 125°C for 5 min under a pressure of 20 kPa. The plates were then quenched, removed from the press, and the films were peeled off the Mylar™ film after cooling was completed.

### Water Sorption Measurements

The water uptake of TPS and APTPS films was measured by (i) directly immersing film pieces in isopropanol-water, ethanol-water, or glycerol-water mixtures and (ii) conditioning film samples at different levels of RH. In the immersion tests, film pieces were directly immersed into mixtures of 100, 90, 80, 70, 60, 50, 40, 30, 20, 10, and 0% (v/v) water in isopropanol, water in ethanol, or water in glycerol for 5 min at room temperature. Samples that remained intact were then removed and dried by wiping the surface before the final mass measurement that was made using an analytical balance. Samples that dissolved partially or completely were excluded. To test the effect of RH, the film samples were contained in desiccators and exposed for 7 days over saturated solutions (in distilled water) of P<sub>2</sub>O<sub>5</sub>, LiCl, CH<sub>3</sub>COOK, MgCl<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub>, Mg(NO<sub>3</sub>)<sub>2</sub>, NaNO<sub>2</sub>, NaCl, KCl, K<sub>2</sub>SO<sub>4</sub>, and pure distilled water. These solutions provided relative humidities of 0, 11, 23, 33, 43, 53, 66, 75, 85, 97, and 100% at 20 ± 1°C.<sup>21</sup> The water content of the equilibrated film samples was determined gravimetrically by firstly weighing the exposed samples and then drying them at 105°C in a laboratory oven for 24 h before reweighing. As the effect of RH on the physicochemical properties of starch-based film is the main concern of this investigation, the amount of water absorbed by the samples is plotted against %RH. Indeed, this is not a typical water sorption isotherm,<sup>22</sup> but it enables a comparison to be made between the data obtained on exposure of the samples to different RH conditions to those data obtained following direct immersion experiments.

### Mechanical Properties of Starch-Based Films

The physicochemical properties of the TPS and APTPS starch-based systems were investigated in accordance with ASTM Method D 882-97. Films were cut into strips of 20 × 100 mm<sup>2</sup>. The measurements were made using an Instron 4465 (Norwood, MA) tensile tester with an R 2797 (500 N) peak load cell and crosshead speed of 50 and 500 mm min<sup>-1</sup> for the TPS and APTPS films, respectively.

### Scanning Electron Microscopy

Micrographs of the APTPS film and TPS film were obtained using a JOEL NeoScope (JCM-5000, Sydney, Australia) scanning electron microscope (SEM). Samples were mounted on an aluminium sample holder and coated with up to 6 nm of gold using a NeoCoater (MP19020NCTR) before obtaining the SEM images. The images were acquired at magnifications of 1700× and 15,000× under high vacuum and using an accelerating voltage of 10 kV.

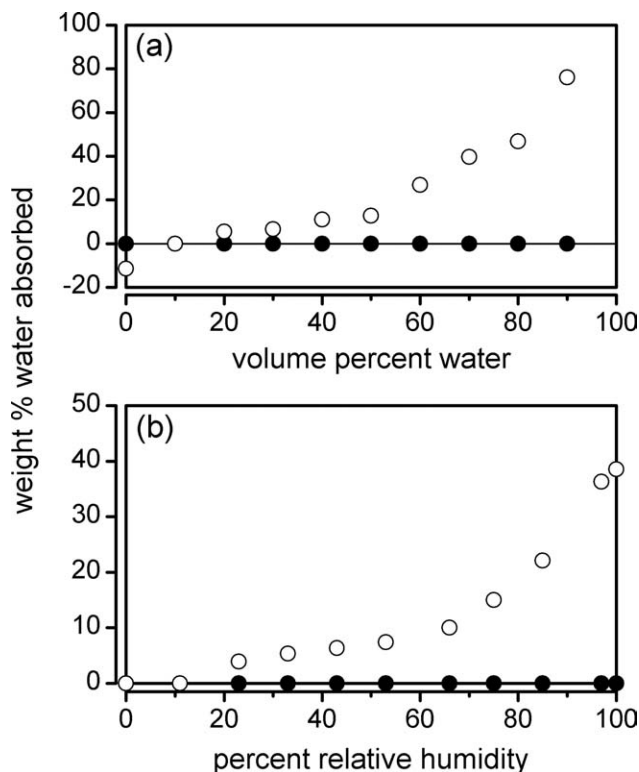
### Data Analysis

Data points represent the mean value of the results obtained for the TPS and APTPS films. Five replicates were taken when making the mechanical property measurements. A total of three replicates were used for the water sorption determination in each of the immersion and RH exposure tests. Data points were subjected to an analysis of variance using the general linear model procedure of the SAS statistical package (SAS version 9.5, SAS Institute, Cary, NC). Differences amongst the results were examined by the least significant differences test at a probability level of  $P = 0.05$ .

## RESULTS AND DISCUSSION

### Water Sorption Measurements

The water uptake of TPS and APTPS films that were directly immersed in isopropanol/water mixtures is shown in Figure 1(a). The TPS film exhibited a significant ( $P \leq 0.05$ ) increase in its absorption of water with increasing water content in this mixture. Films of TPS behaved similarly when immersed in ethanol/water and glycerol/water mixtures (results not shown). The plots in Figure 1(a) indicate that the isotherm obtained for the TPS film is sigmoidal and the water sorption increased steadily with water content in the isopropanol mixture up to *ca.* 40% (v/v) water. When the concentration of water in the mixture was further increased, the TPS material became mechanically unstable and eventually dissolved. The results for TPS films immersed in ethanol/water mixtures also showed a significant water sorption at elevated water content in the mixtures but in such cases there was a steady increase in water content up to *ca.* 20% (v/v) before dissolution of the films became evident. Interestingly, TPS film samples that were immersed in either pure isopropanol or ethanol lost some water, presumably due to water extraction by the isopropanol or ethanol.<sup>23</sup> Furthermore, it appears that the TPS film does not absorb or lose water at a level of *ca.* 10% (v/v) water in the mixture. This corresponds to the amount of water remaining in the film after heat pressing and can therefore be assumed to be equal to the initial water content of the film. The immersion of the TPS films in glycerol/water solutions demonstrated a steady and significant increase in the absorption of water with increasing water content in the mixture up to a level of *ca.* 60% (v/v). When TPS film samples were immersed in pure glycerol, there was no measurable absorption or desorption of water, which is in contrast to the results for isopropanol/water and ethanol/water mixtures. Figure 1(a) reveals that the APTPS film behaves differently to the TPS film in that the former shows little water uptake in any of the isopropanol/water mixtures tested. This behavior was also observed in the case of APTPS films that were immersed in the other mixed solvent systems under investigation in this study.



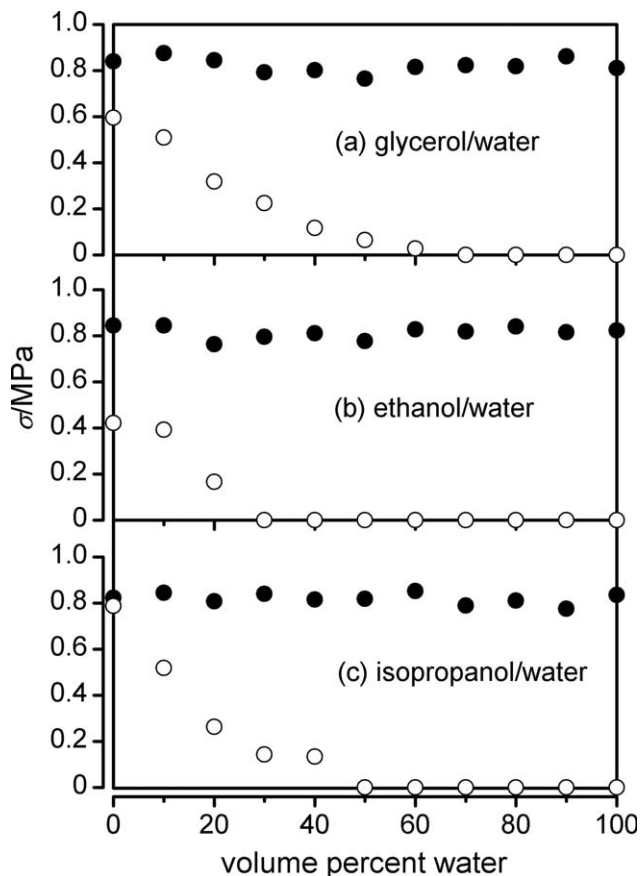
**Figure 1.** Percent water uptake of TPS (○) and APTPS (●) films at  $20 \pm 1^\circ\text{C}$  as a function of (a) isopropanol concentration in the isopropanol/water immersion mixture after 5 min immersion and (b) RH after conditioning for 7 days.

The water uptake of the TPS and APTPS films conditioned at different RH levels is shown in Figure 7(b). The TPS film showed a significant ( $P \leq 0.05$ ) absorption of water with increasing RH. The isotherm obtained for this film exhibits a steady increase in moisture content up to  $\text{RH} \approx 66\%$  with a rapid rise in the moisture absorption beyond this RH level. This water uptake behavior is characteristic of TPS materials and results from their hydrophilic nature.<sup>24,25</sup> A similar trend was observed by Van Soest et al.<sup>26</sup> who reported that absorption and desorption of water by hydrophilic films depend on the RH of the environment. The results of this study are also in agreement with the findings of Talja et al.<sup>27</sup> who reported higher water sorption of starch-based films with increasing RH. The observed RH dependence of the water uptake of TPS films may be attributable to the high percentage of amylose in the cornstarch from which they are comprised. The low moisture uptake of the TPS film at low RH may be due to weak interactions between the water and starch matrix as suggested by Zhang and Han.<sup>28</sup> However, the high moisture uptake exhibited at higher RH by these films is probably due to an increase in hydrogen bonding between the starch, water and plasticizer, resulting in the weakening of the film structure.<sup>28,29</sup> It is interesting to note that the TPS film exposed at 0% RH did not exhibit a loss of water as which might be expected considering the case of TPS samples that were immersed in solvent mixtures containing less than 10% (v/v) water. This may be attributable to the rate at which water can be gained or lost from the substrate when immersed

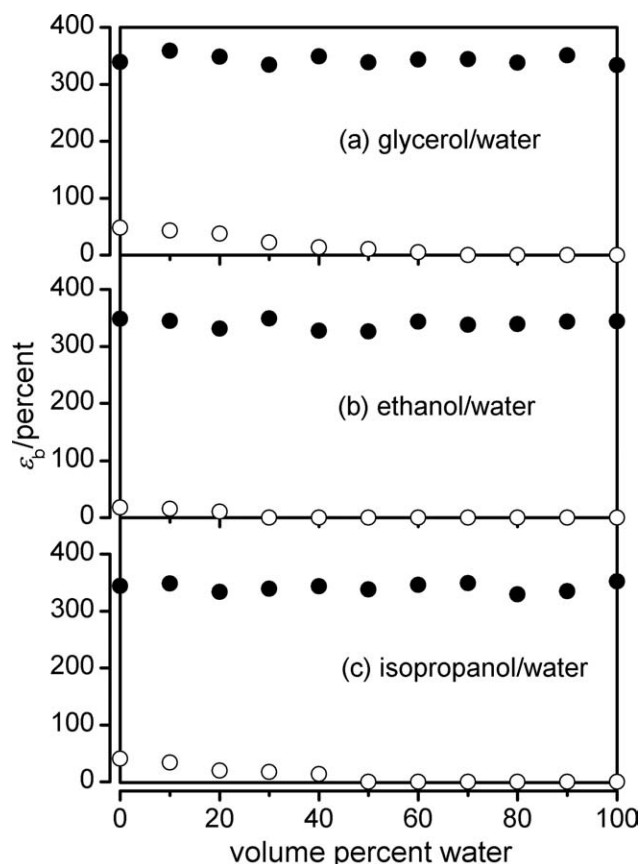
in a condensed mixed solvent medium that can also have a swelling effect on the substrate being greater than the rate attainable in air. In contrast to the TPS film, no measurable water absorption was observed for the APTPS films on their immersion or as a function of RH. These results confirm that the hydrophilicity of starch-based packaging materials can be overcome by their chemical modification and/or blending with other polymers.<sup>30,31</sup>

#### Effect of Water Content on Mechanical Properties

The effect of water content on the ultimate tensile strength ( $\sigma$ ), elongation at break ( $\epsilon_b$ ), and elastic modulus ( $E'$ ) of the TPS and APTPS films was explored by analyzing the stress-strain curves of the films. When immersed for 5 min in glycerol/water, ethanol/water, or isopropanol/water mixtures, all the TPS films demonstrated a considerable decline in their tensile strength with an increase in the water content of the mixtures as shown in Figure 2(a–c), respectively. The tensile strength of the TPS film samples is persistent up to ca. 50% (v/v), 20% (v/v), and 40% (v/v) water in the glycerol/water, ethanol/water, and isopropanol/water systems, respectively. The data obtained for the isopropanol/water systems are comparatively greater than those obtained for the ethanol/water systems presumably as a consequence of the lower polarity of isopropanol compared with



**Figure 2.** Effects of film exposure for 5 min at  $20 \pm 1^\circ\text{C}$  on the ultimate tensile strength,  $\sigma$  of TPS (○) and APTPS (●) films: (a) glycerol/water immersion (b) ethanol/water immersion (c) isopropanol/water immersion and (d) RH conditioning.



**Figure 3.** Effects of film exposure for 5 min on the elongation at break,  $\epsilon_b$ , of TPS (○) and APTPS (●) films: (a) glycerol/water immersion (b) ethanol/water immersion (c) isopropanol/water immersion and (d) RH conditioning.

ethanol and to a lesser extent, the relative molecular sizes.<sup>32</sup> Indeed, the presence of water in the solvent/water mixtures enhances the overall polarity of the mixture, resulting in an increased water uptake. Moreover, the larger isopropanol species are less effective than the smaller ethanol molecules in penetrating the film and extracting water molecules present in the starch structure thus leading to an onset of brittleness. In contrast, the APTPS films show no significant change in tensile strength in any of the solvent mixtures over the concentration ranges studied. This is expected for commercial APTPS films which are essentially nonpolar due to the APTPS and are therefore resistant to the polar solvents.

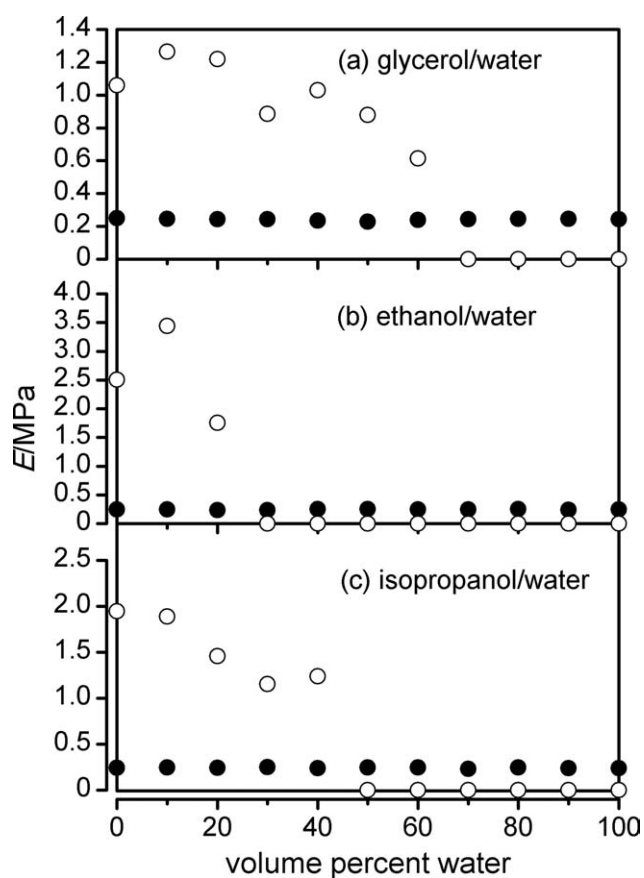
The effect of water on the elongation at break,  $\epsilon_b$ , for the TPS and the APTPS starch-based films is presented in Figure 3. The  $\epsilon_b$  for the TPS film immersed in glycerol/water mixtures decreased with an increase in the amount of water in the mixture up to the water content of *ca.* 50% (v/v). At levels above 50% (v/v), the material had no measurable elongation under the conditions of the test [see Figure 3(a)]. The elongation of the TPS films immersed in the isopropanol/water mixtures exhibited similar behavior to that observed in the glycerol/water mixtures [see Figure 3(c)]. However, the  $\epsilon_b$  values of the TPS films immersed in the ethanol/water mixture severely deteriorated at the lower water content of *ca.* 20% (v/v) [see

Figure 3(b)]. These observations can be explained again by the ability of ethanol and isopropanol to extract water molecules from the TPS matrix thereby rendering it brittle. In contrast, the APTPS film retained its  $\epsilon_b$  over the entire concentration range for each solvent mixture and the magnitudes of the  $\epsilon_b$  values are considerably higher than those of the TPS films, which would be expected for an extruded biaxially orientated film such as APTPS of this composition.

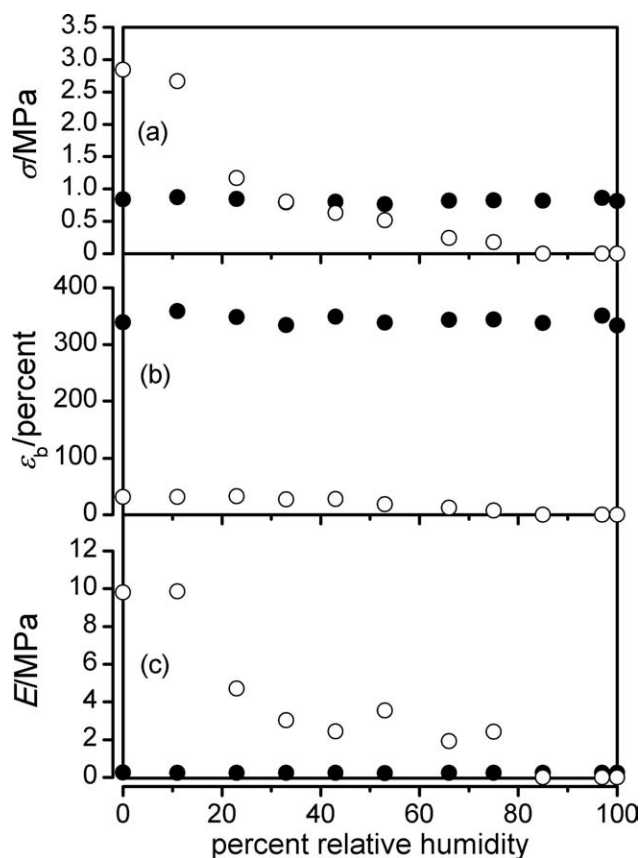
The effect of water on the Young's modulus,  $E$ , of the TPS films immersed in glycerol/water mixtures is shown in Figure 4(a) where the value of  $E$  decreased with an increase in the water content. In the ethanol/water and isopropanol/water mixtures, the modulus of the TPS was observed only up to *ca.* 20 and 40% (v/v) water content, respectively, as shown in Figure 4(b,c). The value of  $E$  for the APTPS films did not change appreciably with increasing water content in any of the solvent systems. Moreover, the value of  $E$  for the APTPS film was considerably lower than that of the TPS film before immersion.

#### Effect of RH on Mechanical Properties

The tensile strength,  $\sigma$ , of the TPS and APTPS films after conditioning at different values of RH is shown in Figure 5(a). A significant difference ( $P \leq 0.05$ ) between the  $\sigma$  values of the TPS and those of the APTPS films, at a given value of RH, can be



**Figure 4.** Effects of film exposure for 5 min on the elastic modulus,  $E$ , of TPS (○) and APTPS (●) films: (a) glycerol/water immersion (b) ethanol/water immersion (c) isopropanol/water immersion and (d) RH conditioning.



**Figure 5.** Effects of percent RH on TPS (○) and APTPS (●) films: (a) ultimate tensile strength,  $\sigma$ , (b) elongation at break,  $\epsilon_b$ , and (c) elastic modulus,  $E$ . Exposure was for 7 days at  $20 \pm 1^\circ\text{C}$ .

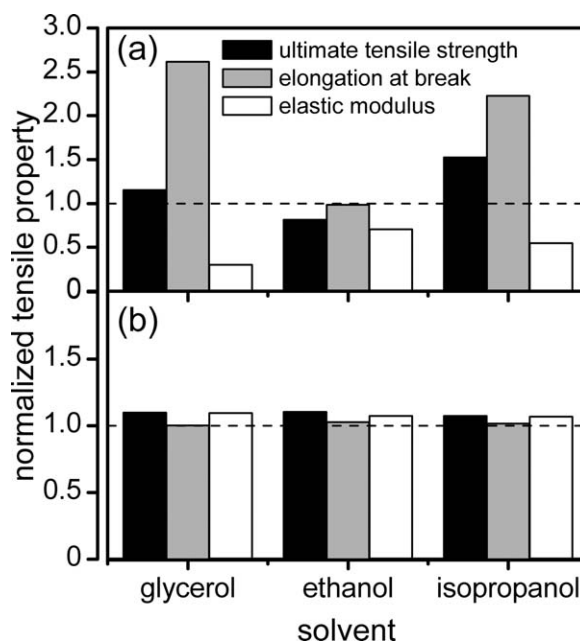
seen over most of the RH range. The tensile strength of the TPS film is considerably higher than that of the APTPS film up to  $\text{RH} \approx 23\%$  but decreases to a much lower level than that of the APTPS material with a further increase in RH. Interestingly, the tensile strength of the TPS material appears to be similar to that of the APTPS films at  $\text{RH} \approx 33\%$ . At  $\text{RH} > \text{ca. } 85\%$  the integrity of the TPS material was completely destroyed and no meaningful tensile measurements could be made. Forssell et al.<sup>33</sup> studied the sensitivity of starch-based films to RH in an ambient environment and observed a decline in the tensile strength of the films when exposed to increasing RH levels. Chantong and Lumdubwong<sup>34</sup> reported a significant effect of RH on the mechanical properties of thermoplastic sheets extruded from starch-based materials and Mehyar and Han<sup>35</sup> observed a decrease in tensile strength of high amylose rice starch and pea starch films when the RH was increased from 51 to 90%. In contrast, the APTPS material in this study maintained its tensile strength across the entire range of RH in accordance with the water content of the different solvent mixtures reported above in the present work (see Figure 2). These results suggest that the TPS material, in contrast to the APTPS material, is sensitive to systems of high water content and/or high RH levels at which its mechanical properties of its films are severely impaired.

The effect of RH on  $\epsilon_b$  for the TPS and APTPS films is shown in Figure 5(b). The results indicate that  $\epsilon_b$  values for the TPS

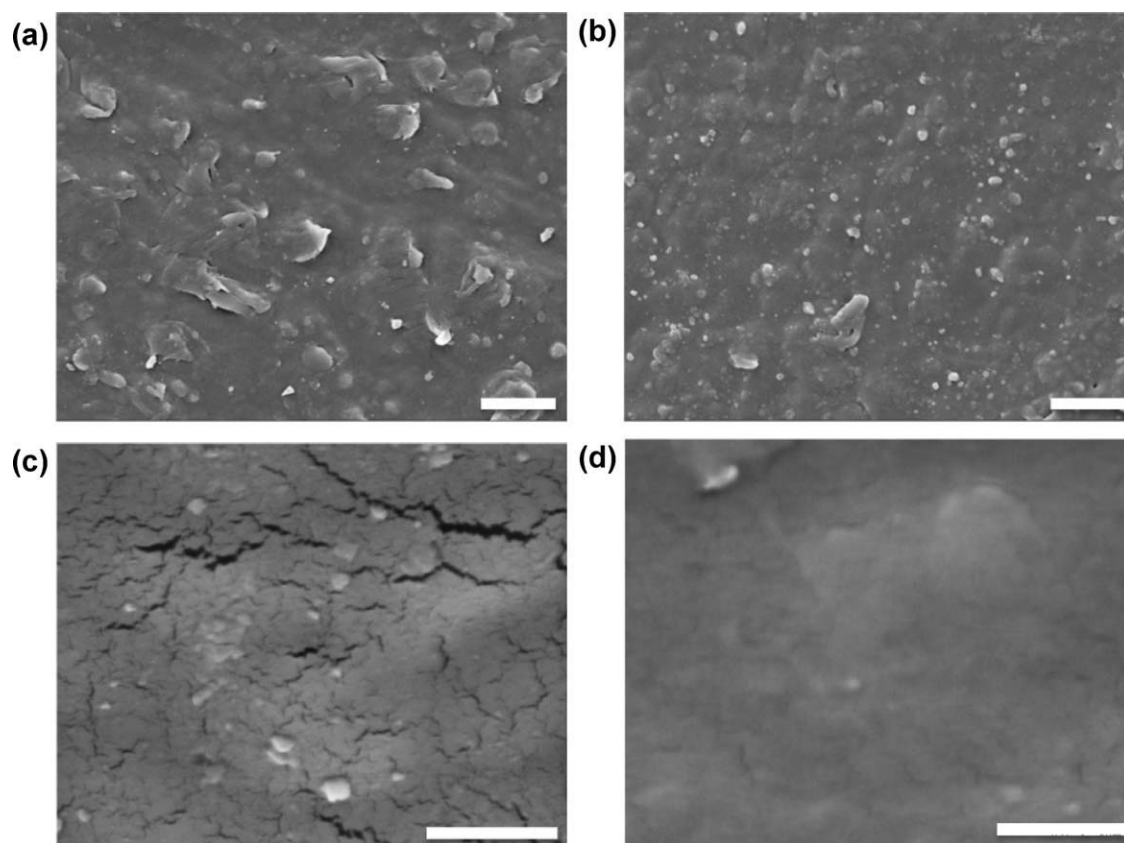
films are significantly lower ( $P \leq 0.05$ ) than those of the APTPS films at all values of RH. Furthermore,  $\epsilon_b$  for the TPS films decreased to zero as the RH increased to *ca.* 80% and the integrity of the film was lost. The TPS film samples were found to be relatively flexible at low RH values but lost their flexibility at  $\text{RH} \geq 75\%$ . Conversely,  $\epsilon_b$  of the APTPS film samples did not vary significantly across the RH range which is consistent with the immersion results obtained in the studied range of glycerol/water, ethanol/water, and isopropanol/water mixtures (see Figure 3).

The effect of RH on the value of  $E$  for the TPS and APTPS films is shown in Figure 5(c). For values of  $\text{RH} > \text{ca. } 85\%$ , the  $E$  values for the TPS film could not be determined due to the partial dissolution of the material and subsequent loss of integrity. In contrast, the  $E$  values for the APTPS film remained relatively steady at all RH levels, which is similar to the observations made in the investigation of the water-based mixtures. Chaléat et al.<sup>36</sup> reported that increasing RH reduces the Young's modulus of plasticized starch and this is in agreement with the results of this study. Stading et al.<sup>37</sup> also observed a decrease in  $E$  of starch films when the RH increased from 20 to 80%.

An indirect comparison of mechanical properties before and after immersion in solvent/water mixtures is possible at ambient RH conditions (*ca.* 50% RH). Figure 6 shows the change in each mechanical property for the APTPS and TPS films immersed in the pure solvents with respect to the values determined at 53% RH. Thus, values above and below the dashed line indicate that the measured property is higher and lower than the same value measured at 53% RH, respectively. As expected, the plot shows that the APTPS films appear to be relatively stable in all the solvents tested. Moreover, the solvents



**Figure 6.** Change in mechanical properties of: (a) TPS film and (b) APTPS film after immersion in pure solvents relative to 53% RH (dashed line).



**Figure 7.** SEM micrographs of: (a) TPS film at 1700 $\times$  magnification, (b) APTPS film at 1700 $\times$  magnification, (c) TPS film at 15,000 $\times$  magnification, and (d) APTPS film at 15,000 $\times$  magnification. Scale bars are 10  $\mu\text{m}$  for images (a) and (b) and 2  $\mu\text{m}$  for images (c) and (d).

glycerol and isopropanol have a greater influence on the mechanical properties of the TPS films than ethanol.

#### Imaging of Film Surfaces

SEM images at 1700 $\times$  magnification of the TPS and APTPS films are shown in Figure 7(a) and 7(b), respectively. In each case, there is evidence of what appears to be insoluble starch particles on the surface with relatively small particles in the APTPS film and larger but fewer heterogeneous particles in the TPS film. At the higher magnification of 15,000 $\times$ , the TPS film shows evidence of significant microcracking on the surface [Figure 7(c)] and this may contribute to both the poor mechanical strength and the unimpeded moisture diffusion and uptake of the films ultimately leading to the observed dissolution. In contrast, no microcracks were observed on the surface of APTPS films [see Figure 7(d)]. In the case of the APTPS films, the dominant structure is the APTPS with the starch incorporated as a cost-reducing agent.

#### Food Packaging Opportunities

The observed effects of water content of the immersion mixtures on the properties of TPS and APTPS films suggest that TPS film cannot be used for packaging of foodstuffs that have a moisture content higher than *ca.* 20% (v/v) as the mechanical properties of the film are severely impaired beyond this point (see Figures 2–4). The results also show that the presence of water in solvents has the ability to dissolve the hydrophilic

matrix of the TPS material. When compared with immersion in pure solvents, the mechanical properties varied significantly in glycerol and isopropanol but were relatively stable in ethanol (see Figure 6). Indeed, the compatibility of TPS films with aqueous, acidic and/or alcoholic foods could limit its packaging applications unless it is modified or blended. However, the APTPS films were observed to be mechanically stable in all of the water-based mixtures investigated.

The experiments on the effects of water on the mechanical properties of the TPS and APTPS films also revealed that the TPS films tended to dissolve in water-based mixtures that have high water content. It therefore appears that this material is not suitable for packaging of food products with  $a_w > ca. 0.75$  where significant mechanical strength is required as the films lost their mechanical integrity at 75% RH (see Figure 5). Nevertheless, TPS film can be used for the packaging of food products of low  $a_w$  levels. Many food products such as processed meats, bakery and hard dairy goods, some fresh produce, caramel, honey, noodles, and dried fruits have  $a_w$  values ranging from 0.6 to 0.99.<sup>38</sup> Thus, some of these products have  $a_w$  values that are below that of the apparent critical limit for the TPS material in this study. Given the inherent stability in all conditions tested, the APTPS films have the potential to be used in a wide range of food packaging applications. Further research into barrier properties of these films, studies on real food systems, and the possibility of active packaging using these films could reveal their full potential.

## CONCLUSIONS

Films comprising of TPS were successfully heat pressed under compression from a modified cornstarch material. The effects of water on the mechanical properties of the TPS and the commercial APTPS films showed that the TPS samples have a high affinity for water and tend to dissolve in mixtures of high water content with a consequent severe deterioration in their mechanical properties. Conditioning of samples of this film at various RH levels produced a noticeable absorption of water at high RH levels. In contrast, samples of the APTPS film did not demonstrate any discernible decrease in their mechanical properties when immersed in any of the water-based systems or when conditioned at any of the RH values used in this study. High magnification imaging revealed substantial microcracking on the surface of the TPS film samples. This observation along with the chemical structure of this material could contribute to its poor mechanical performance and propensity to adsorb water and dissolve. Nonetheless, the films derived from the TPS material in this study have the potential for packaging certain food products with low  $a_w$  providing high mechanical strength is not required. The APTPS films could be used for packaging at most  $a_w$  levels and therefore could be used with a much wider range of foodstuffs.

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