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Microwave-crosslinked Bio-based Starch/Clay Aerogels

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Abstract

Foam-like robust starch/sodium montmorillonite (Na⁺-MMT) aerogels were fabricated through a freeze-drying process. Glutaraldehyde (GL) was used to crosslink starch aerogels with the assistance of irradiation supplied by a domestic microwave oven. The chemical reaction between GL and starch was analyzed by Fourier Transform Infrared Spectroscopy and water swelling tests. The microstructures and mechanical properties of resultant crosslinked starch aerogels changed with GL concentration. By incorporating 5 w/w% GL, the specific compressive modulus and absorbed energy increased by 2.4 and 3.3 times, respectively. In regard to starch-clay aerogel composites, Na⁺-MMT clay played the role of reinforcement. Moreover, clay addition created more porous structures and hence decreased the thermal conductivity of aerogels. The biodegradability of aerogels was studied in a homemade micro respirometer. Starch aerogel exhibited higher biodegradability than poly(vinyl alcohol)-based one and its corresponding films.

Keywords: starch; aerogel; crosslink; microwave; biodegradability.

Introduction

Aerogels are a type of extremely light solid materials, which are composed by a solid skeleton and large fraction of air. They exhibit a high porosity and a low thermal conductivity, encountering potential applications in construction, packaging or insulation, to mention a few. Of the different types of aerogels, those based on polymers generally exhibit a tough behavior. However, the lack of mechanical properties, namely Young's modulus and yield strength, is a limitation for their wide use. To overcome this handicap, strategies such as filler reinforcement ¹ or matrix crosslinking ² have been applied, obtaining mechanically strong polymer aerogels.

To reduce wastes and raise product sustainability, biopolymer isolated from renewable resources have been received great interest in developing bio-aerogels. Several works have been reported, such as aerogels based on casein ³, alginate ⁴, pectin ⁵, agar and xanthan gum ⁶. These bio-aerogels showed foam-like mechanical performance, making them a promising alternative to polymeric foams derived from petroleum (e.g. expanded polystyrene).

Starch is one of the most abundant plant polysaccharides with numerous applications in different industries. Those include functions as energy nutrient in food products as well as additive in non-food products like textile and paper ⁷. It mainly consists of amylose and amylopectin, of which the relative proportion depends on the

10 starch source (e.g., potato, corn, wheat, tapioca) ⁸. Amylose is linearly comprised of α -(1 $\rightarrow$ 4)-linked D-glucopyranosyl units with an average M_w of 500 kg/mol while amylopectin is composed by a backbone of 1,4- α -D-glucose with 1,6-branched glucopyranosyl units, forming the amorphous part and the crystalline parts, respectively ⁹.

Sodium montmorillonite (Na^+ -MMT), a type of natural clay, has been used to successfully enhance the mechanical properties as well as the thermal stability and flame resistance of bio-based polymer aerogels ^{4, 6}. Therefore, it is expected that starch/clay composite aerogels will show superior properties than pure starch aerogels. On the other hand, chemical cross-linking can also improve the properties of aerogels. Glutaraldehyde (GL), a commonly used cross-linking agent, can react with hydroxyl groups on the starch molecules ^{10, 11}. It has been utilized to crosslink starch foam particles ¹² and starch-polyvinyl alcohol films ¹³. This chemical process is related with a hydrated etherization reaction that is time-costly and needs drastic reactive conditions ¹⁴. Recently, microwave-induced synthesis was considered with great interest due to its high efficiency. Graft copolymerization of polysaccharides has been reported without radical initiators or with very low initiator concentration on the effort of microwave irradiation ¹⁵. Therefore, it is feasible to take advantage of this approach to realize the crosslinking reaction of GL and starch.

In this work, bio-based starch aerogels were prepared using a freeze-drying process. Na^+ -MMT clay was added to starch aerogel, forming an organic-inorganic hybrid aerogel. GL was designed to react with starch with the assistance of a domestic

microwave oven. The effects of clay addition and GL crosslinking on the structures as well the mechanical properties of aerogels were analyzed. Moreover, the biodegradability of the obtained aerogels was investigated.

Experimental

Materials

Modified Potato Starch (E1442) was bought from Tradissimo (Spain). Sodium Montmorillonite (Na^+ -MMT, PGW grade) was purchased from Southern Clay, having a density of 2.6 g/cm^3 and cation exchange capacity (CEC) of 145 meq/100 g. Glutaraldehyde solution with a concentration of 50% (w/v) and Calcium chloride dehydrate (CaCl_2) were Sigma-Aldrich products. Sodium hydroxide (NaOH) was bought from Merck KGaA. All raw materials were used as received.

Sample Preparation

Pure starch aerogels: A predetermined amount of starch powder was hydrated in deionized (DI) water, and then mechanically stirred at 80°C for 2 hours. The prepared homogenous solutions were poured into vials with a diameter of 30 mm then frozen at -30°C in a fridge over night. The aerogel samples were finally obtained when ice was sublimated using a lyophilizer operated at 0.001 mbar and -80°C .

GL cross-linked starch aerogels: GL solution was added to the starch solution with

different GL/starch proportion as shown in Table 1. Hydrochloric acid was then added to adjust the pH value of the solution to pH=3. The mixed solutions were irradiated using a domestic microwave oven under 800 watt for 30 seconds before being frozen. The same freeze-drying process was finally conducted to get cross-linked aerogels.

Starch/clay aerogel composites: Clay suspensions were prepared through dispersing Na⁺-MMT nanoparticles in DI water using an IKA Ultra-turrax disperser. Starch solutions were then added and mixed to get homogenous suspensions prior to being frozen and lyophilized. Glutaraldehyde was also incorporated to crosslink the starch/clay aerogel composites. The starch-clay mixture with GL was treated in the microwave oven for 30 seconds before being frozen.

The identification of samples is according to the containing components followed by their corresponding mass fraction in the precursor suspension, as shown in Table 1. For instance, S, C and G stand for starch, clay and glutaraldehyde in sample S5C5G5, respectively.

Characterization

Fourier Transform Infrared (FTIR) spectra were recorded on a Nicolet 6700 spectrophotometer in the attenuated total reflectance (ATR) mode. Each sample had a spectral result based on 30 scans with a 1 cm⁻¹ resolution across a wavenumber interval between 4000 and 400 cm⁻¹.

The morphologies of the aerogels were investigated by Scanning Electron Microscope (SEM, Jeol-5610, Japan) operated at 10 kV. Prior to observation, the samples were

firstly cryo-fractured, then dried in vacuum and finally coated with a thin Au/Pd layer.

The apparent density (ρ_{app}) was calculated from the sample weight divided by the sample volume. Weights were measured by a balance (± 0.01 mg) and the dimensions were determined using a digital caliper (± 0.01 mm). Five replicas were taken for each composition.

Compression testing was carried out in a Galdabini (Italy) universal testing machine using a load cell of 1kN following ISO 604 standard¹⁶. The crosshead rate and maximum strain were set to 1 mm/min and 70%, respectively. The compressive Young's moduli (E_c) were calculated from the slopes of the initial linear region of the stress-strain curves. Yield stress (σ_y) was taken as the stress at the intersection between the tangent line of the elastic region and the tangent line of the stress plateau segment. The absorbed energy (E_a) was measured as the integrated area of the stress-strain curve at 60% strain. Five replicas of each sample were used.

The biodegradability of aerogels and their corresponding films was conducted using a homemade micro respirometer system according to ISO/FDIS 14855-2 method¹⁷. Compressed air flow was generated by a pump. A flask filled with NaOH pellets was set as a trapper to remove CO₂ in the air before they were injected into a 1000 ml reactive vessel. Prior to the tests, 82 grams of compost composed by 1/3 wt% compost soil, 1/3 wt% soy protein and 1/3 wt% wood pieces were poured into the reactive vessel. Then 5 grams of aerogel or film specimens were embedded into compost. Finally, the reactive vessel was kept in a water base at 65 °C. During the tests, 20 ml of water was added to each reactive vessel every three days. Aerogel gradually

degraded mainly into CO₂ and H₂O. Water was absorbed by CaCl₂ dehydrate and the evolved CO₂ flow was recorded by a K-30 CO₂ sensor. Aerogel films were prepared using an IQAP LAP PL-15 press. A cylinder aerogel was firstly placed between two steel plates covered with a thin polytetrafluoroethylene fabric and then compressed into a film operating the press at 25 °C and 6 MPa for 3 minutes.

Thermal conductivity of aerogels was determined using a QuickLine-30 thermal analyzer according to ASTM D5930¹⁸. Square specimens (6 x 6 cm) with thickness of 1 cm were prepared for the tests.

Results and Discussion

FTIR Spectrum

FTIR analyses were conducted to confirm the cross-linking reaction. Figure 1 shows the obtained spectra. The characteristic peaks of pure starch aerogel are located at 3284 cm⁻¹, 2924 cm⁻¹ and 1637 cm⁻¹, which corresponds to bonded OH stretching, CH₂ asymmetry stretching and OH bending, respectively¹⁹. For the band of CH₂ bending vibration, two peaks appear at 1412 cm⁻¹ and 1337 cm⁻¹, which are associated with CH₂ scissoring and CH₂ twist vibration, respectively²⁰. In the region between 900 cm⁻¹ and 1200 cm⁻¹, the bands at 1147 cm⁻¹ and 1078 cm⁻¹ contribute to the stretching vibration of C-O-C which bridges the glucoses units; the other two bands at 996 cm⁻¹ and 928 cm⁻¹ are related to the C-O stretching in anhydrous glucose ring of

starch molecules²¹. In addition, the peaks at 845 cm^{-1} and 760 cm^{-1} are assigned to the bending of CH in the glucose ring¹⁹. With the addition of clay, new peaks appear at 3618 cm^{-1} and 514 cm^{-1} , which are attributed to the free water in the interlayer of pristine MMT and Al-O bending, respectively²².

Figure 1. FTIR spectra of starch-based aerogels.

When 5 % GL was incorporated, the band at 928 cm^{-1} in pure starch aerogel was shifted to 937 cm^{-1} . This is because OH in the starch molecules reacted with GL as depicted in Figure 2, affecting the vibration of C-O group in the glucose ring. Moreover, a new band appeared in 1716 cm^{-1} due to the unreacted C=O groups of GL and the C-O stretching band at 928 cm^{-1} was moved to higher wavenumber (943 cm^{-1}).

Figure 2. Scheme of chemical reaction of starch and glutaraldehyde.

Solubility-Swelling

Aerogels crosslinked with different amount of GL and unmodified samples were firstly cut to equal dimension, and then placed in three different beakers containing 200 ml of DI water at room temperature. It was observed that the non-crosslinked sample quickly dissolved into cold water (Figure 3a). However, GL-modified samples were not dissolved; instead, they swelled in water and were transformed into stable hydrogels (Figure 3b, 3c and 3d). After staying in DI water at room temperature for 2 weeks, these hydrogels did not show any tendency of dissolution. This suggested that the GL effectively cross-linked the starch molecules. The networks that were formed between the starch molecules created blocked aerogel skeletons. Increasing the temperature to 80 °C at which starch should be dissolved, sample S5G2.5 was just partly dissolved. Nevertheless, the samples containing a higher amount of GL (S5G5 and S5G10) did not show any change in their shapes due to the greater density of crosslinking.

Figure 3. Configurations of starch-based aerogels in water: (a)S5; (b)S5G2.5; (c)S5G5; (d)S5G10.

Morphology

Figure 4. SEM micrographs of starch-based aerogels: (a)S5; (b)S5G2.5; (c)S5G5; (d)S5C5.

The microstructures of the different representative aerogels are depicted in Figure 4.

Sample S5 exhibited an irregular porous layered structure (Figure 4a) which is the path left by the ice crystals formed in different directions after freeze-drying.

The structures of crosslinked aerogels varied with the content of GL. When 2.5 w/w% of GL was added to crosslink 5 wt% starch solution, the resultant sample (S5G2.5) displayed a lamellar structure composed of solid layers connected with struts (Figure 4b). Starch molecules are assembled into large blocks after the cross-linking reaction, limiting the mass mobility and affecting their rearrangement or assemble at the boundaries between the growing ice crystals²³. On the other hand, the crosslinking resulted in an increase in the solution viscosity, retarding the ice crystals growth when it was frozen. With the quantity of GL increased to 5 w/w%, the lamellar structure was substituted by a honeycomb-like architecture (Figure 4c) due to the further increment in solution viscosity and decrease in mass mobility of precursor solutions. However, further increase of GL amount to 10 w/w% did not change aerogel microstructure. This indicates that a GL level of 5 w/w% is the optimum concentration to crosslink starch solution. This is consistent with FTIR investigation, which showed that isolated GL was presented when more than 5 w/w% of GL was added.

When inorganic clay was introduced to the precursor solution, clay platelets were encapsulated by starch molecules and reoriented by the growing ice front upon solution freezing, ultimately resulting in the “house of cards” structure²⁴ in which the layers were linked by the polymeric phase (Figure 4d). The incorporation of GL did not alter the overall structure of sample S5C5. These structural features are consistent

with the observed differences in mechanical properties that are discussed in the following section.

Compressive properties

The compressive stress-strain curves of the samples are shown in Figure 5. Starch based aerogels displayed typical elastic-plastic foam behaviour, which started with linear tendency at low strain followed by a plateau zone prior to the densification at high strains. The parameters defining the compressive mechanical behaviour are summarized in Table 2.

Figure 5. Compressive curves of starch-based aerogels.

Sample S5 was very soft ($E_c = 0.6$ Mpa) due to the low density of cell walls. With

the addition of clay, the mechanical performance was significantly improved. As studied in previous works that were carried out on polymer/clay aerogels^{4, 23, 25}, clay interacted with the polymers, reinforcing the strength of the sheet-like cell walls and thus the mechanical properties of aerogels. In the present work, clay strongly adhered to starch molecules, causing sample S5C5 to exhibit a more robust structure than S5. Sample S5C5 with a density of only 0.092 g/cm³ possessed a modulus of 7.39 MPa and yield strength of 212 kPa. Increasing the starch fraction in the starch/clay composites (*SxC5* series), the specific moduli of aerogels monotonically increased up to 50%. There was no significant change on specific compressive modulus (E_s) when a higher content of starch was loaded (sample S7.5C5). It may be attributed to an uneven dispersion of clay nanoparticles in a highly viscous precursor suspension which contains 7.5 wt% of starch.

The incorporation of GL had a minimal effect on apparent densities of starch aerogels. However, the values of both modulus and yield strength were monotonically increased with the GL concentration as shown in Table 2. By incorporating 5 w/w% glutaraldehyde, the E_s and absorbed energy (E_a) of sample S5 were increased by 2.4 and 3.3 times, respectively. Higher GL concentrations result in structures with greater crosslinked density that can dissipate energy more effectively under applied stress²³. Nevertheless, there were no significant changes on E_s and E_a when the GL content was increased from 5 w/w% to 10 w/w%. This is because of the marginal effect of GL above 5 w/w% of concentration.

The best composition in the present study was the sample S5C5G5 with a

specific modulus of 119 MPa and a yield stress 187 kPa, which were higher than the value of sample S5C5. It is because both the clay reinforcement and crosslinking contributed to the improvement of mechanical properties.

Biodegradability

Aerogel samples and compressed aerogel films that were investigated were based on poly(vinyl alcohol) (PVOH) and starch, respectively. PVOH-clay aerogel that was developed in our previous work ²⁶ and contained 50% clay and 50% PVOH was taken as a reference. Data of compost was listed as a baseline. The evolved released CO₂ amount during the tests, which indicates the biodegradable behaviour of the materials, is shown in Figure 6.

All samples exhibited a similar decomposition trend: a steep increase in the degradation rate occurred in the first 12 days until a plateau stage was reached after 27 days. Starch aerogels exhibited a higher decomposition rate than the PVOH-based sample as deduced from their corresponding slopes of the decomposition plots. On the other hand, starch aerogels released higher quantity of CO₂ during the same testing time. These facts indicated that the bio-based starch has a higher biodegradability than synthetic PVOH.

In comparison with the solid starch film, porous aerogels showed a faster degradation. This is attributed to the higher specific surface area of aerogels, which is beneficial to organism cell adhesion, growth and proliferation ²⁷. As a result, a faster consumption of matrix in aerogels occurred than in films, which was shown as

CO₂ release rate (higher slope in the patterns) and evolved CO₂ released amount.

Figure 6. Biodegradability behaviours of aerogels and compressed aerogel films.

Thermal conductivity

Sample S5 had a thermal conductivity value of 0.059 W/(m K). It is comparable to those of glass fiber or rock wool (0.04-0.05 W/(m K))²⁸ and is lower than that of wood wool (0.09 W/(m K))²⁹. Nevertheless, it is higher than those of close-cell foams, such as polystyrene (0.02–0.04 W/(m K))³⁰.

The increase in thermal conductivity with aerogel density is a known phenomenon and has been reported, for instance, in silica aerogel³¹. It is attributed to the increase of solid mass and pore wall thickness leading to the increase in solid phase conduction. However, introducing 50 % clay to sample S5, the thermal

conductivity was decreased to 0.053 W/(m K) with the apparent density increased in the present work. This is possible because the starch-clay hybrid aerogel exhibited a more porous structure than the pure starch aerogel. Even though clay has a higher thermal conductivity than starch, the tortuous paths created by the complex orientation of clay layers retard the heat transfer in the aerogels. The latter factor plays a dominant role for the thermal conductivity.

Conclusions

Bio-based starch/clay aerogels were prepared using a freeze-drying process. Na⁺-MMT clay addition not only increased the mechanical properties of the aerogels, but also reduced their thermal conductivity. Glutaraldehyde rapidly crosslinked with starch solution under the assistance of a domestic microwave oven. Microstructures of the resultant modified aerogels varied with GL concentration. The mechanical properties were significantly enhanced by cross-linking and GL level of 5 w/w% was the optimum concentration. Starch-based aerogels showed a higher biodegradability than synthetic PVOH-based aerogels. This work suggested a simple and effective way to improve the properties of bio-based starch aerogels.

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Table 1. Composition of the precursor suspensions for preparing aerogels.

Samples	Starch (wt%)	Na ⁺ -MMT (wt%)	Glutaraldehyde (w/w %) ^a
S5	5	0	0
S5G2.5	5	0	2.5
S5G5	5	0	5
S5G10	5	0	10
S2.5C5	2.5	5	0
S5C5	5	5	0
S5C2.5	5	2.5	0
S7.5C5	7.5	5	0
S5C5G5	5	5	5

^a (w/w %) indicates the weight percentage of GL relative to starch in precursor suspension.

Table 2. Compressive properties of starch-based aerogels.

Samples	ρ_{app} (g/cm ³)	E_c (Mpa)	E_s (MPa/g cm ³)	σ_y (kPa)	E_a (kJ/m ³)
S5	0.059±0.001	0.60±0.10	10.17±1.69	-	20.36±0.86
S5G2.5	0.071±0.002	1.60±0.28	22.07±3.34	57±3	52.65±4.22
S5G5	0.063±0.001	2.05±0.29	32.63±4.42	91±10	87.76±5.94
S5G10	0.068±0.001	2.42±0.47	35.79±6.96	96±10	84.74±17.87
S2.5C5	0.082±0.002	1.34±0.11	16.38±1.86	49±14	37.34±8.09
S5C5	0.092±0.01	7.39±1.64	80.25±18.32	212±51	189.27±14.38
S5C2.5	0.086±0.003	2.98±0.11	35.27±1.86	123±6	88.06±8.09
S7.5C5	0.113±0.002	8.85±1.87	78.12±15.10	333±29	285.77±15.66
S5C5G5	0.101±0.002	11.93±2.27	118.86±22.01	187±25	170.33±26.33

Microwave-crosslinked Bio-based Starch/Clay Aerogels

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Santana

Glutaraldehyde rapidly crosslinked starch via microwave irradiation, resulting in a significant improvement on mechanical properties of aerogels. Porous aerogels degraded faster than their solid film counterparts.

80x60mm (300 x 300 DPI)

Figure 1. FTIR spectra of starch-based aerogels.
243x205mm (300 x 300 DPI)

Figure 2. Scheme of chemical reaction of starch and glutaraldehyde.
46x19mm (300 x 300 DPI)

Figure 3. Configurations of starch-based aerogels in water: (a)S5; (b)S5G2.5; (c)S5G5; (d)S5G10.
59x48mm (300 x 300 DPI)

Figure 4. SEM micrographs of starch-based aerogels: (a)S5; (b)S5G2.5; (c)S5G5; (d)S5C5.
127x96mm (254 x 254 DPI)

Figure 4. SEM micrographs of starch-based aerogels: (a)S5; (b)S5G2.5; (c)S5G5; (d)S5C5.
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127x96mm (254 x 254 DPI)

Figure 5. Compressive curves of starch-based aerogels.
235x180mm (300 x 300 DPI)