



Surface-coated fly ash reinforced biodegradable poly(vinyl alcohol) composite films: part 2-analysis and characterization

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ABSTRACT

Composite films of poly(vinyl alcohol) (PVA) reinforced with 5, 10, 15, 20 and 25 wt.% surface-coated fly ash by surfactant, sodium lauryl sulphate (SLS-FA) along with 1 wt.% cross-linking agent, glutaraldehyde (GLA) were prepared by aqueous casting method. The tensile strengths of the composite films were increased proportionally with the addition of SLS-FA. The maximum 75% higher strength of the composite with 20 wt.% was achieved compared to that of neat PVA. The modulus of the composites was also increased proportionally with SLS-FA and the maximum 218% reached in composite with 20 wt.%, but the strain at break was decreased with addition of SLS-FA. Changes in FTIR spectra reflect the chemical and/or physical bonding in the ternary PVA, SLS-FA and GLA component systems. In the study of surface morphology, the connectivity was visualized in SEM images along with interstitial voids. The films with SLS-FA show 53% smoother surface calculated with AFM compared to unmodified FA composite films.

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1. Introduction

Composite materials composed of polymers and fillers show high modulus, strength and heat resistance, and low gas permeability and flammability compared to neat polymers [1]. The mechanical properties of composites generally depend on the filler's size and distribution, aspect ratio, volume fraction, and the intrinsic adhesion between the surfaces of filler and polymer [2]. High aspect ratio (fibre type) fillers generally increase the yield strength because the filler is capable of transferring high local stress from the polymer matrix [3]. The formation of interfacial interactions between filler and polymer can be influenced by the choice of surface functional groups and can lead to the fabrication of higher performance polymer composites.

PVA is a biodegradable and water-soluble polymer that is used in the fabrication of environmentally compatible composite films. The incorporation of materials as filler, such as sugar cane [4], starch [5], clay [6] carbon nanotubes [7], wood dust [8], cement [9], organo-ceramics [10] and TiO₂ [11] with PVA is an ongoing technology for fabrication of biodegradable composite. The most of the composites showed lower tensile strength compared to that of pure PVA film. PVA and its composites are currently using in variety of industrial applications, in the fibre and textiles industries for sizing

and finishing, coatings, adhesives, emulsifiers, colloidal stabilizers, film packaging for food and optical holographic applications [12].

Coal burning thermal power stations generates a huge amount of fine powdery by-product known as fly-ash (FA). The storage and handling of FA are challenging tasks in the context of environmental impact, as the material is usually disposed of by the landfill method in dams and lagoons [13]. FA is a mixture of alkali and transition metal oxides, mainly of silicon, aluminium and iron, with a small percentage of calcium, magnesium, potassium, sodium and titanium depending on the processing conditions [14]. Research on recycling and re-use of FA as a filler in eco-friendly composites is reported with metal [15] and polymer matrices such as polyester [16], epoxy [17], polypropylene (PP) [18–20].

The tensile strength of PP-FA composites was shown to dramatically decrease with the addition of spherical FA particles [19]. Considering the current status of the PVA composite formations, we have recently reported our initial research work on binary mixtures of PVA and fly ash from renewable resource for fabrication of composite films with higher strength by 193% compared to the neat PVA control [21]. We have also reported the overview role of physical and chemical treatment of fly ash on the strength of the composite film and cross-linking mechanism in our series of research works [22–24].

The surface modification of FA particles is considered as a potential pathway to achieve further enhancement of mechanical strength in composite films. The surface modification of single

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wall carbon nanotubes (SWCN) in a PVA composite was shown to significantly improve mechanical and magnetic properties [25].

The nature and concentration of surfactants were considerable parameters to play a role in phase behaviour and the isolated phases are not being an effect system for enhancement of interfacial interaction. Poulin et al. [26] conducted a series of reactions in fabrication of PVA fibre with different concentration of sodium dodecyl sulphate (SDS) and lithium dodecyl sulphate (LDS) coated on SWCN in aqueous solution [27] which homogeneously oriented the SWCN in PVA matrix to establish super-tougher composite fibres.

At the higher concentration of SDS, black textures were observed which denoted the clusters of surfactant. The clusters became larger and denser with increasing the SDS concentration and appeared as surfactant aggregation, micelle. The micelles easily separated the phase of the composites. The optimum concentration therefore, was set 0.3–1.5 wt.% SDS and used in their rest of works. Besides, the 1 wt.% SDS is also used in other research groups to achieve improved mechanical strength of PVA and SWCN composite films [7,25–28].

We recently have reported the modification process of fly ash with sodium lauryl sulphate along with application approach of SLS-FA as filler in fabrication of composite film with PVA [22]. The water sensitivity of the cost-effective and eco-efficient composite films was reduced by cross-linking agent, hexamethoxymethylmelamine (HMMM) [4] and glutaraldehyde (GLA) [5,8,22–24]. The composites with surface-modified fly ash along with cross-linking agent, GLA showed improved mechanical strength and water resistance which can be used as a tuneable structural feature for customized applications [22–24].

The objective of this study is to investigate the effect of surface modification of FA on mechanical properties and morphological characterization of the composite films of PVA and SLS-FA.

2. Experimental procedure

2.1. Materials

The FA sample was obtained from Swanbank coal fire plant, Cement Australia, Brisbane, Queensland. Poly(vinyl alcohol) (PVA) (Mw: 125,000 and degree of hydrolysis approx. 89%) was purchased from S.D. Fine-Chemical Ltd, Mumbai, India. Glutaraldehyde (GLA) (25 wt.% contents in water) from Laboratory Unilab Reagent, Sydney, Australia and sodium lauryl sulphate (SLS) from Ajax chemicals, Sydney, Australia were purchased and used as received.

2.2. Preparation of PVA-FA composite films

The modification process of the FA particles (50 g) were conducted with surfactant SLS in 0.5 wt.% aqueous solution (200 ml) for 10 h at 50 °C under vigorous stirring. The suspension then was filtered and washed by distilled water several times to remove non-adsorbed SLS. Finally the modified FA (SLS-FA) was dried under vacuum for 2 days at 50 °C [22–28].

The composite films were fabricated by casting from an aqueous solution of PVA and SLS-FA. The neat PVA was dissolved in distilled water at 80 °C to prepare a 1.2 wt.% solution. The SLS-FA particles in concentrations of 5, 10, 15, 20 and 25 wt.% were dispersed and sonicated for 5 min (Ultrasonic cleaner, FXPQM, frequency, 50 MHz). For the preparation of cross-linked composite films, 1 M HCl (50 μ l) and 1 wt.% GLA solution (0.50 ml) in distilled water were added sequentially to the PVA-FA solution. The resulting solution was cast in glass Petri- dishes (10 cm diameter) and any bubbles were removed by shaking. These were kept at room temperature until they appeared dry. The films were then peeled out and further dried in an oven at 60 °C under vacuum for 6 h. The thickness of the films was 0.05–0.07 mm. The thickness of the films was controlled by

using the same amount of materials (PVA and SLS-FA) in constant volume and used same sized glass petri-dish [21,22].

2.3. Testing and analysis

- (a) Philips CM200 TEM (transmission electron microscopy) was used with a field emission gun (FEG), which provides very high resolution but requires more care than a normal TEM filament. It is very important that a high vacuum is maintained in the gun chamber at all times. The FA/SLS-FA particles were dispersed in ethanol with sonication and a drop of diluted suspension was poured onto copper grid, which is directly injected in sample injection holder.
- (b) The mechanical properties tensile yield strength, strain to break and modulus of elasticity were determined using an Instron 1185 tensile testing machine with 50 mm/min crosshead speed. The specimens were prepared to ASTM D882-95a (length 22 mm, width 5 mm and thickness 0.05 mm) [6,8]. Five samples were tested for each condition and the results quoted are the average values.
- (c) Composite films were characterized by FTIR spectroscope (NEXUS-870, Thermo Nicolet Corporation) running with Omnic software. The condition for the measurement of FTIR: detector, MCT/A; base splitter, KBr; window, diamond; velocity, 0.6329; aperture, 100; scan, 64.
- (d) A Hitachi 4500-II scanning electron microscope was used to examine the morphology of the tensile-fractured PVA composite films at accelerating voltages 20 kV and working distance $Z=15$. The electrically non-conductive surfaces of the films were coated with thin layer of chromium using two sputtering cycles to improve the image quality.
- (e) The surface topography of the composites was investigated by a digital instruments 3000 atomic force microscope (AFM) in contact mode. Digital silicon nitride probes (DNP) with 0.58 N/m cantilever were used with nominal tip radius of curvature >10 nm, cantilever lengths 100 and 200 μ m, and tip height 2.5–3.5 μ m.

3. Results and discussion

3.1. Mechanical properties of neat PVA and composite films

The comprehensive physical, chemical, mineralogical and morphological characterization of FA particles as received and modified fly ash by surfactant (SLS-FA), was reported recently [22]. The TEM surface images of unmodified FA and modified SLS-FA particles were shown in Fig. 1(a) and (b). The existing interfacial interactions between the unmodified particles were disappeared in the modified particles and the thin layers will play a significant role in reduction of agglomeration of particles in fabrication of composite films.

The SDS coatings on fly ash particle surfaces shown in TEM images in Fig. 1(b) clearly remove the existing particle-particle interfacial interactions which play a significant role in reduction of agglomeration of particles in fabrication of composite films. The homogeneously distributed filler particles in polymer composite attribute to the higher mechanical strength compared to neat polymer matrix with increment of PVA-particle interactions. The spherical particles generally decreased the mechanical strength of the composites as a result of weak interfacial interactions between the polymer and filler. Thus it is proposed that the mechanical strength can be improved by the formation of interfacial interactions [10].

In addition, the agglomeration of spherical particles in composites can lead to a decrease in the mechanical strength. This

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