Original Article

Investigations on electrostatic dissipative materials derived from Poly(vinyl alcohol)/ferrofluid composites

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Abstract

Biodegradable polymer composites based on polyvinyl alcohol (PVA) and ferrofluid (FF) were prepared by solution casting method. Such composites were characterized by various methods in order to evaluate their potential for use as electrostatic dissipative (ESD) materials. Effects of ferrofluid content on mechanical, thermal, and electrical properties of the composites were investigated. The morphology of the composites was examined by SEM and the water contact angle on the composite surface was also measured. Experimental results showed that surface resistivity of the composites can be reduced by the addition of FF. The abrupt transition of such resistivity occurred in the concentration range 20-30 wt.% FF. The conductive mechanism of the proposed composites is a complex manner, including contact conduction and tunneling conduction.

Keywords: biodegradable polymer composites, electrostatic dissipative material, ferrofluid, surface resistivity, conduction

1. Introduction

The estimated annual losses in products containing sensitive electronics due to electrostatic discharge (ESD) during manufacturing, assembling, storage and shipping are in billions of dollars (Al-Saleh and Sundararaj, 2008). A variety of materials has been developed to package sensitive electronic devices and prevent damage during storage and shipping. According to the Electronic Industry Association (EIA) standards, conductive materials have a surface resistivity of less than 1.0×10^5 Ω/sq, dissipative materials have a surface resistivity from 1.0×10^5 to 1.0×10^{12} Ω/sq and insulative materials have a surface resistivity greater than 1.0×10^{12} Ω/sq (Narkisa *et al.*, 1997). Too high surface resistivity results in an uncontrolled discharge (Vakiparta *et al.*, 1995) and static dissipative materials are often used to slow down the charge removal process and prevent a damaging ESD event (Cambell and Tan, 1995). A variety of polymeric composites has been

developed for use as ESD materials. Insulating polymer matrices can be formulated by adding suitable amount, type and shape of electrically conductive fillers (Feller *et al.*, 2004; Jin *et al.*, 2010). One main parameter determining the ESD material properties is the conductive pathway structure, which is depending on many parameters, such as filler content, dispersion, distribution, conductivity and shape, surface free energy of the filler and the matrix, polymer matrix crystallinity, and processing conditions (Feller *et al.*, 2004; Al-Saleh and Sundararaj, 2008). The critical amount of filler necessary to initiate a continuous conductive network is referred to as the percolation threshold, which varies from polymer to polymer for a given filler. A small increase of the filler concentration above the percolation threshold has a much smaller effect and subsequently a plateau is reached.

Many ESD materials have been recently developed based on combining a number of polymeric materials with various electrically conductive fillers, such as glass fibers and carbon black (Narkis *et al.*, 1996; Zhang *et al.*, 2010), carbon nanofiber, carbon nanotube (Al-Saleh and Sundararaj, 2009), aluminum powder (Strzelec and Pospiech, 2008), polyaniline (Martins and De Paoli, 2005), immidazolium ionic liquid (Ding

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et al., 2008), and conductive copolymer (Kobayashi *et al.*, 2006). However, there are not many research works reporting the preparation and characterization of ESD materials derived from biodegradable polymer composites.

Polyvinyl alcohol (PVA) is one of the promising bio-degradable polymeric materials (Jin *et al.*, 2010). There are numerous proposals for its application in electronics, packaging, textile and food products, due to its easy preparation, high clarity, high gloss, and excellent durability, chemical resistance and physical properties. PVA can be brought into the dissipative range by incorporating conductive fillers.

Many types of conductive filler are available, such as metal particles, metal-coated glass, carbon powder, also known as carbon black, carbon fibers, and others. Ferrofluid (FF) is one of the interesting conductive fillers. FF is a colloi-dal dispersion of small single-domain magnetic particles, suspended in a carrier fluid. Recently, FF has been the subject of much interest because of its unusual optical, electronic, and magnetic properties, which can be changed by applying an external magnetic field. FF-composite materials have a potential for electromagnetic interference (EMI) shielding ap-plication, due to fact that magnetic particles have formed a hillock and tend to elongate in the direction of the applied magnetic field in polymer composites (Pant *et al.*, 2002; Pant *et al.*, 2004).

In this study, a novel biodegradable ESD material from PVA and FF was proposed. The effect of FF content on the electrical conductivity of the composites was investigated. The changes of the effective parameters including the composite morphology and the presence of conductive network in the polymer matrix were also studied. In addition, water contact angle, thermal and mechanical properties of the composite films were revealed.

2. Materials and Methods

2.1 Materials

Polyvinyl alcohol (PVA, Mw 85,000-124,000 g/mol) was purchased from Sigma-Aldrich Company, Germany. Glutaraldehyde (GA, 1.2%, w/v), the crosslinking agent, was from Fluka Company, Switzerland. The other chemical reagents used (hydrochloric acid, sulphuric acid, acetic acid and methanol) were from QRëc Company, New Zealand. Ferrofluid (FF), a colloidal suspension of Fe_3O_4 particles, with the viscosity of 11.2±0.1 cP, was synthesized in our laboratory by co-precipitation method between iron (III) chloride hexa-hydrate and iron (II) chloride tetrahydrate, using tetramethyl ammonium hydroxide (TMAOH) as a surfactant. The preparation method was described in a previous study (Jareonthanawong *et al.*, 2013).

2.2 Preparation of composites

A PVA solution with a concentration of 10 % (w/v) was prepared by dissolving PVA powder in deionized water at 90°C under stirring for 2 hrs. To prepare the composites, 10 mL of PVA solution was mixed with FF (20, 30, 40, 50 and 60% w/w), in which the amount of TMAOH was 0.125, 0.250, 0.375, 0.500, 0.625, and 0.750%, respectively. Then 2.8 mL of crosslink solution (50% w/v methanol (the quencher), 10% w/v acetic acid (the pH controller) 1.20% (w/v) of glutaraldehyde and 10% (w/v) sulfuric acid (the catalyst), make up a 3:2:1:1 weight ratio solution) was added into the mixture under constant stirring for 15 min, in order to obtain uniform distribution of the filler. The mixture was poured into a Petri disk and cured at 50°C for 12 hrs in a hot air oven and dried at 40°C for 24 hrs in a vacuum oven. The thickness of the dry film was about 0.1 mm.

2.3 Characterization

The water contact angle (WCA) on the composite surface was measured after a drop of DI water was set on the composite films and the side view pictures of the droplet were taken. The averaged WCA value was obtained by measuring five randomly selected droplets. The tensile stress-strain test of the composite films was achieved using a tension mode of Testometric (Micro 350) at room temperature. The size of the cured film was 5 cm × 0.5 cm and thickness was about 100 μm (sample number, n = 10). The gauge length was 3 cm and the cross-head speed was 6 mm/min. Surface morphology and the conductive filler distribution in the composite films were carried out using scanning electron microscope (model LEO-1450 VP) at 10 kV accelerating voltage. Thermogravimetric analysis (TGA) was carried out with Perkin Elmer, TGA 7HT at a heating rate of 10°C/min in the temperature range 50-700°C under nitrogen atmosphere. Differential scanning calo-rimetry (DSC) was performed, with Perkin Elmer Diamond DSC, from 50 to 200°C at a heating rate of 10°C /min under nitrogen atmosphere. Surface resistance of the composite films was carried out according to ASTM D-3359-97, and the corresponding surface resistivity was calculated using the equation:

$$\rho_s = R_s \frac{P}{g}; \quad P = 2(a + b + 2g) \tag{1}$$

where R_s is measured surface resistance in Ohm, P is the effective perimeter of the guarded electrode for the particular arrangement employed in cm, g is dimensions of probes digital multimeter in cm, and a, b are lengths of the sides of rectangular electrodes in cm.

3. Results and Discussion

SEM measurements were performed in order to inves-tigate the change in PVA matrix morphology after the addition of FF, together with the distribution of Fe_3O_4 particles in PVA matrix. Figure 1a displays SEM micrograph for crosslinked PVA film, showing smooth and relatively homogeneous appearance. Figure 1b to f displayed the SEM micrographs for the PVA-FF composites. It can be observed that the Fe_3O_4
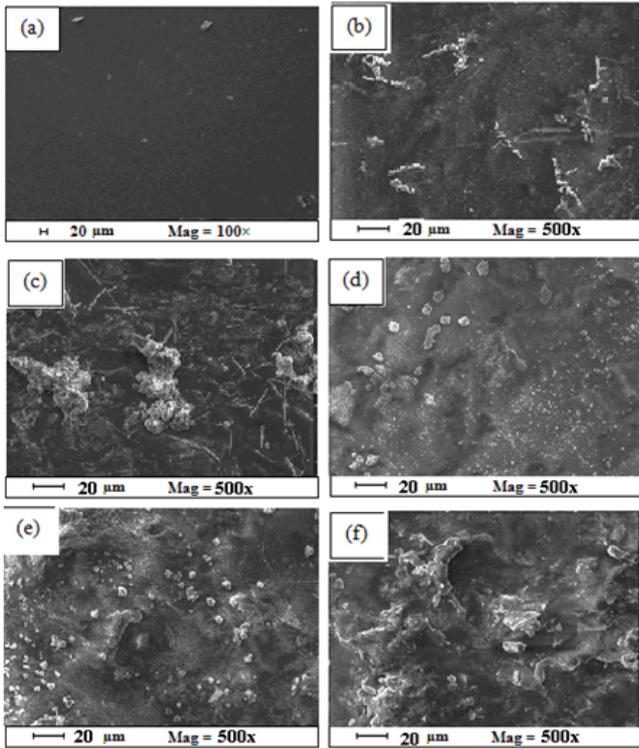
Figure 1. SEM micrographs of PVA-FF composites filled with (a) 0, (b) 20, (c) 30, (d) 40, (e) 50, and (f) 60 wt% FF.

particles displayed clear phase boundaries within continuous PVA matrix. At the lower filler concentration (Figure 1b), Fe_3O_4 particles and some Fe_3O_4 agglomerates discretely distributed, with relatively large inter-particle distances. At the higher filler concentrations (Figure 1c-f), the SEM micrographs revealed large Fe_3O_4 agglomerates dispersed within the PVA matrix, yielding a conductive network throughout the polymer matrix. This was due to Fe_3O_4 particles exhibited superparamagnetic behavior and, therefore, were easily prone to aggregation (Berger *et al.*, 1999; Pant *et al.*, 2002).

Mechanical properties are important criteria for practical applications of materials. In order to study the effects of FF content on the mechanical properties of PVA-FF composites, tensile stress-strain test was carried out. Figure 2 depicts the Young's modulus and elongation at break of the PVA-FF composites films as a function of FF content. It was seen in Figure 2 that with increasing FF content up to 30 wt%, the elongation at break of the composites decreased while the Young's modulus increased. The presence of hard fillers was responsible for the reduction in ductility of the composite films, as well as the increment in their stiffness, as Fe_3O_4 particles restrained the movement of the polymer chains. However, when more than 30 wt% of FF was added, the elongation at break of the composite films increased, whereas the Young's modulus decreased. Such a drop in the Young's modulus could be caused by the discontinuity of the PVA matrix due to the excessive content of FF. However, the noticeable decrease in Young's modulus accompanied by the

increase in elongation at break was mainly due to the surfactant, TMAOH, within FF acted as a plasticizer. Such plasticizer diluted polymer matrix and weaken interaction between polymer chains, thereby the Young's modulus of polymer matrix was decreased (Ying *et al.*, 2012; Jareonthanawong *et al.*, 2013).

Thermal decomposition of PVA-FF composites was determined by thermogravimetric analysis technique, which is based on continuous measurement of weight on a sensitive balance as temperature is increased in nitrogen atmosphere at a constant heating rate. Figure 3 shows TGA and DTG
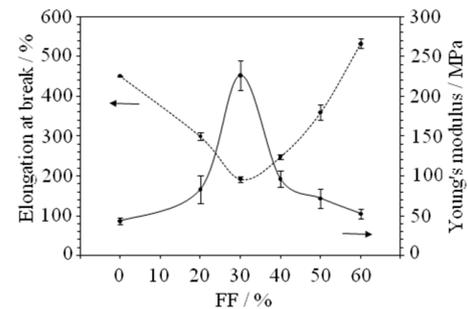

Figure 2. Effect of FF content on Young's modulus and elongation at break of the PVA-FF composite films.
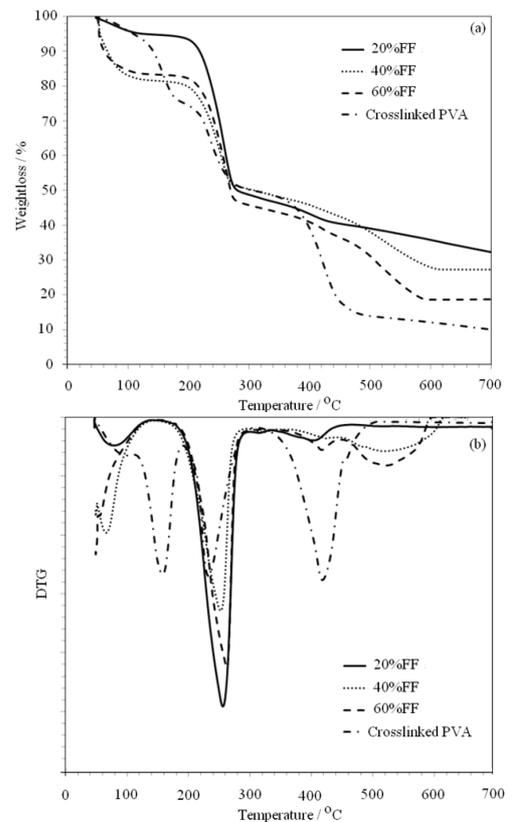

Figure 3. TGA thermograms (a) and DTG curves (b) of crosslinked PVA and PVA composites filled with 20, 40, and 60wt% FF.

Table 1. Summary of TGA results for crosslinked PVA and PVA/FF composites.

Sample	$T_{d,1}$ (°C)	$T_{d,2}$ (°C)	$T_{d,3}$ (°C)	$T_{d,4}$ (°C)	Residue (%)
Crosslinked PVA	162	235	419	nd.	10.2
PVA/20%FF	nd.	256	400	nd.	33.6
PVA/40%FF	nd.	260	nd.	518	27.3
PVA/60%FF	nd.	266	420	524	18.7

nd. = not detected.

curves of crosslinked PVA and PVA-FF composites. The summary of TGA results for crosslinked PVA and its composite films is shown in Table 1. Crosslinked PVA showed three-step degradation behavior. The first degradation step around 160°C was due to the thermal degradation of GA crosslinker. The second degradation step in the range 200-240°C was associated with the degradation of PVA molecule. The third degradation step in the range 390-420°C was due to the degradation of by-products generated during the TGA thermal degradation process. The weight residue of PVA at a temperature of around 700°C was 10.2 %, owing to various carbonaceous matters.

It can be seen in Figure 3 that all the composites showed the initial weight loss at temperatures 60-120°C. This may be due to the evaporation of the surfactant (TMAOH), which has boiling point in the range 99-110°C. PVA-FF composites also showed three consecutive weight loss steps. Each step is responsible for a thermal degradation of the PVA (at 190-280°C), thermal degradation of by-product generated by PVA during the TGA thermal degradation process (at 380-450°C) (Chen *et al.*, 2011), and a thermal degradation due to the decomposition of FF (at 450-580°C), respectively. The introduction of 20, 40, and 60 wt% FF increased the degradation temperature ($T_{d,2}$) of PVA from 235 to 256, 260, and 266°C, respectively. This was due to the fact that the degradation of polymers starts with the formation of free radicals at the ends of polymer chains, which gets transferred to adjacent chains via hydrogen bonds. With the presence of Fe_3O_4 particles, the iron bound PVA chains are not quite as mobile as the PVA chains due to the reduction of chain mobility; the chain transfer reaction is suppressed and consequently the degradation process is slowed and decomposition takes place at higher temperatures (Bhattacharya *et al.*, 2011). Consequently, the incorporation of Fe_3O_4 particles has significantly enhanced the stability of the polymer matrix. It can also be seen in Table 1 that the amount of residue reduced from 33.6 to 18.7% when FF content increased from 20 to 60%. This was because FF could react with the degradation products at high temperatures and produced new volatile products. Therefore, as FF content increased, there were more reactions occurred, as a result, the residue was reduced. However, the reason for this is still uncertain.

Structural changes of polymers are almost invariably accompanied by energetic effects. Hence, thermal properties of the PVA-FF composites were also investigated by DSC

measurements. Figure 4 shows the melting endotherms of PVA and PVA-FF composites. A sharp melting peak of PVA was found at 178°C, consistent with that reported by Cozzolino *et al.* (2012). Weaker melting peaks of PVA segments in the PVA-FF composites shifted to lower temperatures when FF was introduced to the composite. This indicates the reduction in the degree of the reorientation of PVA chains in the composites, owning to the presence of Fe_3O_4 particles. It is believed that Fe_3O_4 may hinder the crystallite growth. As a result, the significant decrease in the crystallinity of the PVA was observed (Miyazaki *et al.*, 2010; El-Sayed *et al.*, 2011; Mahmoud *et al.*, 2011).

The wettability of a solid surface is the property of that surface that determines how fast a liquid such as water or a solvent will adhere to such substrate. The ability of the liquid to spread uniformly over the solid surface is defined as the wetting ability of that liquid. The wettability can be measured by determining contact angle between the liquid and the solid surface. The wetting of a solid surface decreases as the contact angle increases. In our work, water contact angle (WCA) on various substrates as well as on the PVA-FF composite surface was measured and the data are shown in Table 2. It was proved that the neat PVA and crosslinked PVA films coated on the glass substrate is hydrophilic showing the WCA of 53° and 58°, respectively. This indicated the ability of water to wet the PVA surface. By introduction of FF, the composite films showed lower values of WCA, as a function of FF loading content. The values of WCA for the composites were in the range 33-44°. The presence of Fe_3O_4 particles in PVA matrix is responsible for such the decrease.
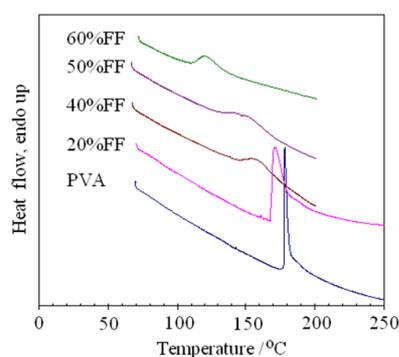

Figure 4. DSC thermograms for PVA-FF composites.

This was due to the very strong hydrophilicity of Fe_3O_4 particles, which possess a WCA of 0° (Lui *et al.*, 2008). Therefore, all the proposed composites shows good wetting with low water contact angle and is considered as hydrophilic materials.

The electrical properties of materials are of considerable importance in various applications, for examples, the insulation of electrical or telecommunication cables, electrical components, electrical appliances and accessories, printed circuits, radar and electronics. The electrical behavior of the conductive polymer composites generally depends on the concentration of the conductive filler, as the inter-particle separation was high at low loading content, and gets lower when the filler loading increases. Figure 5 depicted the effect of FF content on surface resistivity of PVA-FF composites. The resistivity of the composites can be reduced by addition of FF. With 20 wt % FF, when a conductive Fe_3O_4 particles network was not fully formed within the PVA matrix, the resistivity of the composites reduced from 1.00×10^{10} (that of the crosslinked PVA) to 9.42×10^8 Ω/sq. This is believed to be due to existence of the tunneling conduction (Zhang *et al.*, 2010). The resistivity showed a sudden drop and reached 1.83×10^7 Ω/sq when FF content increased to 30 wt%. The FF content in the range 20-30 wt% was, therefore, recognized as the electrical percolation threshold of the proposed composites. A significant decrease in the resistivity of the composites indicated the formation of filler networks

Table 2. Water contact angle.

Material	Contact angle / °
Untreated glass substrate	31±0.3
APS-treated glass substrate	60±0.8
Neat PVA	53±0.4
Crosslinked PVA	58±0.9
PVA/20% FF	44±0.8
PVA/30% FF	41±0.3
PVA/40% FF	39±0.5
PVA/50% FF	37±0.3
PVA/60% FF	33±0.4

Figure 5. Surface resistivity of PVA composites as a function of FF content.

(Subramaniama *et al.*, 2013). Beyond the percolation threshold, the resistivity decreased slowly with the increase of FF content, and reached 4.39×10^6 Ω/sq when 60 wt% FF was applied. By integration of the results form SEM and resistivity measurement, we can assume that the conductive mechanism of the proposed composites is a complex manner including contact conduction and tunneling conduction. In addition, the results obtained from WCA measurement revealed the increment in hydrophilicity of the proposed composites with increasing FF loading content. This is good for EDS applications, since with higher water uptake the composites possess even lower resistivity that can lead them to materials with interesting properties for the EDS applications (*Maya et al.,* 2012). As mentioned by Narkis *et al.* (1999) that the optimal surface resistivity of ESD materials is in the range 10^6-10^9 Ω/sq, the surface resistivity of the proposed composites, thus, revealed their capability in ESD applications.

4. Conclusions

A novel biodegradable ESD material from PVA and FF, known as colloidal dispersions of Fe_3O_4 magnetic particles suspended in a carrier fluid, was successfully synthesized by solution blending method. WCA measurement discovered the hydrophilic nature of the composites that increased with increasing FF loading content. The crystallinity of the PVA-FF composites decreased significantly, comparing with the neat PVA, as the FF loading content increased. However, the introduction of FF helped to improve the thermal stability of PVA matrix. The proposed composites exhibited the variation in mechanical properties depending on FF loading content. With increasing the FF content up to 30 wt%, the elongation at break of the composites decreased while the Young's modulus increased. On the other hand, when more than 30 wt% of the FF was added, the elongation at break of the composite increased, whereas the Young's modulus decreased. However, the mechanical properties of the proposed composites are in the range of those for commercial ESD materials. Since our proposed composites showed low values of tensile modulus, but quite high values of elongation at break, they are suitable for use as ESD bag. Although the microstructure showed good dispersion of Fe_3O_4 particles in the PVA matrix, some degree of aggregation was also observed. Such aggregation helped to construct the conductive network within the PVA matrix when the FF content reached the critical concentration. The percolation threshold was found in the range 20-30 wt% FF, in which the surface resistivity of the composites was in the range 10^7-10^9 Ω/sq, demonstrating the potential of the proposed composites for use as ESD materials.

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