



Electrical and mechanical properties of antistatic PVC films containing multi-layer graphene



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ABSTRACT

In order to explore practical application of graphene as novel conductive fillers in the field of composite materials, we prepared anti-static multi-layer graphene (MLG) filled poly(vinyl chloride) (PVC) composite films by using conventional melt-mixing method, and investigated electrical conductivity, tensile behavior, and thermal properties of the MLG/PVC composite films. We found that the presence of MLG can greatly increase electrical conductivity of the MLG/PVC composites, and the surface electrical conductivity of the MLG/PVC composites is less than 3×10^8 Ohm/square when the MLG loading is about 3.5 wt%, meeting anti-static requirement for commercial anti-static PVC films. On the other hand, the MLG/PVC composites exhibited higher tensile modulus and higher glass transition temperature than neat PVC, which is closely associated with crumpled morphology of the MLG and good compatibility between components of the MLG/PVC composites. By virtue of its satisfied anti-static performance and high mechanical properties, the MLG/PVC composites exhibit great potential to be used as high-performance antistatic materials in many fields.

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1. Introduction

Poly(vinyl chloride) (PVC), as one of the most consumed thermoplastics, has been widely used as cable sheathes, flooring materials, and construction pipes by virtue of its low cost, excellent corrosion resistance, good flame retardance, and easy operation. However, PVC is one of the typical insulating materials, and electric charges can be easily generated and accumulated on PVC surfaces, resulting in static-electric spark and even fire disasters [1]. In some circumstances such as chemical plants, gas stations, and coal mines, anti-static properties of PVC products are extremely important for ensurance of safety and reliability [2]. In that case, developing anti-static PVC materials has aroused much attention from academic and industrial fields for several decades.

Some methods for improving electrical conductivity of PVC materials have been developed for several decades [3–6], such as coating of conductive layers onto PVC surfaces, blending with conductive polymer, addition of conductive inorganic fillers or liquid anti-static agents. Among these methods, conductive coating

layers are easily peeled off due to weak interfacial bonding, while using conductive polymers can inevitably result in complex process and high cost [5]. At present, the most used method for preparation of anti-static PVC films in plastics industry is to add low molecular weight anti-static agents into PVC matrix, forming conductive gradient layers in PVC surface zones as a result of gradual migration of anti-static agents from bulk to surface of PVC products [7]. However, these soft PVC films containing anti-static agents possess intrinsic disadvantage of poor environmental stability, for example, their electrical conductivity strongly depends on atmospheric humidity and gradually decreases with service time of the products. In that case, replacing these liquid anti-static agents with solid conductive fillers will be an effective method for obtaining anti-static PVC films with good environmental stability.

Carbon black (CB) is one of the most widely-used conductive fillers because of its low cost and good compatibility with PVC matrix. While the CB generally possesses low electrical conductivity due to its intrinsic amorphous microstructure, in that case, high CB-loading of up to 25 wt% is generally needed for obtaining acceptable anti-static properties of CB/PVC composites, but inevitably sacrificing mechanical strength and processing performance of the CB/PVC composites. Recently, nanocarbon materials like carbon nanotubes (CNTs) have aroused great interests, only a small

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amount of CNT loading (0.1 wt%) can greatly increase electrical conductivity of PVC-based composites due to large aspect ratio and high intrinsic electrical conductivity of CNTs [8,9]. However, there still exists a critical issue of uniform dispersion for these entangled CNTs, greatly restricting large-scale commercial application of the CNT/PVC composites.

Graphene, as promising conductive fillers, exhibit extraordinary properties of high modulus, high electrical and thermal conductivity, and so on [10,11]. Especially, graphene are much easier to be dispersed throughout polymer matrix than these entangled CNTs, due to its large lateral size and unique two-dimensional planar structure [12]. Recently, using graphene as conductive fillers for improvement in electrical conductivity of PVC matrix has been reported in many literature. Vadukumpully et al. prepared graphene/PVC composite films by using solution blending method, and they reported that the graphene/PVC composites exhibited remarkable improvement by 58% in Young's modulus at 2 wt% graphene loadings and low electrical percolation threshold of 0.6 vol.% [4]. Pham et al. prepared graphene/PVC composites with segregated network by using hot-press methods, and the nanocomposites exhibit a percolation threshold as low as 0.4 wt% [13]. Dang et al. fabricated graphene/PVC composites with a threshold of 0.3 wt% by using latex technology [14]. Ma et al. reported remarkable enhancement in electrical conductivity of polyaniline-coated graphene/PVC composites [15], and Joshi et al. found that graphene/PVC composites exhibited high dielectric properties [16,17]. These reports revealed that graphene could be used as competitive conductive fillers to greatly improve electrical conductivity of PVC materials. However, it is worth pointing out that, all these graphene/PVC composites mentioned above have been prepared by using organic solvent, ultrasonic dispersion, and evaporation of solvent, inevitably resulting in complicated procedures, high cost, and environmental pollution. Furthermore, this solution method is extremely difficult to be used for large-scale production of nanocomposites. As a contrast, melt-mixing method is a conventional method for preparing thermoplastic composite materials in a large quantity. Notably, with the breakthrough of high-quality multi-layer graphene (MLG) production at low costs [18], how to prepare high-performance graphene/PVC composites in a simple way has been a critical issue for exploring and pushing practical application of graphene and its composites forward. As far as we know, preparation of anti-static PVC films containing commercial multi-layer graphene (MLG) by using conventional melt-mixing method has not been reported yet.

The aim of this work is to use commercial multi-layer graphene (MLG) as conductive fillers for improving electrical conductivity of the MLG/PVC composites. Electrical and mechanical properties of the MLG/PVC composites were investigated in details. We found that the presence of graphene could greatly increase electrical conductivity, tensile modulus, glass transition temperature of the MLG/PVC composites, which is mainly attributed to good dispersion, excellent electrical conductivity of MLG, and good compatibility between MLG and PVC.

2. Experiments

2.1. Materials

Commercial multi-layer graphene (MLG) powders, produced by using interlayer catalytic exfoliation (ICE) method [18], were supplied from Sichuan Jinlu Group Co., Ltd. China. Layer number of the used MLG is less than 10 graphitic layers (thickness of about 1–3 nm), and its lateral size is as large as 10–15 μm . The MLG powders have high structural integrity and less defects (its C/O ratio is more than 20, indicated by XPS spectrum) [19], and its electrical

conductivity is as high as over 700 S/cm. Poly(vinyl chloride) (PVC) resin powders (general type SG-5) and rare-earth stabilizers were also supplied from Sichuan Jinlu Group Co., Ltd. China. Plasticizers of dioctyl phthalate (DOP) were supplied by Sino-pharm Chemical Reagent Co., Ltd.

2.2. Preparation of the MLG/PVC composite films

The weighed PVC powders and DOP (weight ratio of 2:1) were well mixed together at room temperature by using a high-speed mixer, and then aged for 1 h in a drying oven at 80 °C. Thereafter, the obtained mixtures were mixed with stabilizers and MLG powders, and then heated up to 170 °C and squeezed into thin MLG/PVC composite films by using a two-roll mill. The MLG/PVC composites with various MLG loadings of 0, 0.5, 1.0, 2.5, 3.5, 4.5 wt.% were obtained following same processing procedures.

2.3. Characterization

Microstructure and morphology of the used MLG was characterized by using scanning electron microscope (SEM, NavoSEM430, FEI). Fracture surfaces of the MLG/PVC composites were observed by using SEM technique. Electrical resistivity of nanocomposites was measured by using high resistance meter (6517B, Keithley Instrument Inc.). Tensile properties of the MLG/PVC composite films were performed on a universal testing machine (type WSM-50KG, Changchun Intelligent Equipment Co., Ltd) at a speed of 5 mm/min referring to ASTM D882-10. Thermal properties of nanocomposites were measured by using a differential scanning calorimetric instrument (DSC, STA 449, Netzsch). A dynamic mechanical analyzer (DMA Q800, TA Instrument) was used for measuring storage modulus, loss modulus, and loss factor ($\tan \delta$) of the MLG/PVC composite films in a tensile mode at a heating rate of 3 °C/min from –50 °C to 100 °C in air atmosphere, in order to investigate effect of the MLG on dynamic mechanical behavior and glass transition temperature of the nanocomposites.

3. Results and discussion

3.1. Microstructure of MLG/PVC composite films

Fig. 1 shows microstructure of the MLG powders and MLG/PVC composites. It can be seen clearly from the SEM image of Fig. 1a that the used MLG exhibits typical crumpled morphology, totally different from the so-called two-dimensional planar morphology of graphene [20]. Such crumpled morphology of the MLG is mainly attributed to high flexibility of the MLG due to its large lateral size, thin thickness, and large aspect ratio of over 1000 [12]. Comparing with smooth fracture surfaces of neat PVC shown in Fig. 1b, all the graphene/PVC composites exhibit coarse and uneven fracture surfaces, implying that the presence of MLG can increase fracture toughness of nanocomposites. The MLG/PVC composites possess much richer interfaces and higher flexibility than the neat PVC due to large surface area and high flexibility of the MLG, resulting in much high energy dissipation and high fracture toughness of nanocomposites during fracture [19]. On the other hand, from SEM images of fractured MLG/PVC composite films (Fig. 1c–g), we cannot find obvious aggregation of MLG throughout PVC matrix even at the high MLG loading of 4.5 wt%, indicating that the MLG can be well dispersed throughout PVC matrix by simply using conventional melt-mixing method. Such uniform dispersion of MLG is mainly attribute to large lateral size and crumpled morphology of MLG [12], showing much better dispersion than those entangled CNTs. Moreover, the presence of DOP plasticizer is also helpful for uniform dispersion of MLG throughout PVC matrix

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