

## Properties of Nanoparticles Filled Soft Poly(Vinyl Chloride) Composites Including Antistatic Plasticizer

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**ABSTRACT:** Nanoparticles (NP) filled permanently antistatic poly(vinyl chloride) (PVC) composites, constituted of dibutyl phthalate (DBP) and antistatic plasticizer (AP) which included bis[2-(2-methoxyethoxy)ethyl]phthalate doped with sodium perchlorate ( $\text{NaClO}_4$ ), were prepared in a Haaka torque rheometer. Surface resistivity measurement, mechanical test, scanning electron microscopy (SEM) investigation, and thermal gravimetric analysis (TGA)-differential scanning calorimetry (DSC) analysis were used to investigate the comprehensive properties of PVC/AP/NP (100/40/x) (A40/NP) and PVC/AP/DBP/NP (100/40/40/x) (A80/NP) composites. The results demonstrated that the surface resistivity of A40/NP composites was lower than that of pure A40 composites at a humidity of 60% and 0.1% as the nano  $\text{SiO}_2$  or  $\text{TiO}_2$  content is 2 phr, respectively. Moreover, the surface resistivity of A40 composites was decreased by about half an order of magnitude even at the humidity of 0.1% when 2 phr of NP was added. The surface resistivity of A80/NP composites achieved the optimum value as the  $\text{SiO}_2$  and  $\text{TiO}_2$  content were 1 phr and 2 phr, respectively. Because the DBP functioned as small molecule plasticizer which endowed PVC composites with comparatively large free volume, the surface resistivity of A80/NP composites is much lower than that of A40/NP composites. The tensile strength and elongation at break of A40/NP (100/2) and A80/NP (100/2) were increased to some extent with respect to pure PVC/AP composites. DSC-TGA analysis and rheological properties demonstrated that NP filled PVC composites processed good thermostability and thermoprocessability. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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### INTRODUCTION

Static charges, originating from friction, accumulate on the surface of material, which may cause dust contaminations or surface pollution. All these problems will affect the tidiness, transparency of material, and even lead to fire or explosion when these materials are used in coal-mining industry.<sup>1</sup> Thus, several methods have been used to enhance the dissipation of high electrical charge density on the surface of plastics and reduce the surface or volume resistivity.<sup>2–4</sup>

Poly(vinyl chloride) (PVC), for example, with high surface resistivity and excessive electrical conductivity range of  $10^{-13}$  to  $10^{-15}$  S/cm,<sup>5,6</sup> can be modified by doping with antistatic agents or conductive fillers.<sup>7–10</sup> Unfortunately, both of them have shortcomings. The antistatic property of antistatic agents filled PVC composites can be largely affected by the variation of humidity and the compatibility between matrix and antistatic agents. What is more, they cannot endow the polymer with a

permanent antistatic property especially in low relative humidity (RH) environment. Carbon black and metal fibers, functioned as conductive fillers, suffer the problems of migration and make the products nontransparent and hard to process.

Currently, gel polymer electrolytes (GPE),<sup>11–14</sup> composed of polymer, high-dielectric constant solvent and alkali metal salt, have been widely studied because of their instinctive ionic conductive ability even at low humidity. For this advantage, they are widely applied in rechargeable lithium-polymer batteries. Plenty of studies have been done in this respect.<sup>15–18</sup> In order to extent the application field of GPE, nano fillers, for the advantage of nano size, are blended with GPE, thus GPE with better ionic conductive ability are obtained. Much work has been done to explore the mechanism of polymer-nanoparticles (NP)-salts interactions which proposes that nano-sized particles can improve the ionic conductivity and mechanical stability of polymer matrix to a certain extent.<sup>10,15–18</sup> Croce studied the

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effect of NP on polymer electrolytes and found that the addition of NP greatly enhances the properties of electrolytes by improving ionic conductivity for about one order of magnitude.<sup>15–17</sup>

As have been reported, the antistatic plasticizer, newly synthesized by our laboratory, prepared by the mixture of an aromatic ester plasticizer with ether oxygen bonds called bis[2-(2-methoxyethoxy)ethyl]phthalate (BMEP) and sodium thiocyanate (NaSCN), functions as permanently antistatic agent. PVC, doped with certain content of antistatic plasticizer (AP), shows good antistatic properties even at a RH of 0.1% and reasonable mechanical properties.<sup>19</sup> However, the antistatic and mechanical properties of PVC/BMEP/NaSCN composites are not sufficient to satisfy the special need in some antistatic fields. Since sodium perchlorate ( $\text{NaClO}_4$ ) can be easier dissociated by BMEP than that of NaSCN, in this article, an improved AP is prepared by doping  $\text{NaClO}_4$  with BMEP. Because NP plays an important role in improving the ionic conductivity and mechanical properties of GPE,<sup>5,17</sup> in this article, NP is added into PVC/AP matrix to enhance antistatic property. PVC/AP (100/40) (A40) and PVC/AP/dibutyl phthalate (DBP) (100/40/40) (A80) are modified with different contents of nano  $\text{SiO}_2$  and  $\text{TiO}_2$ , which is studied for the first time. The effect of NP concentration on the ionic conductivity has been investigated by surface resistivity analysis in different temperatures and humidity. The composites have also been characterized by mechanical tests, scanning electron microscopy (SEM) measurement and thermal gravimetric analysis (TGA)-differential scanning calorimetry (DSC) analysis. The correlation between the antistatic properties and morphology of NP dispersing in PVC composites is established.

## EXPERIMENTAL

### Materials

Analytically pure dimethyl phthalate, analytically pure DBP, tetrabutyl titanate, and sodium perchlorate ( $\text{NaClO}_4$ ) were supplied by Kelong Chemical Reagent (Chengdu, China). Diethylene glycol monomethyl ether was purchased from Tianjin Bodi Chemical (Tianjin, China) and was used as received. Besides, nano  $\text{TiO}_2$  (20–50 nm) was supplied by Jinan Yuxing Chemical Reagent (Jinan, China) and fumed  $\text{SiO}_2$  (10–40 nm) was purchased from Nanjing Haitai Nano (Nanjing, China). PVC powder (SG-V; average degree of polymerization = 1000–1100), compound stabilizer (Baeropan SMS 318) were provided by Tianyuan (Yibin, China) and Baerlocher (Lingen, Germany), respectively.

### Synthesis of BMEP and Preparation of AP

The BMEP was synthesized by ester-exchange reaction between dimethyl phthalate and diethylene glycol monomethyl ether. Then AP was prepared by doping  $\text{NaClO}_4$  with BMEP. Relevant synthetic details could refer to our former literature.<sup>19</sup>

### Preparation of PVC/AP/NP and PVC/AP/DBP/NP Antistatic Composites

PVC powder, AP, compound stabilizer, and nano  $\text{TiO}_2$  or  $\text{SiO}_2$  were mixed in a high-speed mixer (Yongtong SDF-400, Chengdu, China) with a mixing speed of 1500 rpm at room temperature for 5 min. The mixture was dried at 75°C for 2 hr. Then the resulting PVC composites were mixed in a Haake tor-

que rheometer (Thermo Fisher Scientific, Walldorf, Germany) at 170°C for 8 min and the mixing speed was 30 rpm (anisotropic).

### Characterization

**Surface Resistivity Measurement.** Soft A40 and A80 composites doped with different fractions of NP were processed into  $20 \times 20 \times 1 \text{ mm}^3$  sheets by preforming machine at the temperature of 180°C. A surface resistivity meter (ZC46A, Shanghai, China) was utilized to measure the surface resistivity of these sheets at room temperature under the RH of 0.1% and 60%. It was also used to measure the surface resistivity of samples at different temperature at the RH of 0.1%. The testing equipment and conditions could refer to our previous articles.<sup>19,20</sup>

**Mechanical Characterization.** The tensile strength and elongation at break of the samples were tested with an Instron 4302 (Instron) at a tensile rate of 100 mm/min. The hardness data of the samples were obtained with a Shore hardness meter (LXA-D, Shanghai, China).

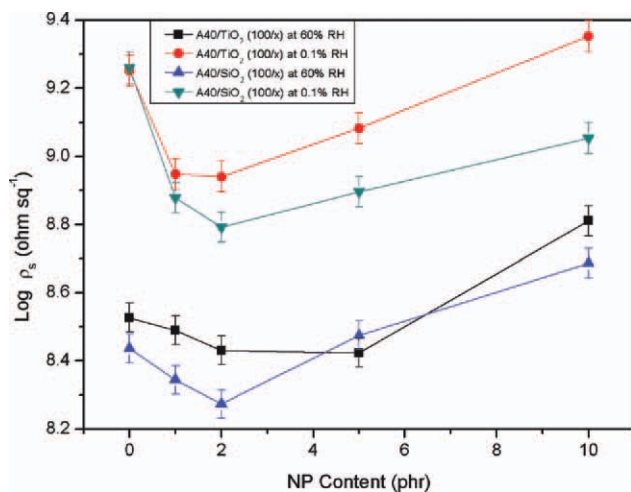
**SEM Analysis.** The liquid nitrogen frozen fracture surfaces of A40/ $\text{SiO}_2$  (100/x) composites were obtained. The SEM samples were gold-sputtered prior to observation. SEM morphologies of tensile fracture surfaces of samples were observed by JEOLJSM-5900LV SEM (Japan).

**DSC-TGA Studies.** DSC-TGA studies of the A40/NP (100/2) composites and the pure A40 composite were carried out in a TA instrument SDT-Q600 thermal analyzer from 25 to 450°C in air atmosphere, with a heating rate of 10°C  $\text{min}^{-1}$  and ca. 10 mg of each sample. All the samples were previously dried at 80°C for 4 hr. The DSC-TGA curves were analyzed by a TA universal analysis program.

## RESULTS AND DISCUSSION

### Surface Resistivity Analysis

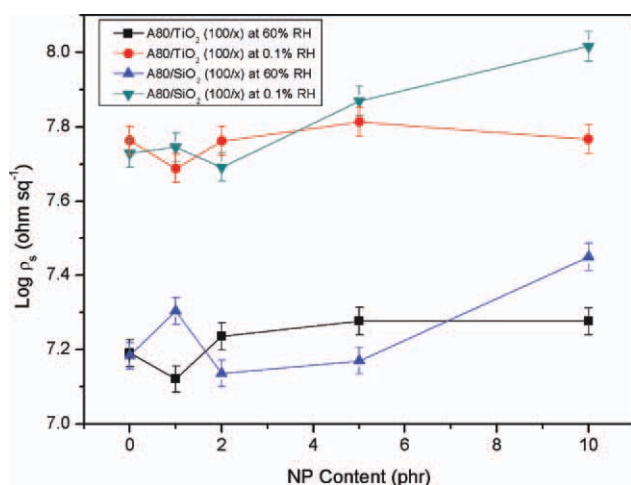
The effect of NP content ( $\text{TiO}_2$  or  $\text{SiO}_2$ ) on surface resistivity of PVC/AP composites is shown in Figures 1 and 2. As for the A40/NP (100/x) composites, the surface resistivity decreases to a minimum value as NP content increases from 0 to 2 phr and then gradually increases with NP content. The surface resistivity of A40/NP (100/x) composites at a humidity of 0.1% is about 0.7 orders of magnitude higher than that at a humidity of 60%, which demonstrates that PVC composites filled with NP are slightly sensitive to humidity. While the addition of NP is 2 phr, the surface resistivity of A40/ $\text{SiO}_2$  (100/x) composites is lower than that of A40/ $\text{TiO}_2$  (100/x) composites either at a humidity of 60% or at a humidity of 0.1%, which demonstrates that  $\text{SiO}_2$  is more effective in decreasing the surface resistivity of PVC/AP composites. As shown in Figure 2, the surface resistivity of A80/ $\text{TiO}_2$  (100/x) composites decreases as the addition of  $\text{TiO}_2$  is less than 1 phr and then slightly increases with the  $\text{TiO}_2$  content. However, the surface resistivity of A80/ $\text{SiO}_2$  (100/x) composites achieves the minimum value as the nano  $\text{SiO}_2$  content is 2 phr. The variation tendency of surface resistivity of A80/NP (100/x) composites is much smaller than that of A40/NP (100/x) composites and the surface resistivity of A80/



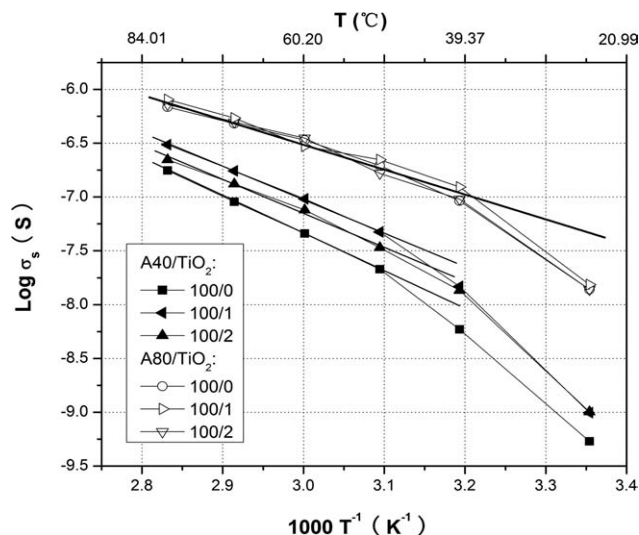
**Figure 1.** Surface resistivity of the A40/NP (100/x) composites with different NP contents. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

NP (100/x) composites at humidity of 0.1% is also 0.7 orders of magnitude higher than that under humidity of 60%.

The mechanism of NP reducing the surface resistivity of PVC/AP composites is that weak Lewis acid or Bronsted-type acid induces the interactions in the polymer-salt-filler system.<sup>5,21,22</sup> Because of the appropriate specific surface area and the existence of Lewis acid, 2 phr of NP is favorable for the departure of alkali metal ions. Lewis acid on the surface of NP can interact with the O atoms in  $-C-O-C-$  of the BMEP and  $ClO_4^-$ . Meanwhile Lewis acid weakens the interaction between these O atoms and  $Na^+$  cations and then release free  $Na^+$  cations, which increases the transference number of  $Na^+$  cations. The interaction between NP and O atoms in the BMEP and  $ClO_4^-$  induces structural modification of polymer chain, which is favorable to conduct the path for faster migration of  $Na^+$  cations on the surface of the loaded NP. Thus, more carriers used for the migration of charges can be



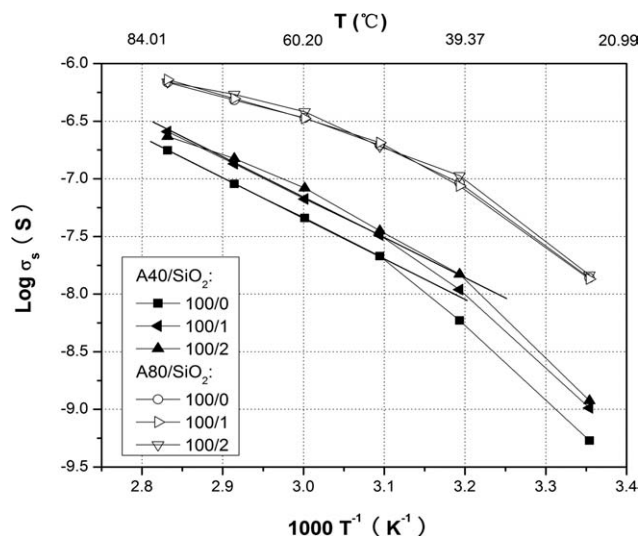
**Figure 2.** Surface resistivity of the A80/NP (100/x) composites with different NP contents. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



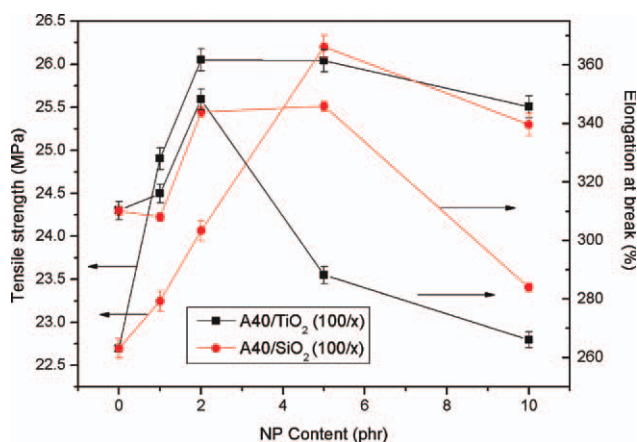
**Figure 3.** Surface conductivity of A40/TiO<sub>2</sub> and A80/TiO<sub>2</sub> composites with different temperatures.

obtained. As a result, the surface resistivity of NP filled PVC/AP composites are decreased. However, excessive NP may result in the aggregation of nano fillers and the decrease of free volume for the ionic transportation, which may reduce the interaction between NP and composites and result in the higher surface resistivity. Therefore, SEM study will be introduced to confirm this assumption. As have been discussed in our former work,<sup>19</sup> comparing with A40/NP composites, DBP in A80/NP composites, functioned as small molecule plasticizer, endows PVC composites with comparatively large free volume, thus the surface resistivity of A80/NP composites is much lower than that of A40/NP composites for the fact that the transportation of ions is much easier with the addition of DBP.

The temperature dependence of ionic conductivity of the A40/NP (100/x) and A80/NP (100/x) composites was evaluated under a constant humidity of 0.1%, and the plots were



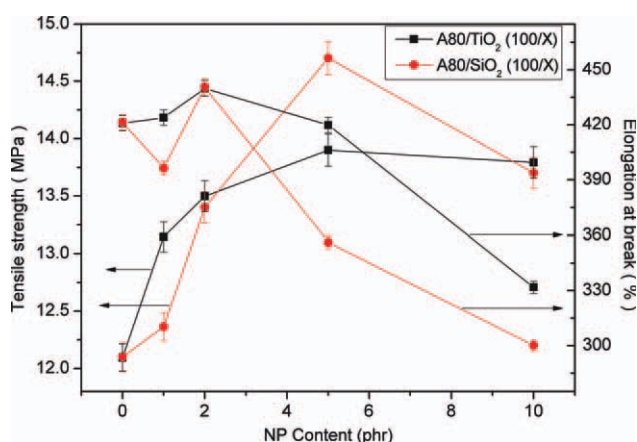
**Figure 4.** Surface conductivity of A40/SiO<sub>2</sub> and A80/SiO<sub>2</sub> composites with different temperatures.



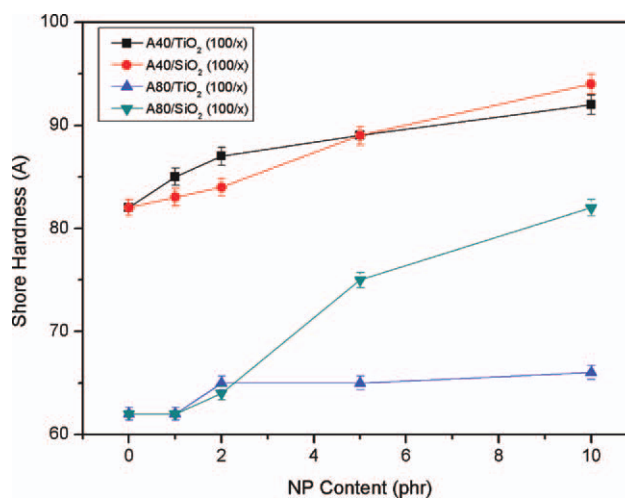
**Figure 5.** The tensile strength and elongation at break of the A40/NP (100/x) composites with different NP contents. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

presented in Figures 3 and 4. As can be noticed from Figures 3 and 4, with temperatures ranging from 25 to 80°C, the surface resistivity of these composites decreases for about 2-3 orders of magnitude. The surface resistivity of A40 composites doped with different contents of NP shows similar variation trends with the increase of temperature, so as the surface resistivity of A80 composites. However, because of the function of additional 40 phr of DBP as we have discussed earlier, the surface resistivity of A80/NP composites is about 0.5-1.0 ohm/sq lower than that of A40/NP composites with the variation of temperature. As shown in Figures 3 and 4, the surface resistivity of A40/NP composites is in good agreement with Arrhenius relationship when the temperature is above 40°C, which demonstrates that a liner relationship exists between surface resistivity of composites and  $1/T$ .<sup>15,23</sup> In this condition, electric resistivity is dominated by thermal motion. The surface resistivity of A80/NP composites also shows a good Arrhenius relationship with temperature above 30°C as shown in Figures 3 and 4.

With the increase of temperature, both the mobility of molecular chains and the diffusion of Na<sup>+</sup> cations enhance, which also



**Figure 6.** The tensile strength and elongation at break of the A80/NP (100/x) composites with different NP contents. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 7.** Shore hardness of A40/NP and A80/NP composites with different NP contents. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

reinforces the interaction between NP and  $-C-O-C-$  in BMEP and thus reduces the interaction between Na<sup>+</sup> cations and polymer composites and releases more Na<sup>+</sup> cations. The faster migration of ions will lead to the decrease of surface resistivity. In addition, the increase of temperature will result in the flexing of polymer chain, thus more free volume is produced, which leads to the enhanced polymer segment mobility. Figures 3 and 4 also demonstrate that the PVC composites will keep good antistatic properties at the temperature range of 25–80°C, which guarantees that these composites can be applied in the field that a lower surface resistivity is requested at a higher temperature.

### Mechanical Properties

To find out the influence of TiO<sub>2</sub> or SiO<sub>2</sub> content on the tensile strength and elongation at break of PVC/AP composites, the mechanical properties are tested in ambient environment. As shown in Figure 5, the tensile strength of A40/NP composites gradually increases to the maximum as the TiO<sub>2</sub> content is 2 phr and the SiO<sub>2</sub> content is 5 phr. However, the lowest tensile strength of A40/NP composites remains higher than that of A40 composites without any NP fillers. The elongation at break also achieves the maximum value as the NP content is 2 phr. And for composites with higher NP contents, the elongation at break decreases sharply. As for A80/NP (100/x) composites in Figure 6, the tensile strength of A80/NP (100/x) composites gradually increase to the peak as the addition of NP is 5 phr, while the elongation at break of A80/NP (100/x) is maximum when the NP content is 2 phr. As can be seen from Figure 6, when the NP content is 5 phr, the elongation at break of A80/NP composites decreases sharply, though the tensile strength is optimal. On the whole, the optimal mechanical property is obtained as the NP content is 2 phr.

The increase of mechanical properties can attribute to the interaction of NP with Na<sup>+</sup> cations and BMEP chain which promotes a network in the PVC matrix, thus the NP is hard to fall off from the matrix by the external force. Traditionally, NP is functioned as excellent reinforcing materials for polymer

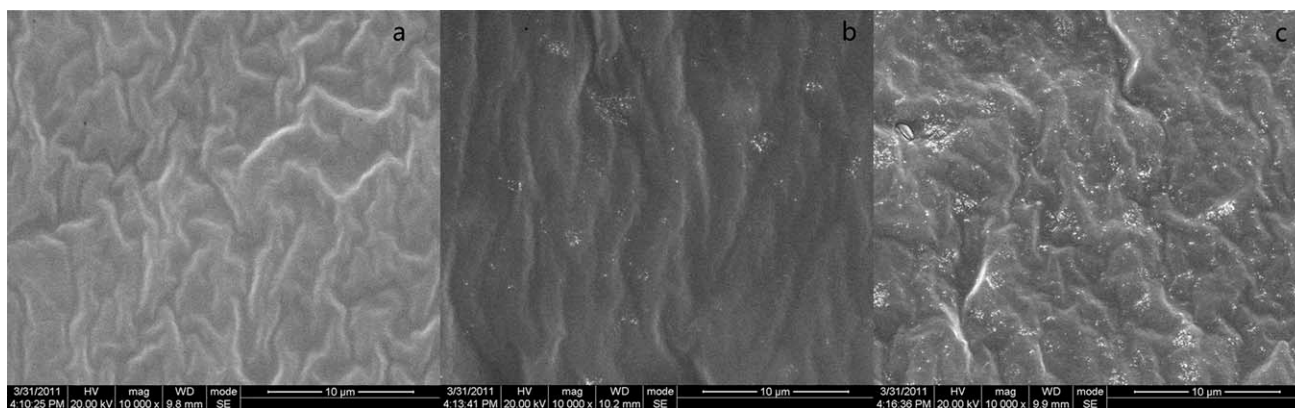


Figure 8. SEM micrographs of A40/SiO<sub>2</sub> (a) SiO<sub>2</sub>: 0 phr, (b) SiO<sub>2</sub>: 2 phr, (c) SiO<sub>2</sub>: 10 phr.

materials,<sup>24</sup> however, the aggregation of NP will occur as the additive amount gets larger, which will be confirmed by SEM micrographs later. Aggregated fillers will function as points of stress concentration so that the elongation at break of PVC composites will get worse when the addition of NP is too much. Earlier discussion demonstrates that the addition of 2 phr NP to PVC composites can effectively improve the antistatic property, which is also favorable for the enhancement of mechanical properties.

Figure 7 demonstrates that with the addition of NP content, the hardness of composites increases. Owing to the existence of other plasticizer DBP in A80/NP composites, the shore hardness of A80/NP composites is lower than that of A40/NP composites. The shore hardness of A40 composites by introducing different contents of either TiO<sub>2</sub> or SiO<sub>2</sub> shows similar variation trends. However, the hardness of A80/SiO<sub>2</sub> is higher than that of A80/TiO<sub>2</sub> composites, which demonstrates that SiO<sub>2</sub> is more favorable in enhancing the shore hardness of A80 composites.

### SEM Investigation

Since the antistatic and mechanical properties achieve a relatively optimum value as the NP content is 2 phr and the addition of SiO<sub>2</sub> shows better effect on the properties of PVC composites. In addition, the properties of A40 change larger along

with the addition of NP than that of A80. Therefore, the SEM observations of A40 blended with different contents of SiO<sub>2</sub> composites are shown in Figure 8(a-c). The surface of the pure A40 composite shows a relatively rough and uneven topography as can be seen in Figure 8(a). As for A40/SiO<sub>2</sub> (100/2) composite as shown in Figure 8(b), almost no aggregation exists in fractured face and the surface of composite is more smooth than that of the pure A40 composite. Because of the existence of hydroxyl radical on the surface of SiO<sub>2</sub>, the addition of 2 phr of SiO<sub>2</sub> into A40 composite is propitious to form hydrogen bond with the polar group in A40, which enhance the interaction between nano SiO<sub>2</sub> and A40 matrix. Therefore, nano SiO<sub>2</sub> can well disperse in A40 composite when the additive amount is 2 phr. When nano SiO<sub>2</sub> is 10 phr as shown in Figure 8(c), the aggregation of SiO<sub>2</sub> in A40 matrix is obvious, which leads to the worse antistatic and mechanical properties of A40/SiO<sub>2</sub> (100/10) composites than that of A40/SiO<sub>2</sub> (100/2) composites. The morphology observation results of A40 blended with different contents of SiO<sub>2</sub> composites indicate that composites with better interface structure may have better antistatic and mechanical properties as we have mentioned earlier.

### DSC-TGA Analysis of PVC/AP/NP Composites

As have been discussed earlier, the optimal antistatic and mechanical properties of PVC composites as the NP content is 2

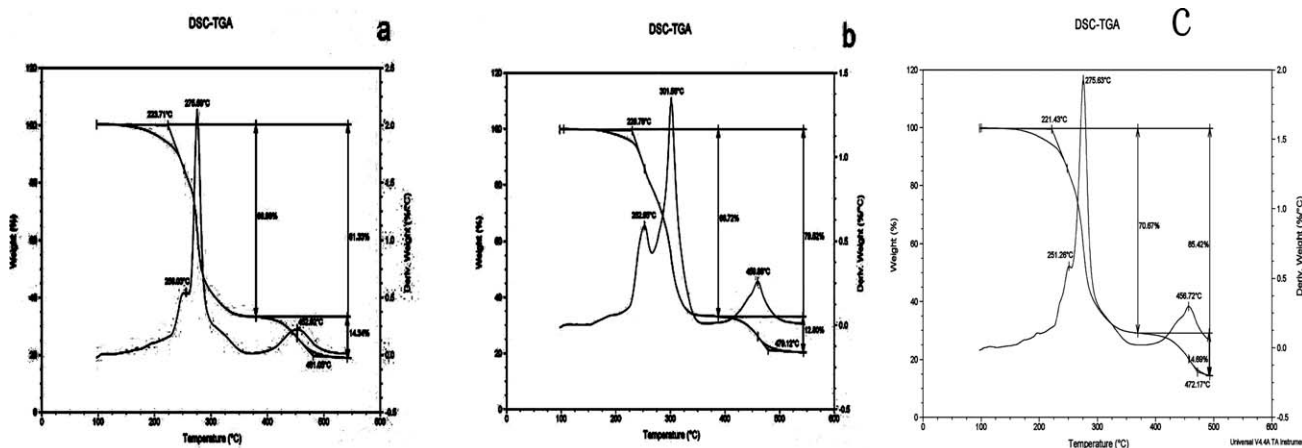


Figure 9. DSC-TGA thermographs of A40/NP (a) SiO<sub>2</sub>: 2 phr, (b) TiO<sub>2</sub>: 2 phr, (c) NP: 0 phr.

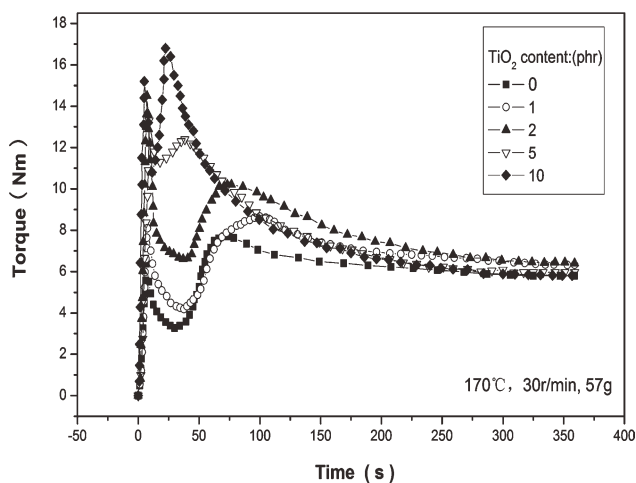


Figure 10. Fusion curves of the A40/TiO<sub>2</sub> with different TiO<sub>2</sub> contents.

phr in A40 composites. Therefore, the DSC-TGA analysis is introduced to identify the thermostability of A40/NP (100/2) composites. As shown in Figure 9(a–c), the initial and maximum decomposition temperature has a little improvement when the SiO<sub>2</sub> content is 2 phr comparing with that of pure A40 composites. However, the maximum decomposition temperature of A40/TiO<sub>2</sub> (100/2) composite is 26°C higher than that of A40/NP (100/0) composites, which illustrates that the addition of TiO<sub>2</sub> is favorable for the enhancement of the thermostability of A40 composites. These results can attribute to the excellent heat stability and heat resistance of nano TiO<sub>2</sub>. Moreover, the dispersion of TiO<sub>2</sub> in PVC matrix restricts the thermal motion of polymer chain, which may enhance the decomposition temperature of PVC composition.<sup>25,26</sup>

#### Rheological Properties of the Processing of Antistatic PVC Composites

The continuous stable fabrication of the thermal plastic polymers is the most important property for their final use which is usually demonstrated by thermal processability. As shown in

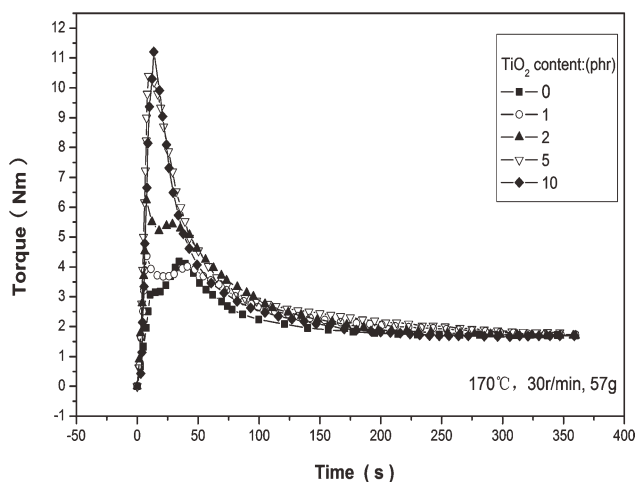


Figure 11. Fusion curves of the A80/TiO<sub>2</sub> with different TiO<sub>2</sub> contents.

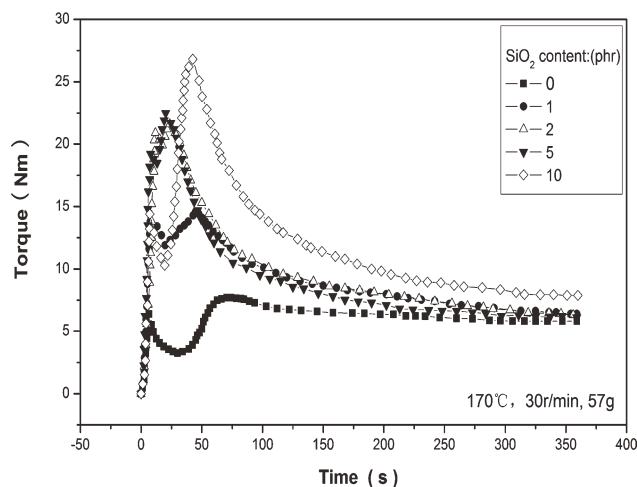


Figure 12. Fusion curves of the A40/SiO<sub>2</sub> with different SiO<sub>2</sub> contents.

Figures 10–13, the fusion peaks of composites will be brought forward and the peak intensities improve with the increase of NP content. However, the torque balance of both A40/NP (100/x) and A80/NP (100/x) composites has no significant change with the increase of NP content. Since the change of torque balance reflects the change of melt viscosity of PVC composites<sup>27</sup> that the melt viscosity has no significant change with the increase of NP. Thus, the proper addition of NP is favorable for the thermo processability of composites. Because of the additional 40 phr DBP in A80/NP (100/x) composites, the torque balance and plasticizing time is lower than that of A40/NP (100/x) composites. For instance, as shown in Figures 10 and 11, the plasticizing time and torque balance of A40/TiO<sub>2</sub> (100/2) composites are 53 s and 6.2 Nm, respectively. However, the plasticizing time and torque balance of A80/TiO<sub>2</sub> (100/2) are 46 s and 1.9 Nm, respectively, which owing to the function of small molecule plasticizer as we have mentioned before. Thus, a conclusion can be drawn that the addition of NP will not affect the melt viscosity and the thermo processability of PVC/AP composites.

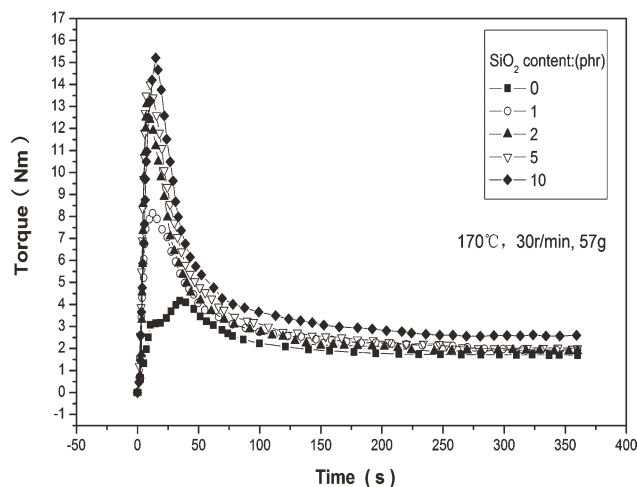


Figure 13. Fusion curves of the A80/SiO<sub>2</sub> with different SiO<sub>2</sub> contents.

## CONCLUSIONS

In this study, we concentrated our attention on studying the effect of NP on the properties of the permanently antistatic PVC composites prepared by dispersing NP into PVC-BMEP-NaClO<sub>4</sub> matrix with a Haake torque rheometer. Nano SiO<sub>2</sub> and TiO<sub>2</sub> are effective in decreasing the surface resistivity of soft PVC composites respectively. Meanwhile, NP is favorable for the improvement of mechanical properties. Especially, the surface resistivity of A40/SiO<sub>2</sub> (100/2) composite is 10<sup>8.78</sup> ohm/sq even at a RH of 0.1%. The mechanical properties and thermo processability of A40/SiO<sub>2</sub> (100/2) composite are also optimum. As for the DBP is added into PVC-BMEP-NaClO<sub>4</sub> matrix, it endows PVC composites with comparatively large free volume, the surface resistivity of A80/NP composites is much lower than that of A40/NP composites. Permanently antistatic PVC composites possessing better antistatic property, mechanical property and thermo processability are obtained by introducing 2 phr NP into the soft PVC/AP composites, which has potential application in electrical and electronic antistatic fields that request lower surface resistivity at a higher temperature.

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