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Electrical Properties Investigation in PA12/PANI Composites

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Summary: Volume conducting PA-12 based composites powders were chemically prepared by in situ polymerization and aniline doping at room temperature. These kinds of polyamide / PANI composites were investigated regarding their electrical properties. Their ac and dc electrical properties measured in the frequency range of 10^{-2}–10^{7} Hz are reported and the frequency dependence of electrical conductivity was investigated as a function of PANI concentration leading to the determination of the conductivity. The experimental conductivity was found to increase continuously with PANI content and explained by percolation theory with a relatively low percolation threshold of about 0.4 wt.%. The dielectric behavior of various PANI polymer composites has been characterized by the critical frequency ω_c (denoting the crossover from the dc plateau of the conductivity to its frequency dependent ac behaviour). Modelling the conductivity behavior versus volume fraction using Slupkowski approach has revealed that the considered parameters are not sufficient to describe the electrical conductivity behavior.

Keywords: composites; conducting polymers; electrical conductivity; PANI; percolation

Introduction

Substantial progress in conductive polymer composite materials is mainly due to the increasing importance of such materials for the electronic industry in the past few years.[1,2] Electrically insulating and non-magnetic nature of polymers can be modified by the introduction of conducting and/or magnetic fillers into the polymers. The obtained conductive composites are mainly used for electromagnetic and radio-frequency interference (EMI/RFI) attenuation; they are also used in applications that require electrostatic dissipating parts, low power resistance heaters and artificial dielectrics.[3]

Today's enormous theoretical and applied interests are concerned with the physics of electrical transport in conductive composites. For practical reasons, the composite effective conductivity dependence on the filler concentration has been primarily investigated in this type of materials. These systems exhibit commonly an insulator – conductor transition at a concentration threshold, which have been earlier elucidated in terms of percolation – like phenomenon. Reducing threshold concentrations by the elaboration of new composite materials represents a technological interest [1] since an excessive amount may affect other properties of the materials such as mechanical strength or color.[4]

For a random binary mixture of conducting and insulating hard spheres, much lower volume fraction of the conducting polymer were reported (sometimes far below 0.16 for a classical percolation estimate). Such a low content of a conducting polymer is desirable for applications where

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high mechanical properties and good electrical conductivity are required.[3–6] In this context, conducting polymers have attracted much attention not only from fundamental scientific interest but also from practical point of view for various functional applications. However, the non fusibility and non solubility in any solvents of most of them restricted their practical application, except for only few conducting polymers such as poly(3-alkylthiophene), poly(2,5-dialkoxy p-phenylenevinylene) and polyaniline which have found their application in the organic battery field, anti corrosion products (PANI), antistatic protection of photographic films (Polythiophènes) and packages for electronic components protection (PANI).[7–9]

Improvement of processability by various attempts to incorporate such conducting polymers in processable insulating matrices have lead to remarkable change in electrical conductivity at some concentration and was explained in terms of percolation process.[7]

The insulating nature of Polyamides, which are widely used in microelectronics and aerospace applications, may cause significant accumulation of electrostatic charge on their surface, resulting in local heating and premature degradation of electronic components or space structures.[10] This work describes a polyamide/ PANI composite having superior electrical conductivity level than polymer matrix which permits electrostatic discharge. In this paper we discuss the dependence of their electrical conductivity change on the microstructure and filler concentration.

Experimental Part

Polyaniline (PANI) as a conducting filler and polyamide (PA-12) as the polymer matrix (PM) have been chosen to study the properties of conducting polymer materials. The used PM is provided by Arkema as powder of 14 to 243 μm particle's diameter. The Aniline is reacted after additional purification with Dodecyl Benzene Sulfonic Acid (DBSA) and Ammonium PerSulfate (APS) as an oxydant.

The bulk conductive composite materials were prepared on the basis of the PA-12 powders of different dispersion. Organic acid (DBSA) was used as acid dopant.

For the preparation of the anilinium salt, the estimated amount of aniline was dissolved in the necessary volume of proper acid solution. The formed solution of the anilinium salt was vigorously stirred by magnetic stirrer for 1 hour. Then a definite amount of PA powders was added to this solution and the obtained dispersion was continuously stirred during 1 hour till the formation of homogeneous mixture. The solution of oxidant (water solution of APS) was next added to the dispersion. The obtained dispersion was left stirring for further 24 hours to ensure complete aniline oxidation process. After the completion of the process, the obtained dispersion of dark green colour was flooded with the 10-fold surplus of distilled water, and then the precipitated PA-PANI powder was filtered through a Buchner filter funnel. This is made for the removal of reagent livings soluble by-products. After the filtration the product slurry of the composite material was dried under vacuum at 70–80 °C for 24 h.

A part of resulting powders was redoped by stirring for 15 h in 0.2M water solution of appropriate acid producing doped PANI. Finally, the polymer doped powders were dried under dynamic vacuum at 70 °C for 24 hours till a constant weight.

A series of PA/ PANI composite pellets were prepared from obtained composite powder by pressing mould under 5 Tonnes of pressure with PANI concentrations ranging from 0.1 to 1 wt.%. The samples were disc shaped, 10 mm in diameter and thickness varied from 1.349 to 1.668 mm.

The low frequency (10^{-2}–10^{7} Hz) ac conductivity measurements were made using a Novocontrol broad band dielectric spectrometer. Gold circular electrodes are positioned onto both flat sides of the samples. The dc conductivities σ_{dc} of the composites were determined from the frequency dependences of the conductivity $\sigma_{ac}(f)$ as the value of the conductivity in the region of the low-frequency plateau.

Results and Discussion

This section describes the measured electrical conductivity versus the frequency for different amounts of PANI. An attempt to explain the observed behaviour based on the evolution of the filler arrangement within the matrix is proposed. A conductivity modelling based on microstructure parameters is used to give a best understanding of the conduction mechanism.

Figure 1 is a plot of the volumetric conductivity as a function of frequency for the PA/PANI composite system with different concentrations of PANI varying from 0.2 to 1 wt.%. The data are labelled in weight percents (X_c), but can be readily converted into volume percent by the following mixture rule:

$$\phi_c = X_c d_m / d_c \quad (1)$$

and

$$1/d_m = X_c/d_c + (1 - X_c)/d_p \quad (2)$$

where d_c, d_p and d_m are the specific densities of PANI, polyamide and the composite respectively. The implicit assumption is the existence of only two phases in the composite (PANI and PA without voids).

Since the densities of the PA and PANI compounds are approximately both equal to 1.02 g/cm^3,[11] volume fractions and weight fractions of PANI are not very different.

For the composite with relatively low PANI content (below 0.5 wt.%), the composite conductivity demonstrated the polarization coupling between the conductors and the continuous PA matrix. As expected, an increase in PANI concentration (0.5–0.6 wt.%) enhances the conductivity. The latter also increases as a function of frequency above 100 Hz, showing a typical dielectric behavior. Above 0.4 wt.%, a finite conductivity led to a plateau at low frequencies corresponding to the electrical response of the percolating network. The conductivity increased at higher frequencies due to the growing importance of the polarization effects on the macroscopic conductivity. Further increase in concentration (0.8–1 wt.%) increased the low-frequency conductivity and led to a broader plateau: in this concentrations range, the frequency dependence in conductivity decreases and shows a near constant conductivity for frequencies below 10 kHz. Similar results have frequently been reported and related to the formation

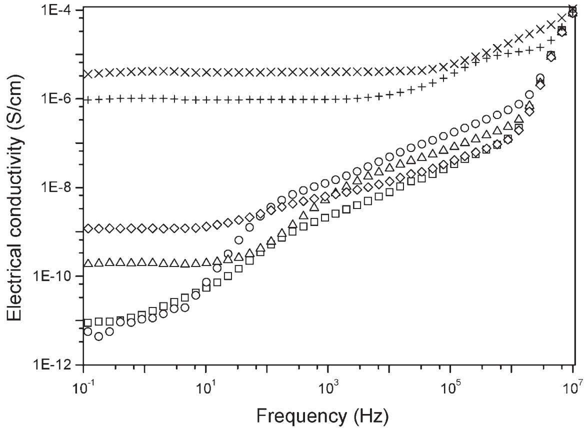

Figure 1.

Electrical conductivity profiles of PA/PANI system as a function of frequency at different wt.% content of PANI. □: 0.2%, ○: 0.4%, Δ: 0.5%, ◇: 0.6%, +: 0.8%, x: 1%.

of a percolating network in the material.[12,13] On the other hand, a jump in the conductivity at the concentration of 0.8 wt.% was measured.

Only the samples having a dc conductivity in the range 10^{-10}–10^{-5} $S \cdot cm^{-1}$ (concentrations above 0.4 wt.%) show a crossover from the dc plateau to dielectric behaviour within our experimental frequency window. As composites exhibit usually a typical dielectric behaviour below the conduction threshold, the results suggest that the conduction threshold resides between 0.4 and 0.5 wt %. Indeed, sample with 0.4 wt.% PANI content exhibits a beginning of dc conduction, i-e, a short plateau at very low frequencies (0.1–1 Hz). This result is clearly noticed on Figure 2-a representing dc conductivity versus low weight concentrations of PANI and confirms the already low value of p_c predicted by previous work [14] which reports electrical response versus frequency for PA/PANI composites with concentrations ranging from 1 to 10 wt.%: When conductivities of the PA-12/PANI-acid powders are plotted as a function of the PANI content in a semi- logarithmic scale, a sharp change in conductivity is observed at ~1 wt.% of PANI as shown in Figure 2-b. At this composition, the conductivity changes significantly (over more than 5 decades) compared to that of pure polyamide powder (0 wt.% PANI content). This value was primarily considered as the percolation threshold.

The conductivity shows the universal power law relationship expressed by (3) and (4), leading to a 0.6096 wt.% conduction threshold for PA12/PANI-DBSA composites.[14]

Figure 2-a represents a comparison between experimental and simulation data derived from classical percolation theory for concentrations ranging from 0.2 wt.% to 1 wt.%.

Using the following equations:

$$\sigma = \sigma_i (p_c - p)^{-s} \quad \text{for } p < p_c \tag{3}$$

$$\sigma = \sigma_c (p - p_c)^{t} \quad \text{for } p > p_c \tag{4}$$

where p is the volume fraction, p_c the percolation threshold, σ_i the matrix conductivity and σ_c the filler conductivity. t and s are critical exponents:

A best fitting is obtained for exponents $s = 1.06$ and $t = 2.19$ where percolation threshold is found to be about 0.004 of volume fraction. This value is less than the previously estimated for monodispersed particles.[14] Many researchers reported low values of percolation threshold for

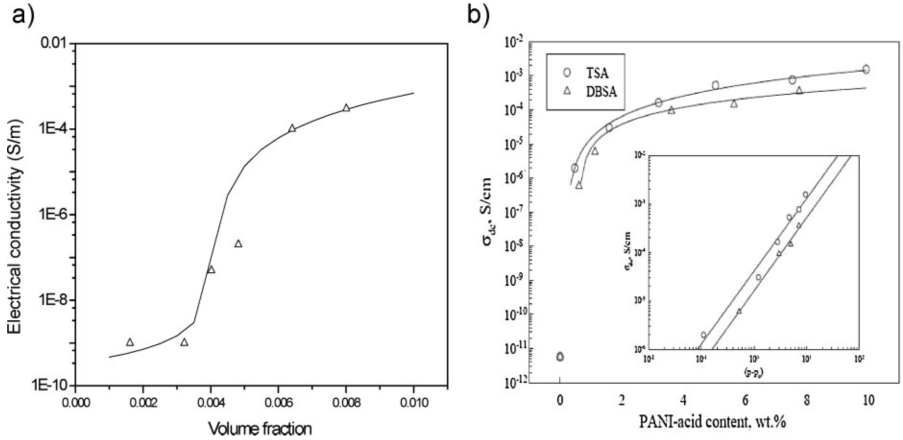

Figure 2.

Electrical conductivity versus PANI content (a) Low concentrations (0.2–1 wt.%) with polydispersed particles of PA-12 at ambient temperature, (b) High concentrations (1–10 wt.%) at T = 303 K with monodispersed particles of PA-12 [14] (5 μm of diameter); Δ : experimental points, —: percolation power law fitting.

composites showing segregation of filler particles around the insulating ones, forming by this manner a conductive shell.[15,16] This is the most interesting result in this study, since we reach the objective of the better estimation of percolation threshold by using low concentrations as well as lowering percolation threshold. The little discrepancy between found exponent values and those reported in the literature is probably due to the particulate microstructure of such composites. Polydispersity of insulating particles also may contribute to this disagreement. The exponent t is referred to as the morphological characteristic of the composite system. Most *t* values for composite systems lie in the "universality" range of 1.65 to 2.0,[17] however, *t* values which exceed this "universality" range can be accounted for by the extreme geometry of the conducting particle. Our composites ($t = 2.19$) adhered to that of Deprez and McLachlan which obtained values of 1.5–2.8 for synthetic graphite flakes.[18]

In summary, this material changes singularly with a variation of PANI content of only 0.5 wt.%, from a good insulating behavior to a rather good conducting behavior equivalent to that of totally agglomerated composite.

Figure 3 shows the frequency dependence of the electrical conductivity for redoped PA/ PANI samples. The real part σ' of complex conductivity $\sigma^* = \sigma' + j\sigma''$, remains nearly constant in the low frequency range up to some value ω_c where it starts to increase with increasing frequency for all samples, when the critical frequency ω_c (the frequency of transition from conducting to dielectric behavior) increases with the increasing dc conductivity. For charge carrier diffusion on percolation structures, this so-called crossover frequency ω_c is related to the transition from normal diffusion ($\sigma'(p < p_c) = \sigma_{dc} = \text{const}$) to anomalous diffusion ($\sigma'(p > p_c) \sim \omega^n$). The increase of conductivity value with the frequency is due to the presence of various kinds of inhomogeneity in the composite materials.

The poor frequency dependence of the conductivity at higher PANI-DBSA contents indicates that more homogeneous materials are produced.[19]

Table 1 gives ω_c values for different samples σ_{dc} values deduced from Figure 3.

All samples exhibit a dc plateau indicating their conducting behaviour even for concentrations below 0.4 wt.%. Figure 4 shows also power law behaviour of ω_c characteristic of percolating systems. The evolution of ω_c is linked to σ_{dc} (Figure 4-a). When this latter increases, the transition is shifted to high values of frequency, which is in turn related to PANI content (Figure 4-b).

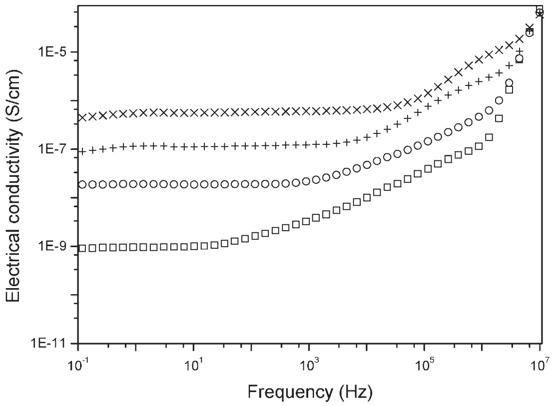

Figure 3.
A plot of the real part of the electrical conductivity against frequency for a series of redoped powder based Polyamide/ PANI samples. □: 0.2 wt.%, ○: 0.4 wt.%, +: 0.8 wt.%, x: 1 wt.%.

Table 1.
Cross over frequencies for samples with different concentrations of PANI.

σ_{dc} (S/cm)	1E-9 (0.2 wt.%)	3E-8 (0.4 wt.%)	1E-7 (0.8 wt.%)	7E-7 (1 wt.%)
ω_c (Hz)	12	170	1600	13000

High values of ω_c are observed for concentrations above 0.4 wt.% which is in accordance with the assumed conduction threshold.

Conduction mechanism

The low percolation threshold found is probably due to the segregation of PANI forming a thin conducting shell around PA particles [20–22] and thus occupying a very low fraction of the composite volume. When these shells are very thin and surround adjacent particles of PA, conducting paths can be established at low concentrations. It is expected that percolation threshold will depend on insulating particles diameter (core) and shell thickness. The latter were the two controlling parameters of Slupkowski's model:[23]

$$\sigma = 2\pi\sigma_c \frac{d([x]+P)}{D\ln\left(1+\frac{D}{([x]+1)d}\right)} \tag{5}$$

where σ is the specific conductance of the composite, σ_c the specific conductivity of PANI, d the diameter of the conducting particle, D the diameter of the insulating particle and [x] (the number of totally filled sublayers of thickness d) is defined by the following equation:

$$[x] = \left(\sqrt[3]{\frac{1}{1-V}} - 1\right)\frac{D}{2d} \tag{6}$$

P is the probability for the occurrence of a conductive network consistent with a percolation threshold and was obtained using the Scarsbrick model:

$$P = V^{V^{-2/3}} \tag{7}$$

where V is the volume fraction of conductive filler.

Simulation of figure 2-b data gives a best fitting of experimental results as shown in Figure 5.

For low concentrations, the best fitting parameters are those of case (b), while for high concentrations, values of case (a) are consistent with experimental results.

One can notice that at low PANI content, the assumed conductivity is low than the known intrinsic value. This is probably due to other parameters affecting the electrical conductivity which are not accounted for in the model, such as tunnelling resistance and constriction resistance. Previous works on PET/PANI films [24]

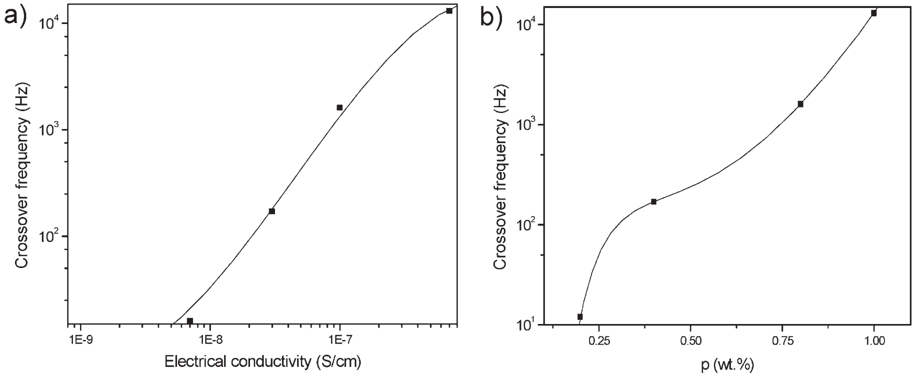

Figure 4.
log-log plot of cross over frequency ω_c versus σ_{dc} (a) and weight percentage of PANI (b); ■: Experimental data, —: Fit.

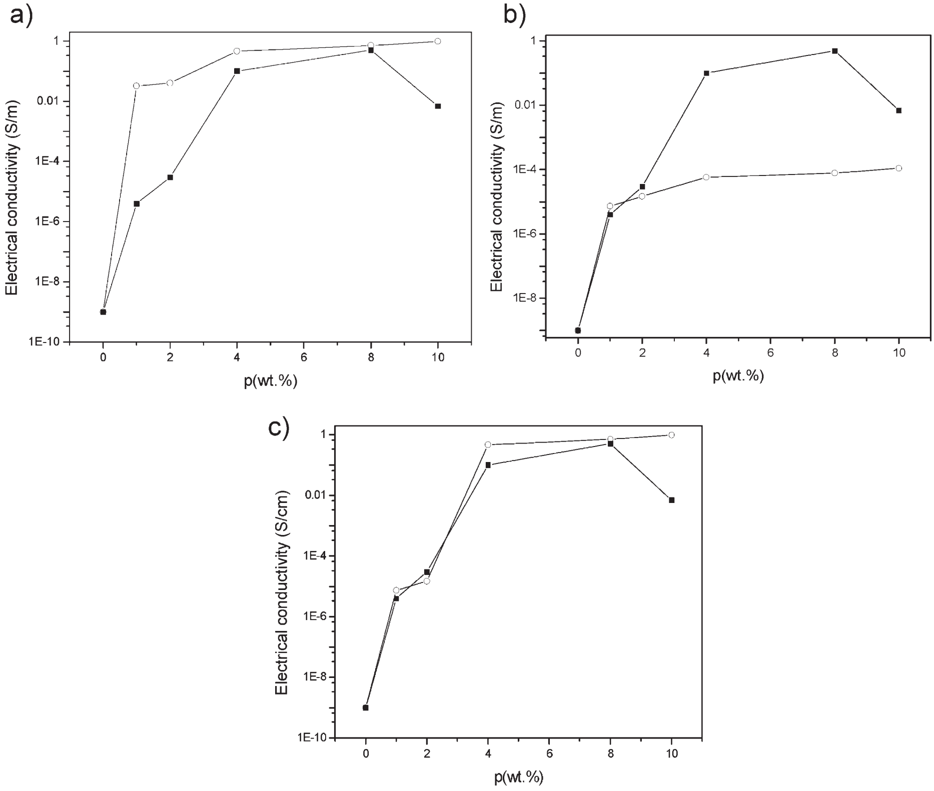

Figure 5.
log plot of electrical conductivity of PA powders versus volume fraction of PANI ○: Slupkowski model, ■: Experimental curve; (a) $\sigma_c = 49$ S/m, d = 15 nm, (b) $\sigma_c = 4{,}9.10^{-3}$ S/m, d = 6 nm, (c) combination of case (a) and (b).

have shown the existence of particles with different conductivities in the film's surface. The difference between the two assumed conducting particle diameters d may be due to the fact that PANI can agglomerate in non spherical shape. Another possibility is that polyamide particles are not totally covered by PANI [25] at low concentrations which gives average value of d different from the one obtained for higher concentrations.

By combination of the two cases, one can obtain the best fitting curve shown in Figure 5-c.

Numerous experiments proved that the percolation threshold is sensitively dependent on the diameter of particles.[3,7,26–28] The smaller particle size is the lower threshold is.

PANI particles diameter of about 10^{-8} m are found to give good simulation results. This value is in the range of those reported in the literature.[29]

We have also used the equation of logarithmic conductivity to investigate concentrations ranging between 0.2 and 1 wt.%:

$$\log \sigma = \log \sigma_i + (\log \sigma_F - \log \sigma_i) \cdot \left[\frac{p - p_c}{F - p_c}\right]^k \tag{8}$$

derived from Mamunya's model[30] (equation 9)

$$\log \sigma = \log \sigma_c + (\log \sigma_i - \log \sigma_c) \cdot \left[\frac{p - p_c}{F - p_c}\right]^k \tag{9}$$

where F is the packing factor and σ_F the conductivity at the maximum packing factor.

Eq. (9) differs from Eq. (4) by the use of the logarithmic conductivity (log σ instead of conductivity σ) resulting in the replacement of the critical exponent t by the exponent k. The value of k is not a constant for various composites and equals

$$k = K \cdot \frac{p_c}{(p - p_c)^n} \tag{10}$$

K is a value depending on the conductive phase topology and n is a constant equal to 0.75.[31] The topology, in turn, can be determined by the extent of interactions between the host polymer and filler surface and by other factors, for example, by shell structure formation.[17,25] Using the modified Mamunya model, good agreement of the calculated values with experimental results was achieved at $K = 0.4$ and $n = 0.8$ (Figure 6).

We note that the morphological exponent *t* remains constant for the percolation equation, where for the modified Mamunya (equations 8 and 10) and Additive (equation 11) models the exponent changes as the volume fraction changes.

$$\log \sigma = \log \sigma_i + H \cdot (p - p_c)^{\frac{G}{(p-p_c)^n}} + E \tag{11}$$

$$H = D.\log(\sigma_F) \tag{12}$$

D is an arbitrary constant.

$$G = K.p_c \tag{13}$$

The parameter E combines the structure and surface energy terms of the additive model which are related to aspect ratio, filler orientation and surface tension γ_{pf} between the polymer and the filler. Since all these factors are constant, E is taken to be a constant.

This model depends on at least two experimentally measured data points (σ_p at p_c and σ_F at F. For $p \le p_c$ σ is equal to σ_i [32]).

The conductivity of the composite σ in the modified Mamunya model is an interpolation between the conductivities of the highest volume fraction σ_F at $p = F$ (at the maximum packing fraction F) and the polymer σ_i at $p = p_c$ using two additional parameters (K and n). The modified Additive model has four parameters (H, G, E, and n) but is not exact at F.[17]

As shown in figure 6, the best fitting of experimental values is obtained for $H = 17.7$, $G = 1.5.10^{-2}$, $n = 0.42$, $E = 0$ and $p_c = 3.10^{-3}$

For the Mamunya and Additive models, the critical volume fraction is located where the electrical conductivity rapidly increases

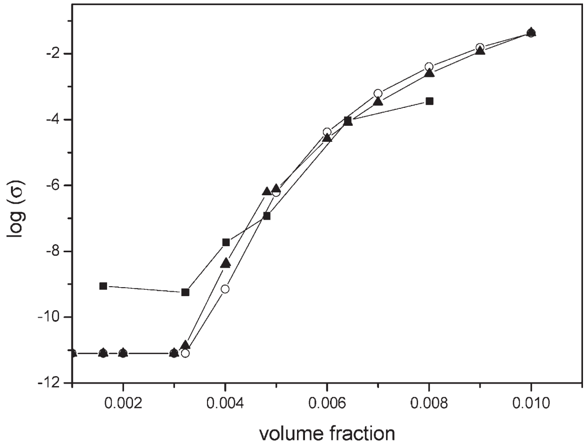

Figure 6.
log plot of electrical conductivity of PA/PANI composites versus volume fraction of PANI ■: Experimental curve O: modified Mamunya model, ▲: Additives model.

from the baseline polymer value; this explains the discrepancy near the percolation threshold.

Conclusion

In this study polyamide based conductive materials have been successfully fabricated by chemical method for better dispersion of PANI filler into the matrix.

PA/PANI composites were studied with respect to their electrical properties. The composites have the features of a low PANI content and a relatively high conductivity. These materials are insulators below the percolation threshold and the conductivity is 10^{-4} S/cm above the critical points.

Electrical properties of PANI reinforced polyamide composites were studied using ac measurement techniques. The electrical conductivity was studied over a wide frequency spectrum (10^{-2}–10^{7} Hz). Based on the experimental data, the following conclusions can be made.

(1) A drastic change in the electrical conductivity above some threshold concentration was observed. This result can be explained in terms of the percolation process. The percolation threshold of these composites could be observed at a PANI concentration of 0.4 wt.% which is a very interesting result

(2) The electrical conductivity of polyamide-PANI composites increases continuously with increasing PANI content, the conductivity in these composites is resulting from the formation of conducting paths into the polymer by the PANI. Depending on the concentration of PANI, it is possible to enhance the conductivity of the composite over many orders of magnitude.

(3) Other microstructure parameters controlling the electrical conductivity are diameters of particles constituting the composite. Such a low conduction threshold is observed in segregated conducting particles around insulating particles. Results derived from Slupkowski model assuming a core shell structure, fit reasonably experimental data and give an estimate of PANI particles diameter of about few 10^{-8} m.

(4) Equations of classical percolation theory gives values of exponents t and s in good agreement with those reported in the literature.

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