

Surfactant-Dispersed Carbon Black in Polyimide Nanocomposites: Spectroscopic Monitoring of the Dispersion State in the Polymer Matrix

Natthakarn Romyen, Supakanok Thongyai, Piyasan Prasertthdam

Center of Excellence on Catalysis and Catalytic Reaction Engineering, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University, Bangkok 10330, Thailand

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ABSTRACT: The effects of an anionic surfactant on the dispersion of carbon black (CB) for the purpose of forming conducting composite films were examined with ultraviolet–visible (UV–vis) absorption spectroscopy. To obtain a good dispersion and size reduction of aggregated CB in a polymer matrix, sodium dodecyl sulfate (SDS), used as a surfactant, was introduced into a CB suspension. A set of concentrations with various ratios of CB to SDS (ranging from 1 : 0.4 to 1 : 10) was established before mixing with poly(amic acid) (PAA), a precursor of pyromellitic dianhydride and oxydianiline, was performed. The CB/PAA solution mixtures were submerged under an ultrasonic bath for several hours, then cast onto dry plate glasses, and finally subjected to thermal imidization to produce CB/polyimide (PI) nanocomposite films with various CB weight fractions ranging from 0.025 to 0.50 wt %. A method for evaluating the absorbance at 500 nm of the CB/PI nanocomposite films was established. The absorbance of CB/PI nanocomposite

samples of various thicknesses was also normalized to get rid of the effects of the different thicknesses. UV–vis spectra showed that the minimum weight ratio of CB to SDS in the nanocomposite films that achieved well-dispersed CB and still had transparent properties was 1 : 2.0. Transmission electron microscopy demonstrated that CB was dispersed homogeneously in the PI matrix, and the size of the aggregated CB was affected by the amount of the surfactant. The dielectric properties of the nanocomposite films without the surfactant increased by approximately 2 orders of magnitude with an increasing mass weight fraction of CB and decreased when the surfactant was added. The surfactant also reduced the tensile strength of the CB/PI nanocomposites when the CB/SDS ratio was higher than 1 : 2.0. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 115: 1622–1629, 2010

Key words: polyimides; solution properties; surfactants; UV–vis spectroscopy

INTRODUCTION

Carbon black (CB)/polymer composites, including matrices such as poly(lactic acid), polypropylene, ethylene–octene elastomer, poly(vinyl acetate), ultra-high-molecular-weight polyethylene, and polymethacrylate,^{1–6} have already been studied. Furthermore, polyimide (PI) has been widely used in many applications because of its stability at high temperatures and excellent electrical insulation properties. Thus, PI has been used in many high-performance composites and in various microelectronics applications.^{7–9} Therefore, CB/PI composites may be interesting to scientists. However, the properties of CB/polymer composites are greatly affected by the manufacturing process and specific properties of CB and the matrix medium. During the incorporation of CB into polymer matrices, CB particles are quite prone to the for-

mation of primary aggregates because of their high interparticle force.¹⁰ Furthermore, these primary aggregates may combine to produce larger secondary structures, which often are called agglomerates.¹¹ To obtain a good dispersion and aggregate size reduction of CB in polymer matrices, the adsorption of a surfactant on the CB surface is one of the most favorable methods and is still attracting considerable attention from several researchers.^{12–16}

In general, a surfactant is a molecule that contains a hydrophobic hydrocarbon chain and a polar group (hydrophilic) in the same molecule. Surfactants can be classified into four classes: cationic, anionic, zwitterionic, and nonionic. As the concentration of a surfactant increases over the critical micelle concentration in a solution, the surfactant molecules combine together to form spherical aggregates (micelles) in which the core is populated with hydrophobic chains and the corona is populated by polar groups.²⁵ The stability of the dispersed particulate system depends strongly on the amount of the adsorbed surfactant layer and its hydrodynamic thickness.¹² With ultraviolet–visible (UV–vis) spectroscopy, several investigators have studied the quantification or characterization of dispersed

Correspondence to: S. Thongyai (tsupakan@chula.ac.th).

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carbonaceous materials by using a surfactant as a dispersing agent in aqueous, polar organic suspensions and in mixtures of water and polar organic suspensions.^{17–21} However, up to now, investigations using UV–vis spectroscopy to characterize the dispersion state of surfactant-aided CB composite film systems have still been rare.

In this article, using a UV–vis absorption spectroscopy technique, we present a novel method for the characterization of CB dispersed in CB/polymer nanocomposite films affected by an anionic surfactant. This concept is related to interrelationships between the agglomerate size and dispersability of CB and the spectral transmittance in CB/polymer nanocomposite film systems.

EXPERIMENTAL

Materials

CB (N 347 grade) was kindly provided by Thai Carbon Black (Bangkok, Thailand). The average diameter of CB was about 28 nm. The monomers for polymer synthesis were purchased and used without further purification: pyromellitic dianhydride (PMDA; Aldrich, USA; 97%), 4,4'-oxydianiline (ODA; Fluka, USA; 98%), and *N*-methyl pyrrolidinone (NMP; Merck, Germany; 99.5%). The surfactant used for the dispersion of CB was sodium dodecyl sulfate (SDS; Aldrich; 99.5%).

Preparation of the nanocomposite films

The CB/PI nanocomposites were synthesized by *in situ* polymerization with PMDA, ODA, and CB as the starting materials. SDS was employed as a surfactant to reduce the agglomeration of CB. First, CB was added to NMP with vigorous stirring at the ambient temperature. Then, the appropriate amount of the surfactant (SDS) was combined with a homogeneous CB suspension (0.15 wt/vol %) by ultrasonication (40 kHz) for 1–2 h to obtain a homogeneous CB suspension. A set of solutions with the concentration ratio of CB to SDS varying from 1 : 0.4 to 1 : 10 by weight was established.

The diamide (ODA) was completely dissolved in NMP with stirring to obtain an ODA solution. After the stirring, the homogeneous CB suspension was added to the ODA solution, and the solution was continuously mixed with stirring for 10 min before the addition of the dianhydride (PMDA) under an inert atmosphere at room temperature to obtain an approximately 15 wt/vol % solution. The reaction was carried out under an ultrasonic bath (40 kHz) under room conditions while the solution viscosity gradually increased and stabilized. The mixture was stirred continuously overnight to form a CB/poly(amino acid) (PAA) solution. The obtained solutions

had an inherent viscosity of approximately 17,000 Pa s. The PAA solution with CB was then cast onto clean, dry plate glass and subjected to thermal imidization at 100, 150, and 300°C for 0.5, 0.5, and 1 h, respectively, to produce CB/PI nanocomposite films with the CB concentration ranging from 0.025 to 0.5 wt %.

Characterization

Fourier transform infrared (FTIR) was performed to identify the structure of the synthesized polymer with a Nicolet (USA) 6700 spectrometer in the range of 4000–400 cm⁻¹ at a resolution of 2.0 cm⁻¹. UV–vis absorption spectra were recorded with a Perkin-Elmer (USA) model Lambda 650. The morphology and dispersion state of CB within the matrix polymer of the nanocomposite films was observed with transmission electron microscopy (TEM; JEM-2010, JEOL, Japan). The dielectric constants were recorded with an Agilent (USA) E4980A LCR meter at a frequency of 1 kHz and at a voltage of 1 V. Tensile properties were measured with an Instron (USA) universal testing machine with a test speed of 5 mm/min and a sample size of 2 × 10 cm². The tests were conducted according to ASTM D 882-02.

RESULTS AND DISCUSSION

The glass-transition temperatures of all the films were within the vicinity of 425°C, regardless of the percentage of CB in the matrix. The thermogravimetric analysis of SDS at 300°C showed 26 wt % remaining, which was enough weight for the remaining sodium and sulfur and some organic parts; therefore, the metal ions might not have interacted with carboxylic acid from PAA because there was some sulfur that counteracted the metal ions still remaining. The other interesting properties were found as follows.

FTIR spectra of the pure PI films

The FTIR spectra of the pure PI films are shown in Figure 1. The feature wave numbers near 1780, 1730, and 1370 cm⁻¹, which can be observed in the FTIR spectra, correspond to C=O (symmetric stretching), C=O (asymmetric stretching), and C–N (stretching), respectively, and are the common characteristic absorption peaks of the imide group.²² The FTIR spectra indicate that the imidization reaction was completed. Moreover, these results confirm that the PI films were successfully developed.

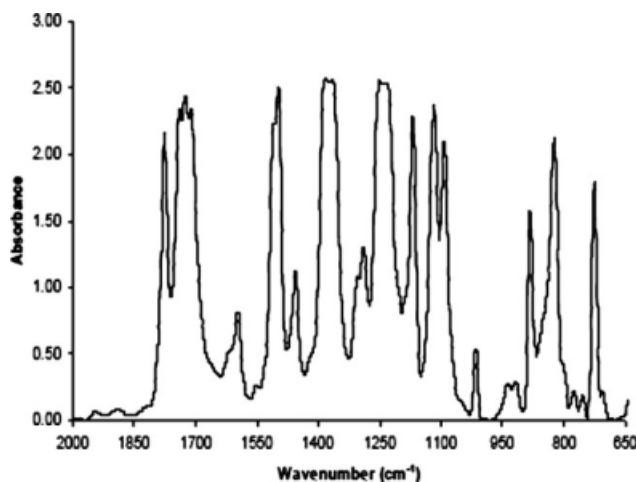


Figure 1 FTIR spectrum of the pure PI film.

Effects of SDS on the dispersion process of CB in solution

Figure 2 exhibits vials of the CB dispersion in NMP as an organic solvent (0.15 wt/vol %) with various amounts of SDS; the ratio of CB to SDS ranged from 1 : 0.4 to 1 : 100. All these suspensions were sonicated for 1 h before being used as a filler for the matrix medium.

After the sonication, the CB suspensions, stabilized with SDS, were kept for several months [Fig. 2(1–3)] as the absorption of SDS onto the CB surface resulted in an electrostatic repulsion force. However, a few carbon aggregates can be observed in Figure 2(1,2) because of insufficient surfactant, but most of the suspension was still homogeneous. In Figure 2(3), a homogeneous and single-phase suspension is shown. The appropriate SDS concentration, slightly below the critical micelle concentration, could have prevented carbon-carbon attraction and limited the aggregate size; this resulted in the stability of the CB dispersion. However, the CB suspensions shown in

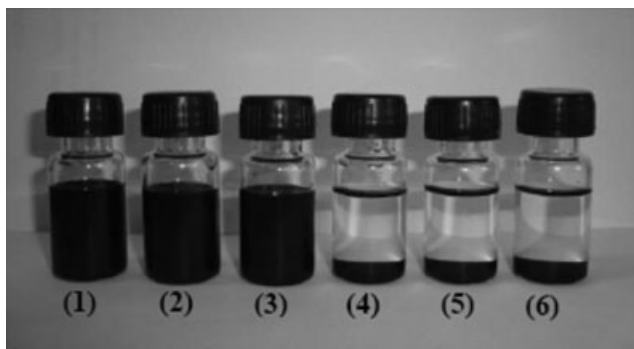


Figure 2 Vials (6 mL) containing CB suspensions (0.15 wt/vol %). The samples with CB/SDS ratios of (1) 1 : 0.4, (2) 1 : 0.8, and (3) 1 : 2 were imaged after sonication for 3 months, whereas the samples with ratios of (4) 1 : 5, (5) 1 : 10, and (6) 1 : 100 were imaged after sonication for 36–48 h.

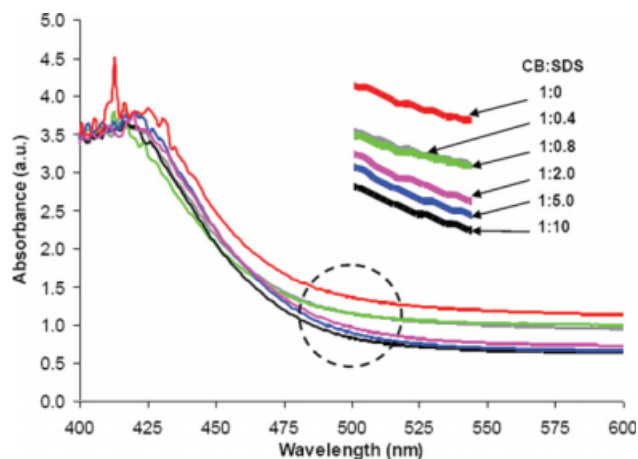


Figure 3 UV-vis spectra of the 0.5 wt % CB/PI nanocomposite films as a function of the CB/SDS ratio. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 2(4–6) contained an aggregation of CB at the bottom of their vials after 36–48 h because of the reduction of the electrostatic repulsion forces between CB. Similar results were also found^{23,24} in a dispersion of carbon nanotubes in an aqueous solution with SDS as a dispersing agent. Comparing the stability of all these suspensions, we can conclude that the stability trend was as follows: 3 > 2 > 1 > 4–6.

UV-vis spectra of the CB/PI nanocomposite films with various amounts of SDS

To investigate the effects of the surfactant on the CB dispersion in the composite films, UV-vis absorption spectroscopy and TEM were used. After thermal imidization, the synthesized films with various CB/SDS ratio were investigated with the scanning mode of UV-vis spectroscopy to search for a suitable wavelength for film analysis. The wavelength of 500 nm was found to be appropriate, as shown in Figure 3. The absorbances of the nanocomposites with different CB/SDS ratios began to approach equilibrium and were clearly separated from one another at the wavelength of 500 nm. Therefore, the absorbance at 500 nm was suitable for analyzing the films.

However, the synthesized films had various thicknesses ranging from 25 to 40 μm . Therefore, to eliminate the effect of the PI film thickness on absorbance, a linear calibration curve for pure PI was also performed at 500 nm as a reference, as shown in Figure 4. With 500-nm incident light, a calibration curve was built from the absorbance of a stack of commercial PI films, each layer of which had a constant thickness of 25 μm . The absorbance was measured at each layer of the commercial PI films in the stack, which increased from one layer to five layers.

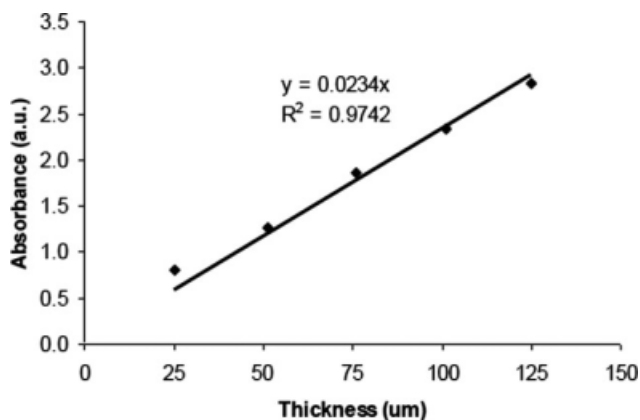


Figure 4 Calibration curves of pure PI at 500 nm (the absorbance value is denoted A_0).

The absorbance of the composite films with various ratios of CB to SDS at 500 nm is represented as A . However, because of the different thicknesses of these composite films, these A values should be normalized. To eliminate the effect of the thicknesses of various samples on the absorbance, each composite film thickness was substituted into the following equation to obtain the absorbance due to the thickness effect (A_0):

$$y = 0.0234x$$

where x represents the thickness and y represents the absorbance. A_0 was established from y as if A_0 came from the commercial PI of the same thickness. The normalization (A/A_0) was performed by the division of the absorbance of the composite film by the absorbance of the pure PI. The normalized values (A/A_0) were plotted as a function of various CB/SDS ratios to assess the degree of dispersion and the transparent properties, as shown in Figure 5.

From Figure 5, it can be clearly seen that the SDS concentration in the nanocomposite films was related to the alteration of the absorbance values. A small addition of SDS to the nanocomposite films caused a slight decrease in the absorbance values. The absorbance of the nanocomposite films was sharply diminished when the SDS concentration reached 2 wt % (CB/SDS = 1 : 2). At this point, the curve exhibited the most optimum ratio of CB to SDS for a good dispersion, an aggregate size reduction, and a balanced interaction within the matrix medium of CB in the nanocomposites, which affected the transparent properties. In contrast, when the SDS concentration grew greater than the optimum point, the absorbance of the nanocomposite films increased. This phenomenon demonstrated that at higher SDS concentrations, CB particulates were clustered and became larger and denser. Such

behavior can lead to considerable absorption in the spectra, resulting in a loss of transparent properties. This was also verified by TEM images of the PI nanocomposite films.

With the methods used for normalizing the absorbance, relation curves of the absorbance (A/A_0) and the mechanical and electrical properties of the CB/PI nanocomposite films were determined, as displayed in Figure 6. This figure clearly shows that if we know the normalized absorbance of a composite film, the properties of the film can be approximated directly.

Effect of SDS on the morphology of the CB/PI nanocomposite films

The effects of the SDS concentration on the dispersion state and agglomeration behavior of CB in polymer matrices were studied with TEM because it is a key factor that influences the composite properties. Figure 7 shows TEM micrographs of 0.5 wt % CB in a PI matrix with various ratios of CB to SDS. The entire specimens were prepared by surface cross-sectioning before analysis. Figure 7(a) exhibits a nanocomposite film without the surfactant; the cross-section fracture surface was rather rough (many agglomeration nodes) because of the presence of large CB particles in the form of agglomerates that were separated from the matrix. A similar result was also observed in nanocomposite films with CB/SDS ratios of 1 : 0.4 and 1 : 0.8 [Fig. 7(b,c), respectively]. With these amounts of SDS, the addition of SDS was not concentrated enough to isolate CB aggregates to form small particles. The result demonstrated that the dispersion of CB in the PI matrix was poor and inhomogeneous because of the weak

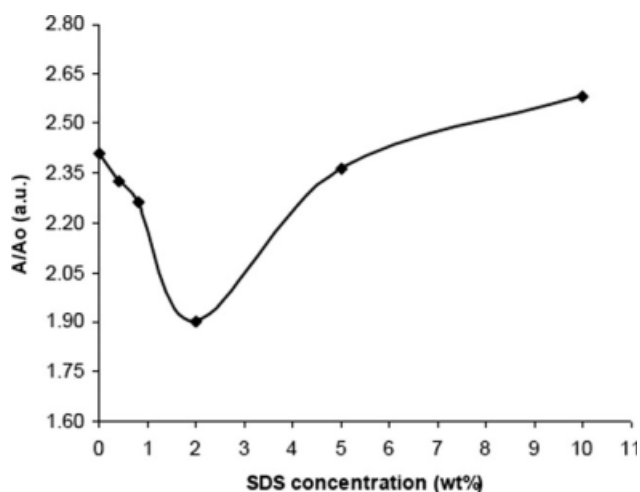


Figure 5 UV-vis spectra at 500 nm of 0.5 wt % CB/PI as a function of the SDS concentration (1 : 2 CB/SDS represents a 2 wt % SDS concentration).

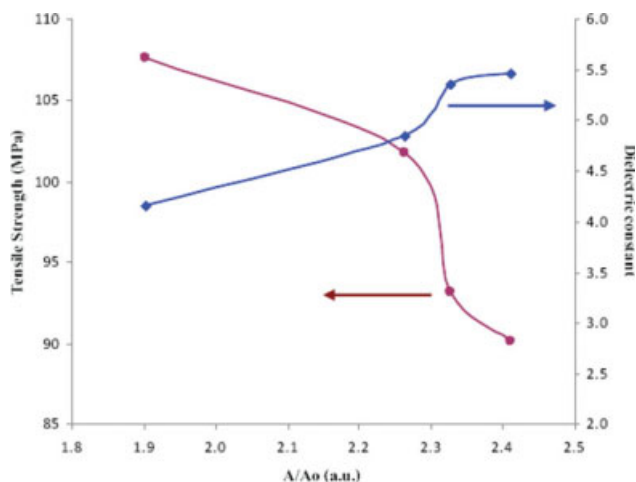


Figure 6 Tensile strength and dielectric constant of the 0.5 wt % CB/PI nanocomposites with various CB/SDS ratios as a function of the absorbance. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

interfacial interaction between the CB and polymer matrix. The TEM pictures of the composites with little SDS (1 : 0.4 and 1 : 0.8) look like the TEM picture of the composite with no SDS because of the inap-

propriate amount of SDS; thus, the absorbance (A/A_0) of the composites with little SDS was only slightly different from that of the composite with no SDS.

The TEM image of the nanocomposite film with a 1 : 2 CB/SDS ratio more clearly highlights the differences between the nanocomposites [Fig. 7(d)]. The obtained nanocomposite films had the smallest amount of CB aggregates (smallest aggregate nodes) in the homogeneous PI matrix, and this meant that the preparation of nanocomposite films with a 1 : 2 CB/SDS ratio in the presence of ultrasonication could overcome the interparticle force of CB. SDS could dramatically decrease the size of the CB agglomerates while increasing the dispersion of the CB and matrix because of the complete coverage of the CB surface by a thin surfactant layer, which caused electrostatic or steric repulsion reversed to carbon-carbon attraction. This clearly demonstrated that the CB and polymer were compatible with each other, and this resulted in a significant reduction of the absorbance value (Fig. 5; 1 : 2 CB/SDS ratio) and improved the transparent properties of the films. With a larger amount of SDS (>1 : 2 CB/SDS ratio), CB was reagglomerated and randomly distributed.

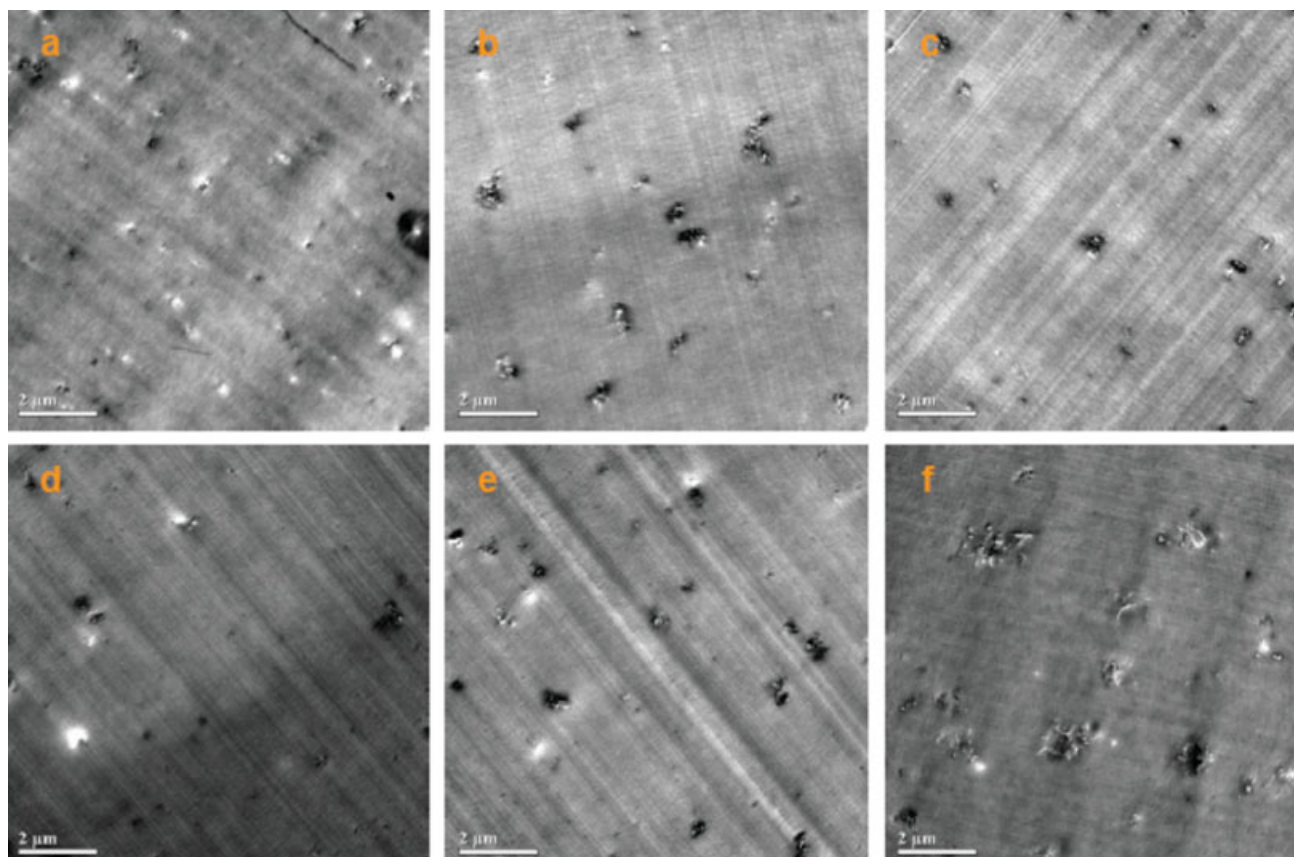


Figure 7 TEM images of 0.5 wt % CB/PI nanocomposite films (a) with no surfactant and (b–f) with various CB/SDS ratios (1 : 0.4, 1 : 0.8, 1 : 2, 1 : 5, and 1 : 10, respectively). The specimens were prepared by surface cross-sectioning. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

When the concentration of SDS was far over the critical micelle concentration point, CB tended to cluster and became aggregated in the suspension, which was probably prone to incorporation into the polymer matrix. Figure 7(e) shows inhomogeneous CB in the PI matrix, and the size of the microagglomerated CB was achieved when the ratio of CB to SDS was over 1 : 10 [Fig. 7(f)]. Thus, the absorbances of the UV-vis spectra for the nanocomposite films were increased at both concentrations. A lower viscosity liquid could ease the agglomeration of CB in comparison with a higher viscosity liquid because of Brownian movement.^{6,25} The differences in the distributed state, particle size, and compatibility with the polymer matrix come from the appropriate ratio of CB to SDS in the media.

Electrical properties of the nanocomposite films

It is well known that the incorporation of a conductive filler can greatly alter the electrical properties of a polymer medium. The conducting behavior of a CB-filled polymer matrix depends strongly on the gaps between aggregate structures. When the aggregates are closely packed, the conductive networks are well established throughout the matrix, and this allows electrons to pass the particles and tunnel between the gaps, resulting in the poor insulation properties of the PI nanocomposite. Figure 8 shows the dielectric constant of the CB/PI nanocomposite films without the surfactant as a function of the CB content. The addition of CB directly influenced the dielectric properties of the CB-containing nanocomposites. The dielectric constant of the nanocomposites increased with an increase in the CB content. The sharp increase when the mass loading of CB was up to 0.1 wt % suggested a critical value of the aggregates, which touched one another to form a

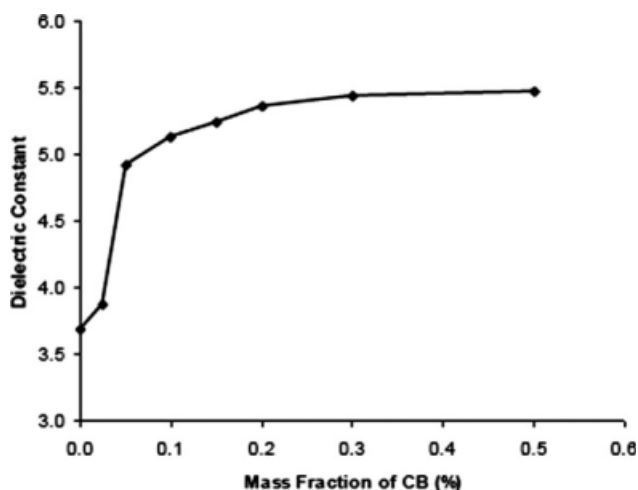


Figure 8 Dielectric constant of the CB/PI nanocomposites versus the content of CB (1 kHz).

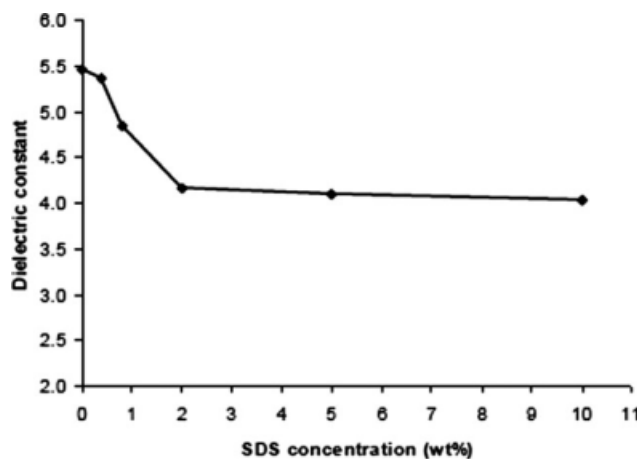


Figure 9 Dielectric constant of the CB/PI nanocomposites containing 0.5 wt % CB as a function of the SDS concentration (1 kHz).

conducting network also known as a percolated structure. When the CB concentration was increased further, the dielectric constant increased slowly because the electron tunnel became saturated.

Figure 9 shows the effect of the surfactant on the dielectric constant of the nanocomposites containing 0.5 wt % CB. The dielectric constant of the nanocomposites decreased with the content of SDS increasing and significantly declined from 5.46 to 4.16 when the SDS concentration was up to 2 wt %. This phenomenon was due to the reduction in the agglomerate size, and the good dispersion of CB in the polymer matrices remained and resulted in the electrical breakage of the network of conducting pathways; this made it difficult for the charge carriers to pass through the material.⁴ At the higher SDS concentration, TEM images of the nanocomposites containing SDS loadings of 5 or 10 wt % [Fig. 7(e,f)] show large aggregates of CB in the polymer matrix, but the dielectric constant of these composites gradually decreased. This phenomenon was related to the specific properties of SDS, which caused the dielectric constant of the pure PI films to decline from 3.50 to 3.09 when 0.5 wt % SDS was added to the PI matrix. Thus, we suggest that at the higher SDS concentration in the CB/PI nanocomposite films (CB/SDS ratio > 1 : 5), the dielectric constant was dominated by the influence of the surfactant. Regardless of the metal ions in the surfactant, the surfactant also contained sulfur, which had the opposite charge of the metal ion and stabilized the charge in the mixtures. The reduction in the dielectric constant might have come from the characteristics of the equal charge remaining in the surfactant in the final imidized films.

Mechanical properties of the nanocomposite films

The influences of the filler and surfactant contents on the mechanical properties of the reinforced polymer

TABLE I
Mechanical Properties of the CB/PI Nanocomposite Films

Composite	CB loading (wt %)	CB/SDS ratio (wt %)	Tensile strength (MPa)	Young's modulus (MPa)	Tensile strain at break (mm/mm)
Pure PI	0	0	84.60	2418.47	0.0683
CP1	0.2	0	88.01	2463.05	0.0908
CP2	0.5	0	90.11	2482.92	0.1189
CP3	0.5	1 : 0.4	93.14	2578.41	0.1004
CP4	0.5	1 : 0.8	101.76	2620.03	0.2004
CP5	0.5	1 : 2.0	107.60	2628.23	0.2200
CP6	0.5	1 : 5.0	92.52	2655.39	0.0927
CP7	0.5	1 : 10.0	87.57	2662.72	0.0746

were investigated, as shown in Table I. At least five specimens were tested for each loading fraction. It was clear that the incorporation of CB into PI improved the tensile strength and Young's modulus in comparison with the pure PI. As the amount of CB in the polymer increased, the tensile strength gradually improved from 84.60 MPa for the pure PI to 90.11 MPa for the 0.5 wt % CB/PI nanocomposites (CP2). At the same CB weight loading in the PI (the 0.5 wt % CB/PI nanocomposites), the tensile strength of the nanocomposites increased at a lower CB/SDS ratio but decreased with higher contents of SDS. The maximum value of 107.60 MPa for the nanocomposites with a 1 : 2 CB/SDS ratio was reached. To explain these results, it should be mentioned that, in relation to what can be observed in Figure 7, it has been widely accepted that interfacial interactions between the filler and matrix are an important factor affecting the mechanical properties of composites. Thus, by increasing the CB/SDS ratio in the nanocomposites, we improved the good dispersion states of CB and optimized them at CB/SDS ratios up to 1 : 2.0, as shown in Figure 7(a–d). The improvement in these dispersion states may have been caused by the strong interaction between CB and PI chains, which led to the improved tensile strength performance of the composites. On the other hand, with the higher CB/SDS ratio in the composites, the formation of aggregates was higher [Fig. 7(e,f)], and this diminished the interfacial filler–polymer adhesion and resulted in the reduction of the tensile strength. Young's modulus of the nanocomposites increased with the CB/SDS ratio increasing, and this indicated that the incorporation of the SDS surfactant into the nanocomposites made them more brittle.

CONCLUSIONS

A method for the evaluation of the absorbance of the CB/PI nanocomposite films was established. The effects of various thicknesses on the normalized absorbance could be eliminated. When only the normalized absorbance was known, the properties of the

CB/PI nanocomposites could be estimated directly. The effects of the SDS surfactant on the dispersion state and electrical and mechanical properties of the CB/PI nanocomposite films were explored. The UV–vis spectra and TEM images evidenced that the addition of the surfactant increased the level of dispersion of CB in the nanocomposite films because of the prevention of CB agglomeration and the improvement of the transparent properties. When the concentration of the surfactant was far above the critical micelle concentration, the dispersion state and agglomerate size of CB in the composite films were reversed. The dielectric properties of the CB/PI nanocomposite films without the surfactant increased with the CB loading increasing and decreased with the addition of the surfactant. The tensile properties of the CB/PI nanocomposite films were improved with the addition of the surfactant up to the 1 : 2 CB/SDS ratio. The incorporation of SDS into the CB/PI nanocomposites made them more brittle.

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