

# Compatibilization of Polyetherimide/Liquid Crystalline Polymer Blend Using Modified Multiwalled Carbon Nanotubes and Polyphosphazene as Compatibilizers

Ganesh C. Nayak,<sup>1</sup> Sumanta Sahoo,<sup>1</sup> Sukanta Das,<sup>1</sup> G. Karthikeyan,<sup>1</sup>  
Chapal K. Das,<sup>1</sup> A. K. Saxena,<sup>2</sup> A. Ranjan<sup>2</sup>

<sup>1</sup>Materials Science Centre, IIT Kharagpur, West Bengal, India

<sup>2</sup>Applied Chemistry Division, DMSRDE, Kanpur, India

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**ABSTRACT:** In this study, compatibilizing ability of silicone carbide modified multiwalled carbon nanotube (MWCNT) and polyphosphazene for the incompatible polyetherimide/liquid crystalline polymer (LCP) blend was investigated in detail. From rheological study, it was evident that the viscosities of binary and ternary blends were lower than those of the neat polymers, which signifying the great ability of LCP as a processing aid. Field emission scanning electron microscopic analysis revealed that the addition of

polyphosphazene and modified MWCNT, together, reduced the average domain size of LCP and improved the filler-matrix adhesion. Measurement of surface energy from contact angle measurement also point towards the improved interfacial interaction, in presence of compatibilizers. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 629–637, 2012

**Key words:** compatibilizer; polyphosphazene; PEI; LCP; modified carbon nanotube

## INTRODUCTION

Polymer blends comprised of thermoplastics and liquid crystalline polymers (LCPs) have been studied by many research groups intensively, for the last few years. The two most interesting factors that draws the attention of the researches towards these types of blend systems are (1) *in situ* fibrillation of LCP, which can reinforce the base matrix and enhance their mechanical properties,<sup>1–4</sup> (2) addition of small amount of LCPs to the thermoplastics can improve the processability of engineering plastics by reducing the melt viscosity of the blend system.<sup>5–7</sup> However, incompatibility between thermoplastic and LCPs is an issue of major concern and this lacuna need to be addressed in such a way so that we can achieve polymer blends having superior mechanical, thermal, and morphological properties. One way to solve the aforementioned problem is to use a compatibilizer. In recent years, a large amount of research focused on the compatibilization of LCP blends has been published.<sup>8–18</sup> Baird and coworkers used functionalized polypropylene, MAH-g-PP, as a compatibilizer for the polypropylene/LCP blends and they found improved interfacial adhesion due to some specific interactions like hydrogen bonding.<sup>11–13</sup> Seo demonstrated the compatibiliza-

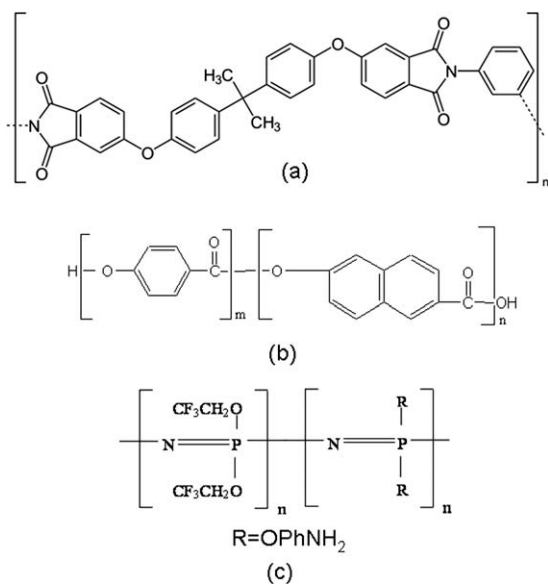
tion of PA6/vectra B950, PA46/Vectra B950, PBT/Vectra A950 blends by MAH-g-EPDM. According to Seo, some chemical reaction between MAH groups and LCP were responsible for the compatibilization.<sup>14–18</sup> In the previous works done at our laboratory, we had explored the effectiveness of polyphosphazene as a compatibilizer for the PES/LCP,<sup>19</sup> Polyetherimide (PEI)/LCP<sup>20</sup> and Nylon/LCP<sup>21</sup> blends. We found that incorporation of polyphosphazene reduced the particle size of the LCP domains indicating towards the improvement of compatibility between the blend partners. We had also studied the effect of silicone carbide (SiC) coated multiwalled carbon nanotubes (MWCNTs) on the properties of ABS/LCP<sup>22</sup> blend and improvement in the dispersion of modified MWCNTs was noticed in the blend matrix as compared with pure MWCNTs. In this work, an attempt has been made to explore the combined effect of polyphosphazene and SiC coated MWCNTs on the thermal, morphology and interfacial property of PEI/LCP blends. The goal of this study is to interpret the effectiveness of polyphosphazene and SiC coated MWCNT, as compatibilizers, for PEI/LCP blend.

## EXPERIMENTAL

### Materials used

PEI, (Ultem 1010), was obtained from General Electric Company. Liquid crystalline polymer (Vectra A950) was supplied by Ticona. This LCP is wholly

Correspondence to: G. C. Nayak (gcnayak\_bls@yahoo.co.in).



**Figure 1** Chemical structure of PEI, LCP, and Polyphosphazene.

aromatic copolyester containing 25 mol % of 2, 6-hydroxynaphthoic acid and 75 mol % of *p*-hydroxybenzoic acid.

Polyphosphazene, which has been used in this research work, was made by us in the laboratory of applied chemistry division, DMSRDE (Kanpur, India) by following a process already reported by Bose et al.<sup>20</sup> Polyphosphazene used in this research work is having inorganic backbone skeletal (P=N) with various organic pendant groups. The chemical structures of the components used, are given in Figure 1.

The MWCNTs (MWCNTs-1000) were procured from Iijin Nano Technology (Korea). The diameter, length, and aspect ratio were 10–20 nm, 20  $\mu$ m and  $\sim$  1000, respectively. The density of MWCNT is 2.16 g/cm<sup>3</sup>.

#### Modification of MWCNT with silicon carbide (SiC)

SiC coated MWCNTs are prepared following a method, already reported in literature.<sup>22</sup> The procedure for modification is as follows:

- i. Solid-state polycarbosilane (PCS), indigenously made by DMSRDE, Kanpur,  $M_w = 1800$ , was put in a beaker containing 50 mL of *n*-hexane, and ultrasonically dissolved using a horn type ultrasonicator.
- ii. Then, the MWCNTs were introduced into the PCS solution and then ultrasonically dispersed for 30 min. The weight ratio of PCS/MWCNTs was 3/7.
- iii. The resultant suspension was dried in a draft chamber at 25°C to remove *n*-hexane.
- iv. Then, the PCS-MWCNTs mixture was put into a quartz crucible and cured at 240°C for 90

min, to prevent agglomeration of PCS during subsequent high temperature treatment.

- v. Finally, the product obtained in the previous step was heat-treated at 1150°C in an oven for 1 h in argon atmosphere.

#### Preparation of composites

Prior to mixing PEI, LCP, and polyphosphazene were dried under vacuum at 80°C. The MWCNTs were dried at 200°C for 12 h under vacuum. PEI/LCP composites with polyphosphazene and SiC coated MWCNTs were prepared by melt blending in a sigma high temperature internal mixture equipped with two sigma type counter rotating rotors. The blending compositions were presented in the Table I. Blending was carried out at 330°C and at a rotor speed of 100 rpm. One set of pure PEI/LCP binary blend was also prepared by the same route for comparison. Samples for the mechanical testing were prepared by compression molding at 350°C using 10 MPa pressure and then rapidly cooled to room temperature.

### CHARACTERIZATION

#### Fourier transform infrared spectroscopy (FTIR)

FTIR analysis of blend systems were done using a NEXUS 870 FTIR (Thermo Nicolet) to investigate the possible interaction between PEI, polyphosphazene, and LCP.

#### Nuclear magnetic resonance (NMR) Study

<sup>1</sup>H NMR spectroscopy study was conducted with a Varian Mercury300 spectrometer using DMSO as solvent and TMS as reference.

#### Scanning electron microscopy (SEM)

The fractured surface of the blend systems were analyzed by using a Tescan VEGA LSU SEM. Before the analysis, the fractured surfaces were sputtered with gold for making the surface conducting.

**TABLE I**  
Sample Codes and Formulation of Nanocomposites

Sample Code	Liquid			SiC/ MWCNT (wt %)
	Poly (etherimide) (wt %)	Crystalline Polymer, LCP, (wt %)	Polyphosphazene (wt %)	
A	100	–	–	–
B	75	25	–	–
C	75	22.5	2.5	–
D	75	22.5	–	2.5
E	75	20	2.5	2.5

### Field emission scanning electron microscopy (FESEM)

A Carl Zeiss-SUPRA<sup>TM</sup> 40 FESEM with an accelerating voltage of 5 kV was employed to observe the morphology of the tensile fractured nanocomposites. A thin layer of gold was sputtered on the fractured surface of the specimens for electrical conductivity.

### Rheology

Rheological study was carried out in a capillary rheometer (Smart RHEO 1000, CEAST) at 330°C, at different shear rates, to investigate the effect of polyphosphazene and modified MWCNTs on the viscosity of PEI/LCP blend.

### Contact angle measurement

The contact angle is defined as the angle between solid surface and a tangent, drawn on the drop surface, passing through the triple-point air-liquid-solid. In this study, the geometric-mean equations were employed to calculate the dispersive and nondispersive components by using the following equation

$$(1 + \cos \theta)\gamma_l = 2(\gamma_s^d \gamma_l^d)^{1/2} + 2(\gamma_s n^d \gamma_l n^d)^{1/2} \quad (1)$$

where,  $\gamma_l$  and  $\gamma_s$  are the surface energies of pure liquid and solid, respectively, and  $\theta$  represents contact angle, which can be measured by the following equation:

$$\theta = \cos^{-1}(\cos \theta_a + \cos \theta_b)/2 \quad (2)$$

$\theta_a$  denotes advancing contact angle and  $\theta_b$  denotes receding contact angle. Contact angle measurement was carried out using a Kruss DSA15S instrument. The contact angle was measured for water and DMF and the respective surface energies were calculated.

### Mechanical properties

Tensile tests were carried out on dumb-bell shaped samples using a Hounsfield HS 10 KS (universal testing machine) operated at room temperature with a gauge length of 35 mm and crosshead speed of 5 mm/min. Tensile values reported here were an average of the results for tests run on at least four specimens.

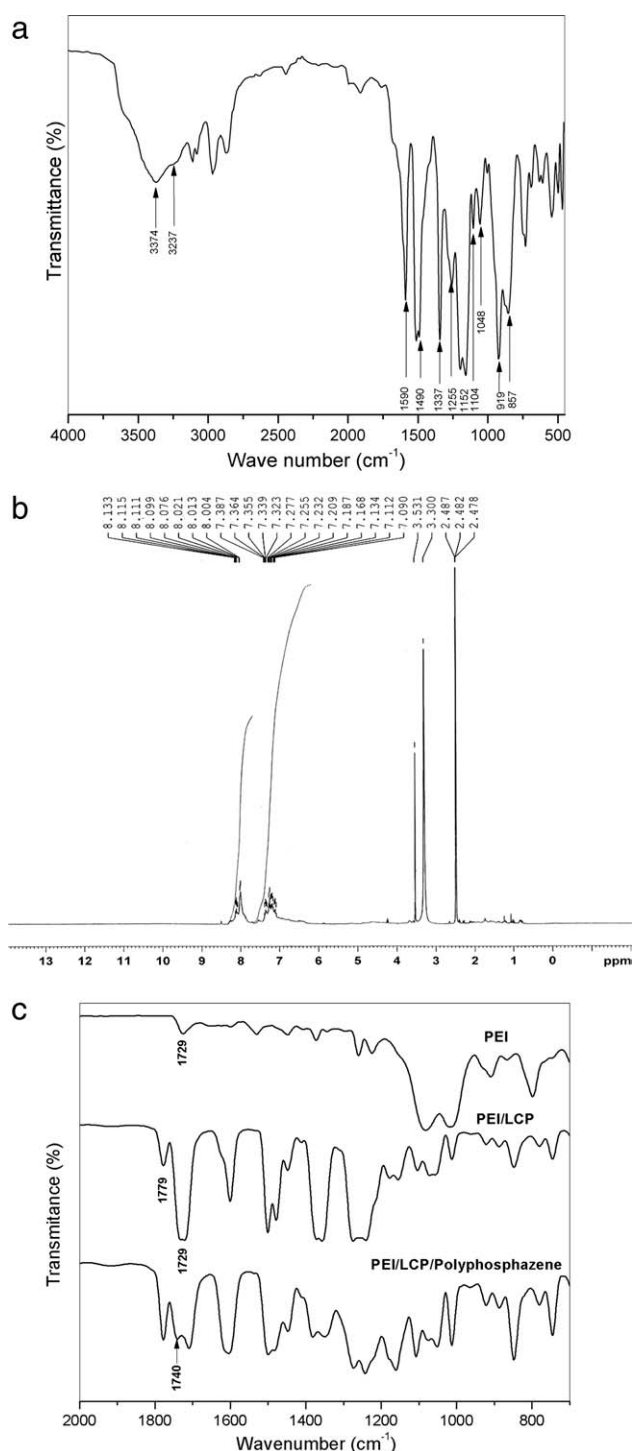
### Thermogravimetric analysis (TGA)

Thermogravimetric analysis curves were recorded with a Dupont 2100 thermogravimetric analyzer. The TGA measurements were conducted with a heating rate of 10°C/min under air atmosphere from 100 to 650°C.

## RESULTS AND DISCUSSION

### FTIR and NMR analysis

FTIR spectrum of polyphosphazene is presented in Figure 2(a). The FTIR peaks of polyphosphazene are summarized in the Table II. The presence of peaks corresponding to P=N, P-N, P-O, C-N, C-F, and



**Figure 2** (a) FTIR spectra of Polyphosphazene. (b) NMR spectra of Polyphosphazene. (c) FTIR spectra of PEI, PEI/LCP, and PEI/LCP/polyphosphazene blend.

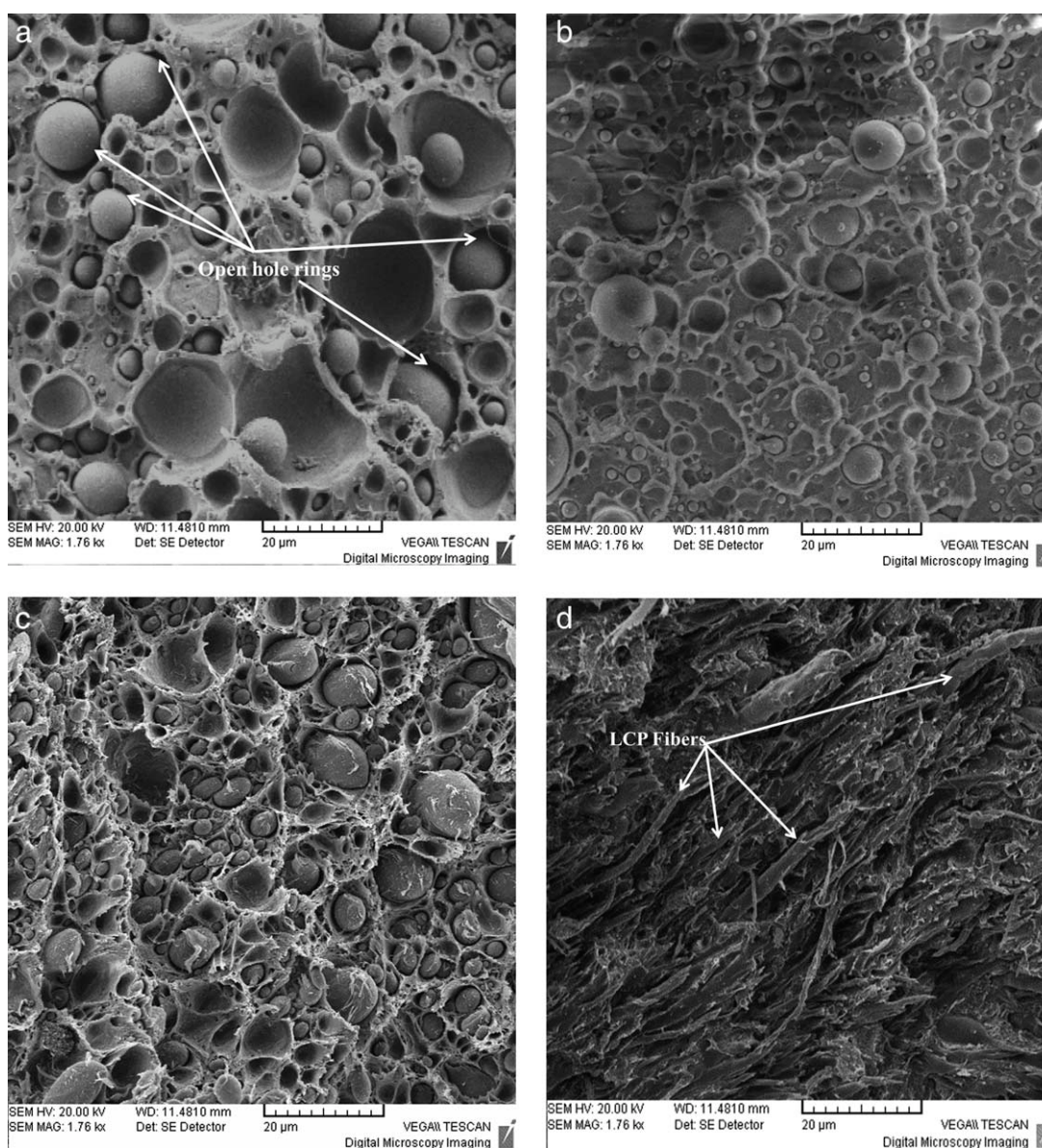


**TABLE II**  
**FTIR Analysis of Polyphosphazene**

Wavenumber (cm <sup>-1</sup> )	Functional groups
857	P—N Stretching
919	P—N—P Stretching
1048	P—OAr Stretching
1104	C—F Stretching
1152	P=N Stretching
1255	C—N Stretching
1337	C—CF <sub>3</sub> Stretching
1590, 1490	C=C stretching of phenyl ring
3374, 3237	NH stretching of amine

NH groups indicates the formation of polyphosphazene with aminophenoxy pendent group. Proton NMR spectra of polyphosphazene is presented in Figure 2(b). The triplet at 2.4 ppm is due to the proton of DMSO. The singlet at 3.5 ppm corresponds to the protons of fluoroalcohol. The multiplets centers around 7.3 and 8 ppm can be due to the protons of phenyl ring and amine group of *p*-aminophenoxy pendent group, respectively.

FTIR analyses of blends were carried out to investigate the interaction of polyphosphazene with the PEI and LCP and the result is shown graphically in



**Figure 3** (a) SEM image of PEI/LCP binary blend. (b) SEM image of PEI/LCP/Polyphosphazene ternary blend. (c) SEM image of PEI/LCP/SiC coated MWCNTs ternary blend. (d) SEM image of PEI/LCP/SiC coated MWCNTs/Polyphosphazene blend. (e) FESEM image of PEI/LCP blend. (f) FESEM image of PEI/LCP/Polyphosphazene blend. (g) FESEM image of PEI/LCP/SiC coated MWCNTs blend. (h) FESEM image of PEI/LCP/SiC coated MWCNTs /Polyphosphazene blend. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

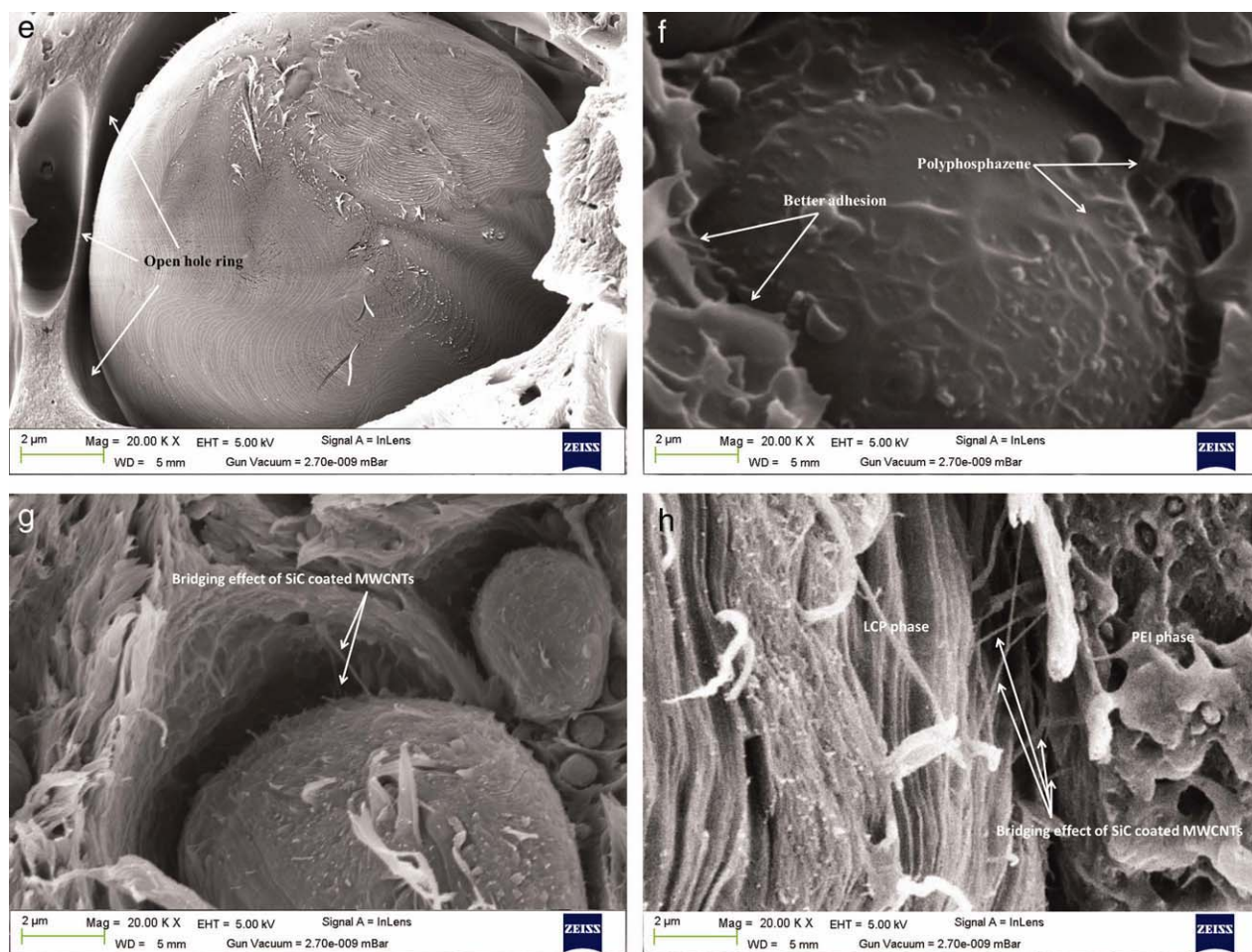


Figure 3 (Continued)

Figure 2(c). From the figure, it is clear that characteristic imide carbonyl peak of PEI appears at  $1729\text{ cm}^{-1}$  for both pure PEI and PEI/LCP binary blend system. The intensity of this peak is much higher than the peak intensity of pure PEI. This is due to the incorporation of LCP, which also contain the carbonyl groups. However, polyphosphazene aided blend system exhibit different spectrum behavior, that is, the imide carbonyl stretching frequency of PEI shifts towards lower frequency region ( $1712\text{ cm}^{-1}$ ) and noticeable change in peak intensity is observed. Another significant observation is splitting of the carbonyl peak, which suggests some chemical interaction between the amine group of polyphosphazene and carbonyl group of PEI and LCP. Henceforth, it may be interpreted that, polyphosphazene is acting as a compatibilizer for the incompatible PEI/LCP blend system.

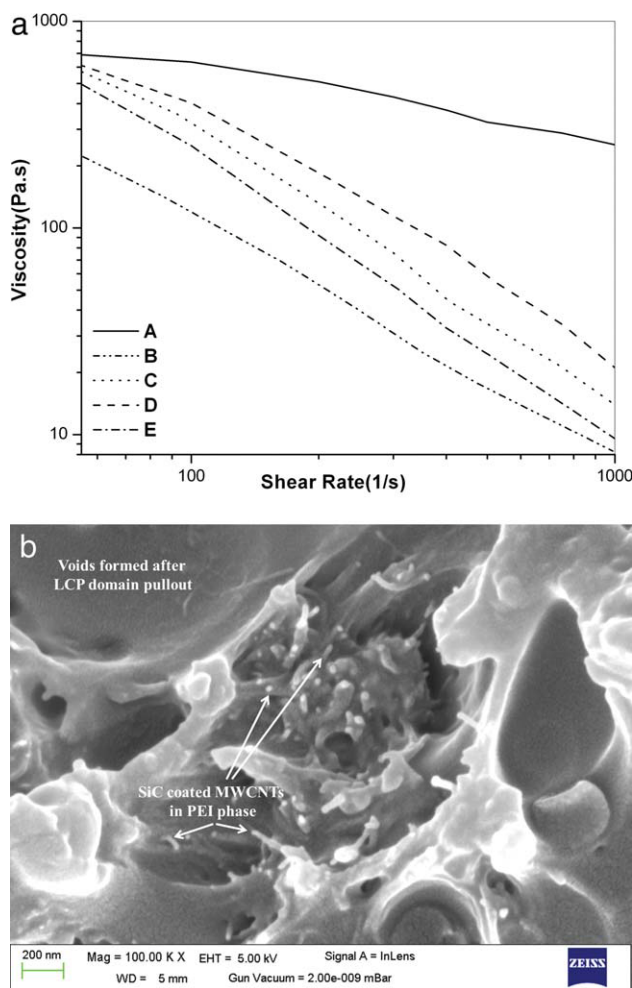
### Morphological study

Figure 3(a–d) shows the fractured surfaces of the noncompatibilized and compatibilized PEI/LCP blends. The domain size of LCP in noncompatibi-

lized binary blend [Fig. 3(a)] is larger as compared to the ternary blends, indicating poor dispersion of LCP. The holes in the micrograph of PEI/LCP binary blend formed due to the LCP domain pullout demonstrated a poor adhesion between the two phases. In contrast, the fracture surface of polyphosphazene compatibilized blend system [Fig. 3(b)] shows better adhesion between the blend partners, characterized by the reduced LCP domain pullout from the PEI matrix. Moreover, the particle size of LCP is finer as compared to uncompatibilized blend. The interfacial adhesion between PEI and LCP is ascribed to the chain interactions of the compatibilizer with the blend partners indicated by FTIR analysis, which shows that there is an interaction between the polyphosphazene and  $\text{C}=\text{O}$  of PEI and LCP.

Addition of SiC coated MWCNTs to the PEI/LCP blend system deforms the spherical LCP domains to slightly elliptical shape [Fig. 3(c)]. The no of voids was also decreased as compared to the binary blend system. However, Figure 3, shows addition of both polyphosphazene and SiC coated MWCNTs produced long LCP fibers. To explain the observed





**Figure 4** (a) Rheological properties of pure PEI and its blend with LCP. (b) FESEM image showing the distribution of SiC coated MWCNTs in the PEI phase. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

morphologies, the interface of the uncompatibilized and compatibilized blends was analyzed by FESEM and the images are presented in Figure 3(e–h). As can be seen the PEI/LCP blend demonstrate poor adhesion between the two phases, which leads to an open ring hole around the LCP domains [Fig. 3(e)]. In contrast, Figure 3(f) shows better adhesion for the polyphosphazene-compatible blend and the surface of LCP is rough in nature. The two phases were well adhere to each other, characterized by the absence of open ring hole. This observation along with the FTIR data confirms that polyphosphazene is acting as a compatibilizer for the incompatible PEI/LCP blend, which results in improved adhesion and decreased LCP pullout.

FESEM image of PEI/LCP/SiC coated MWCNTs blend is depicted in Figure 3(g). As can be seen an open ring hole is visible around the LCP domain but the two phases were bridged by the nanotubes. This bridging effect probably reduced the LCP domain

pullout. Another noticeable feature is LCP domains show some fibrillation on their surfaces. This fibrillation can be developed due to the presence of SiC coated MWCNTs at the interface, which were having a rough surface and enhanced the drag force of PEI upon the dispersed domains. This drag force can form some fibrillation on the LCP surface. However, addition of both polyphosphazene and SiC coated MWCNTs shows better adhesion [Fig. 3(h)]. The bridging effect of SiC coated MWCNTs is clearly visible. This drastic change in the microstructure can be ascribed to the synergistic effect of polyphosphazene and SiC coated MWCNTs, which step up the interfacial adhesion and restricts the interlayer slippage, respectively. Due to this combine effect, the drag force of highly viscous PEI matrix on the dispersed LCP domains might be sufficient to deform into the fibrillar form.

Since the drag force of PEI matrix on the LCP is an important parameter for the deformation of spherical domains, rheological analysis of the blend system can give further information on the development of observed morphology. The rheological characteristics of the blend systems were discussed in the following section.

### Rheological study

Figure 4(a) depicts the viscosity plots of different blend systems along with pure PEI. Binary blend of PEI/LCP shows the remarkable reduction of viscosity as compared to pure PEI. This reduction in the viscosity can be ascribed to the lower viscosity of LCP as well as the interfacial slippage arising due to the incompatibility between the blend partners.<sup>23</sup> Addition of polyphosphazene increases the viscosity of the ternary blend above the binary blend system. The location of the compatibilizer at the phase boundary together with chemical interaction with the two phases reduced the free volume and the chain mobility which induces the increased viscosity. Incorporation of SiC coated MWCNTs shows higher viscosity as compared to PEI/LCP binary and PEI/LCP/polyphosphazene ternary blend system. This enhancement in the viscosity can be attributed to the restricted interlayer slippage arising due to the

**TABLE III**  
Surface Energy Estimation

Sample Code	Contact angle (Degree)		$\gamma_s^d$ (mJ/m <sup>2</sup> )	$\gamma_s^{nd}$ (mJ/m <sup>2</sup> )	$\gamma_{total}$ (mJ/m <sup>2</sup> )
	$\theta_{water}$	$\theta_{DMF}$			
B	60.7	30.1	17.3	23.3	39.6
C	66.3	50.3	12.2	23.0	35.2
D	68.3	56.5	10.2	23.3	33.5
E	71.2	60.1	8.5	25.1	31.6

bridging effect at the interface (discussed in the FESEM section). Apart from restricted interlayer slippage, the distribution of SiC coated MWCNTs in the bulk matrix can also influence the viscosity. Since the viscosity of LCP is much lesser than the PEI, the distribution in the PEI phase will affect the viscosity of the blend system more as compared to that in the LCP. The distribution of SiC coated MWCNTs in the PEI matrix was analyzed by FESEM, which shows that the nanotubes were dispersed in the PEI matrix [Fig. 4(b)]. This result is in well accordance with the previous work reported by Fenouillot et al., where they found the nanofillers more preferably distributed in the high viscous medium.<sup>24</sup>

However, addition of both polyphosphazene and SiC coated MWCNTs shows lowest viscosity among all the blend system. This contradicts the previous phenomenon, where the reduction in the interlayer slippage leads to the reduction in the viscosity because as discussed in the FESEM section this blend system has the better interfacial adhesion due to the combine effect of polyphosphazene and SiC coated MWCNTs. Probably here the dominating factor is the complete fibrillation of LCP. Since the fibrillation of LCP was achieved during blending, the fibers can align along the flow direction during the capillary rheometer analysis. Similar type of effect was also reported by Kim and Kim who ascribed the reduced viscosity of poly(ethylene 2,6-naphthalate)/LCP blend to the improved fibrillation of LCP.<sup>25</sup>

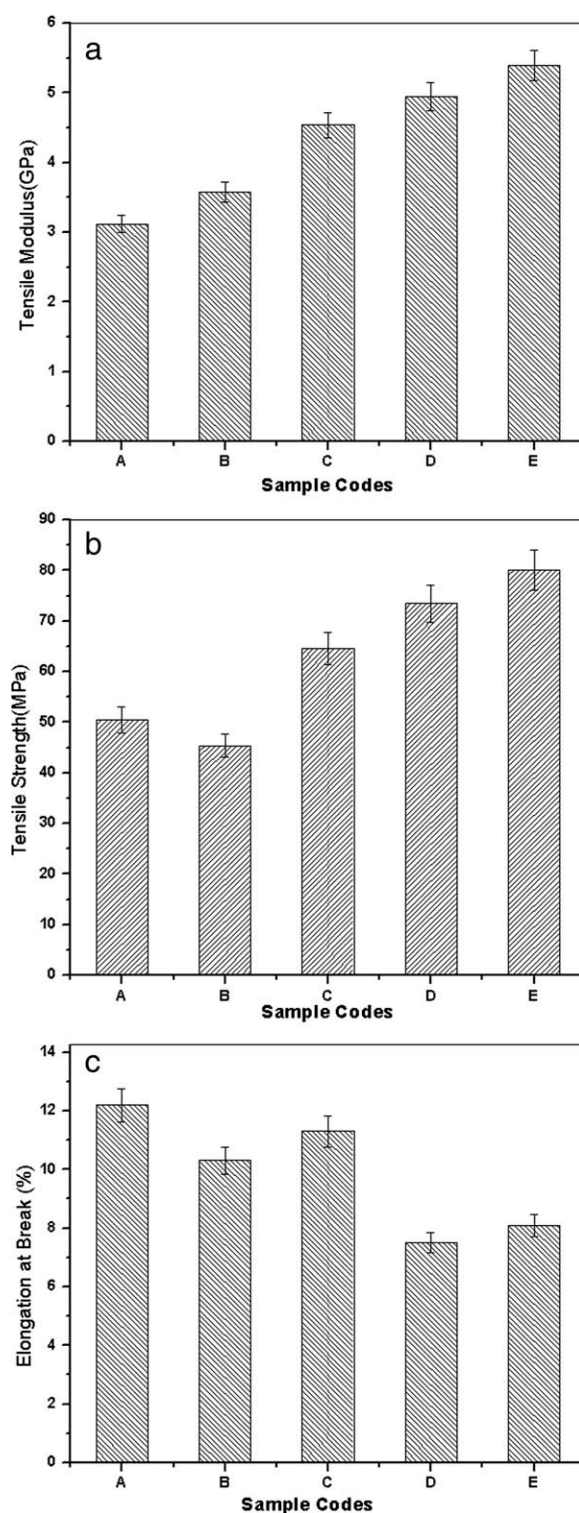
### Estimation of surface energy

Variation in surface energies with contact angle is shown in Table III. From the Table III, it is evident that the surface energies of PEI/LCP/SiC coated MWCNTs and PEI/LCP/polyphosphazene blend system are lower than the PEI/LCP binary blend system. This supports the SEM results where addition of polyphosphazene and SiC coated MWCNTs enhanced the interfacial adhesion by reducing the interfacial tension. However, blend containing both SiC-coated MWCNTs and polyphosphazene shows lowest surface energy, which further add to the effect of polyphosphazene in improving the interfacial adhesion of the concerned blend.

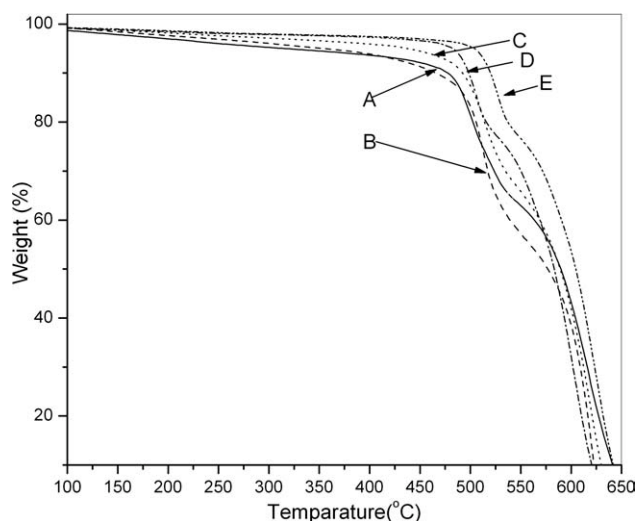
### Mechanical properties

Variations in mechanical properties of pure PEI along with the blends were shown in Figure 5(a–c). In PEI/LCP binary blend system, tensile strength decreases as compared to pure PEI matrix, indicating the poor adhesion between the matrix and dispersed phase. In case of PEI/LCP/polyphosphazene blend system, the tensile strength and tensile modulus were found to be increased in comparison to

PEI/LCP blend system suggesting an enhancement of interfacial adhesion between the PEI and LCP matrix, which helps in the stress transfer from the PEI phase to the LCP phase and thus improves both



**Figure 5** (a) Tensile Modulus of different PEI/LCP blend systems. (b) Tensile Strength of different PEI/LCP blend systems. (c) Elongation at break of different PEI/LCP blend systems.



**Figure 6** TGA analysis of pure PEI and its blend with LCP

tensile strength and modulus of the blend. The elongation at break also increased with the addition of polyphosphazene indicating the plasticizing effect of polyphosphazene in making the blend flexible in nature. The tensile strength and modulus of PEI/LCP/SiC coated MWCNTs blend system increased but elongation at break decreased as compared to PEI/LCP binary blend system. The probable reasons behind this enhancement in tensile strength and modulus are as follows<sup>26</sup>:

- i. Bridging effect of SiC coated MWCNTs at the PEI and LCP interface (discussed in the morphology section) that requires more energy to pull out the LCP phase and thus resists the fracture and increased the tensile strength.
- ii. Presence of SiC coated MWCNTs in the bulk matrix as well as at the interface enhanced the stress transfer from the matrix to the MWCNTs and to the rigid LCP domains, respectively, hence improves the modulus.

However, combine addition of SiC coated MWCNTs and polyphosphazene to the PEI/LCP blend shows a synchronous improvement in strength, modulus, and elongation at break. The enhancement in tensile strength and modulus can be ascribed to the better interfacial adhesion (due to polyphosphazene) and mechanical reinforcement offered by and SiC coated MWCNTs and rigid LCP fibers. However, the increase in elongation at break may be caused due to the plasticizing effect of polyphosphazene elastomer at the interface.

### Thermal stability

Thermal stability of the composites is graphically represented in Figure 6. As showed in the Figure 6,

binary blend of PEI/LCP followed a two-step degradation process at 452 and 527°C. Addition of 2.5% of polyphosphazene and SiC coated MWCNTs to the PEI/LCP blend, increased the first onset degradation temperature to 470 and 478°C, respectively, which suggested that the polyphosphazene and SiC coated MWCNTs aided blend systems were more thermally stable as compared to the uncompatibilized blend system. However, addition of both SiC coated MWCNTs and polyphosphazene to the PEI/LCP blend, improved the thermal stability of the composite to 498°C. The superior thermal stability of PEI/LCP/polyphosphazene/SiC coated MWCNTs blend can be apparently attributed to the restricted motion of polymer chains due to SiC coated MWCNTs<sup>27</sup> and compatibilization effect of polyphosphazene between the PEI matrix and dispersed LCP phase.<sup>20</sup>

### CONCLUSIONS

Ternary blends of PEI/LCP with polyphosphazene and SiC coated MWCNTs were prepared by melt blending. Rheological study confirms that viscosity of ternary blends exhibit higher value in comparison to binary blend system. Combination of polyphosphazene and SiC coated MWCNTs in PEI/LCP blend has improved the tensile strength and tensile modulus as compared to binary and ternary blends as well as pure PEI. In presence of polyphosphazene, reduction in average particle size of LCP was observed as compared to PEI/LCP binary blend. FESEM study reveals that the fibrillation of LCP in presence of SiC coated MWCNTs and polyphosphazene.

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