

Synthesis and Characterization of Novel Poly(dimethylsiloxane)/Polyindole Composites

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ABSTRACT: A series of composites of polyindole (PIN) and poly(dimethylsiloxane) (PDMS) were synthesized chemically using FeCl_3 as an oxidant agent in anhydrous media. The composites were characterized by FTIR and UV-visible spectroscopies, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), X-ray diffraction (XRD), elemental analysis, inductively coupled plasma-optic emission spectroscopy (ICP-OES), magnetic susceptibility, stress-strain experiments, and conductivity measurements. The conductivities of PIN at different temperatures were also measured and it was revealed that their conductivities were slightly increased with increasing temperature. More-

over, the freestanding films of PDMS/PIN composites were prepared by casting on glass Petri dishes to examine their stress-strain properties. From thermogravimetric analysis results it was found that PDMS/PIN composites were thermally more stable than PIN. Thermal stabilities of PDMS/PIN composites increased with increasing PIN content. It was found that the conductivities of PDMS/PIN composites depend on the indole content in the composites. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 121: 1600–1609, 2011

Key words: conducting polymers; composites; mechanical properties; thermal properties

INTRODUCTION

There are many application areas of the conducting polymers. The most exciting developments are the new class of semiconducting devices such as plastic batteries,^{1,2} sensors,^{3,4} conductive surface,⁵ magnetic recording, and solar cells.⁶

In recent years, several conducting polymers have received increasing attention as material coatings for the protection of common metals against corrosion. In particular, polyaniline and polypyrrole have been found to reduce sufficiently the corrosion of steel and other oxidizable metallic materials.^{7–11} Several reports have been published on polyindole and its derivatives despite its close structured similarities with polyaniline and polypyrrole.^{12–15}

Among conducting polymers polyindole (PIN) has relatively been less studied. *N*-containing heteroaromatic organic molecules have very interesting properties. The chemical or electrochemical oxidation of these molecules yields conducting polymers that could find an enormous amount of application in the electronics as new materials. Indole-based some

articles have also been reported in potential applications such as linear and nonlinear optical properties,¹⁶ light emitting oligomers,¹⁷ and dye-sensitized solar cells.¹⁸ Polyindole is an electroactive polymer, which can be obtained from electrochemical oxidation of indole or chemical oxidation using FeCl_3 or CuCl_2 .¹⁹ However, only little investigations have been made on chemically synthesized polyindole.^{20–23}

Polyindole has air stability. Its conductivity is about 10^{-3} – 10^{-1} S cm^{-1} depending upon the synthesis technique and the nature of dopant ions and in its doped state, polyindole has a green color. It is also reported that polyindole films have the advantages of fairly good thermal stability, high redox activity, and slow degradation rate in comparison with polypyrrole and polyaniline.^{24,25} However, there were no reports on the electrosynthesis of PIN film on stainless steel. It is usually obtained by the anodic oxidation of indole under suitable conditions.²⁶ To analyze the polymerization mechanism of indole, several spectroscopic and theoretical calculations have been carried out. As most likely coupling sites, 1 and 3 positions,^{26–28} 3-6 positions,²⁹ 2-3 positions^{30,31} and 3-3, 2-2 couplings³² were suggested. Although recent semiempirical theoretical calculations were indicating 2 and 3 positions of the monomer as the most likely coupling sites, the level of theory used was not adequate to explain the complexity of the processes, and more accurate calculations are required.

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Poly(dimethylsiloxane) (PDMS) is one of the most widely used polymer materials in chemical, biological, and medical sciences because of its excellent properties such as convenient processability, chemical stability, and optically transparency. The most attractive of this material lies in its biological compatibility and gas permeability, making it suitable for biological applications.^{33,34}

PDMS is a homopolymer with repeat units consisting of $-(O-Si(CH_3)_2)-$. A fundamental property of polysiloxane family is the highly pronounced inherent conformational flexibility of the thin main-chain backbones, which results in the high mobility of thin segments and entire molecules.³⁵ Some physical and chemical attributes of PDMS are, compared to other polymers: it maintains a rubbery, amorphous structure at room temperature and a low glass transition temperature ($T_g \approx -125^\circ C$ ³⁶). PDMS can sustain large elastic deformation with low Young's modulus and tensile strength. As a typical elastomer, molecular weights or crosslinking densities can greatly influence the mechanical properties of neat PDMS.³⁷⁻³⁹

In the present work, we report the results obtained via chemical synthesis and the characterization of novel poly(dimethylsiloxane)/polyindole composites using poly(dimethylsiloxane) as insulator polymer matrix. To the best of our knowledge, there are no reports in the literature on the synthesis and characterization of novel poly(dimethylsiloxane)/polyindole composites.

EXPERIMENTAL

Materials

Indole (99% pure) and $CHCl_3$ (99% pure) was supplied from Merck (Germany). Anhydrous $FeCl_3$ (98% pure) was also supplied from Merck. Dimethylformamide (DMF) (anhydrous 99.8% pure) was purchased from Aldrich (Germany). Poly(dimethylsiloxane) was obtained from Across Organics (\bar{M}_w : 95,000 and \bar{M}_n : 30,000, $-CH_3$ terminated, NJ). Dimethyl sulfoxide (DMSO) (anhydrous, 99.9% pure) was supplied from Aldrich. The 1,1,2,2-tetrachloroethane was purchased from BDH Chemicals (UK). All chemicals were used as received.

Characterization

Conductivities of the samples were measured by PC Card 1716 PCLD 8710 model, a four probe instrument, on pressed pellets at room temperature and between 0 and $50^\circ C$. Magnetic susceptibility measurements were carried out using a Sherwood Scientific Model MKI Gouy scale (Sherwood, St. Louis, UK) at ambient temperature. Finely powdered polymer samples were placed into a glass tube at a height of not less than 2.5 cm. FTIR spectra of sam-

ples were recorded using a Mattson-1000 model spectrometer (Ati, Unicam Cambridge, UK) by the KBr pellet technique (under 5 tonne cm^{-2} pressure, diameter = 1.3 cm). Thermal analysis of the samples was taken on a TGA-DSC Instruments Perkin-Elmer Pyris Diamond model thermal analyzer (USA) under nitrogen atmosphere between 30 and $900^\circ C$ at a heating rate of $10^\circ C \text{ min}^{-1}$. X-ray diffraction patterns for PIN and PDMS/PIN were obtained by using a Rigaku D-Max B model diffractometer (USA) with $Cu K\alpha$ radiation ($\lambda = 1.54,018 \text{ \AA}$ at 40 mV and 40 mA). The powder forms of the samples were used for XRD experiments. ICP-OES of the samples was taken on a Perkin-Elmer ICP-OES 3300 DV model instrument (USA). Element analysis of PIN and PDMS/PIN were determined with LECO CHNS-932 model instrument (Midland, ON, Canada). The morphologies of the polymers were examined by Jeol JSM 6060 LV model SEM and the polymer films by Quanta 400F model SEM (Japan). The UV-vis spectra were recorded using ATI UNICAM UV-2 model spectrometer (Cambridge, UK). The stress-strain experiments of the polymers were performed by Instron 3367 model Creep Tester (USA).

Synthesis of polyindole

Polyindole were chemically synthesized using $FeCl_3$ as oxidant. The molar ratio of oxidant to monomer was taken as 5 : 2. About 0.039 mol (6.28 g) of $FeCl_3$ was dissolved into 35 mL of $CHCl_3$. The monomer solution into 10 mL $CHCl_3$ was added dropwise into oxidant solution. The reaction was continued under nitrogen atmosphere for 5 h. The pristine polymer was collected by filtration and then washed with distilled hot water ($70^\circ C$). Finally, the precipitate was dried in a vacuum oven at $70^\circ C$ for 24 h.

Synthesis of poly(dimethylsiloxane)/polyindole composites

PDMS of 0.41, 1.25, 2.00, 2.86, and 3.70 mL were dissolved in 40 mL of $CHCl_3$, respectively. In the synthesis of all composites, the same molar ratio of monomer to oxidant was taken as 2/5. First, $FeCl_3$ and PDMS were dispersed in chloroform in a three-necked flask. Then, indole monomer (dissolved in 10 mL chloroform) was added drop-wise into this solution under N_2 gas atmosphere and the composite formation reaction started. The reaction mixture was continuously stirred at $15^\circ C$ for 5 h. PDMS/PIN composites were dried by evaporating solvent. The precipitates were washed with distilled hot water ($70^\circ C$) and dried at room temperature for 36 h. PDMS/PIN composites including PIN at different amounts were prepared. Finally, the powdered composites were dried in a vacuum oven at $70^\circ C$ for 24 h.

TABLE I
Conductivity, Magnetic Susceptibility, Density, and Polymerization Yield of PIN in Composite Values of the Samples

Polymer, composites	Conductivity (σ , S cm ⁻¹)	Magnetic susceptibility (X_g , cm ³ g ⁻¹)	Density (d , g cm ⁻³)	Polymerization yield of PIN in composite (wt %)
PIN	2.21×10^{-3}	8.42×10^{-7}	0.70	85
PDMS/PIN (91%)	9.62×10^{-4}	6.41×10^{-7}	0.74	96
PDMS/PIN (74%)	8.71×10^{-4}	3.83×10^{-7}	0.76	93
PDMS/PIN (49%)	7.24×10^{-4}	-2.25×10^{-7}	0.78	92
PDMS/PIN (35%)	5.92×10^{-4}	-1.31×10^{-7}	0.80	86
PDMS/PIN (8%)	4.24×10^{-4}	-2.34×10^{-7}	0.82	78

Film preparation

To prepare PDMS/PIN freestanding composite films, 0.9 g powder of composite was dissolved in DMF, DMSO, 1,1,2,2-tetrachloroethane (12, 10, 8 mL, respectively) solvent mixture. Then the solution was casted on glass Petri dish. The film was dried at room temperature for 48 h and it was taken from glass Petri dish by exposing the film to water steam.

RESULTS AND DISCUSSION

Yield, conductivity, magnetic susceptibility, and density results

Table I shows the yield percentage, density, conductivity values, and magnetic susceptibility measurements of PIN and PDMS/PIN composites including

different weight percentages of PIN. Molecular weight of used PDMS is high enough (M_w : 95,000 and M_n : 30,000). Methyl groups in PDMS can be caused to introduce branches or crosslinks in the polymer chain. For these reasons, PDMS/PIN composites were obtained as solid materials. The conductivity of PIN was determined 2.21×10^{-3} S cm⁻¹. As shown in Table I, the conductivities of PDMS/PIN composites were slightly increased with increasing content of PIN. In other words, conductivity values of the composites decreased with increasing amount of PDMS in the composites. Among the composites, PDMS/PIN including 91% of PIN had the highest conductivity with a value of 9.62×10^{-4} S cm⁻¹. The highest PIN yield was also obtained in PDMS/PIN (91%) with a value of 96%. Figure 1 shows the conductivities of PIN [Fig. 1(a)] and PDMS/PIN composite, with 8% of PIN [Fig. 1(b)] at various temperatures were measured by using four-probe technique. Figure 2 shows the conductivities of all composites at various temperatures that were measured by using four-probe technique. It was observed that the conductivities of PIN and PDMS/PIN composites increased with increasing temperature. It can be attributed to the increases of charge carriers and electronic transport properties.⁴⁰

From Gouy scale magnetic susceptibility measurements (Table I), PIN, and the composites including 91 and 74% of PIN were found to have polaron

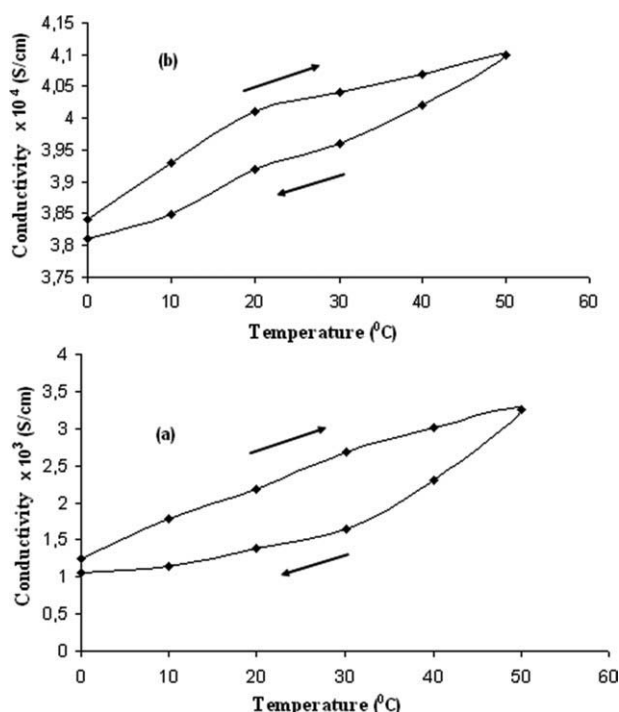


Figure 1 Change in conductivity with temperature of (a) PIN, (b) PDMS/PIN (8%) composite.

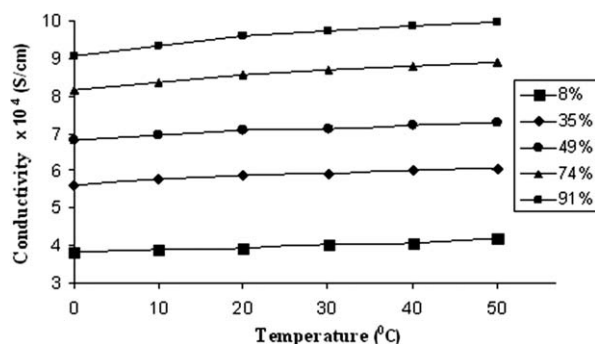


Figure 2 Change in conductivity with temperature of all PDMS/PIN composites.

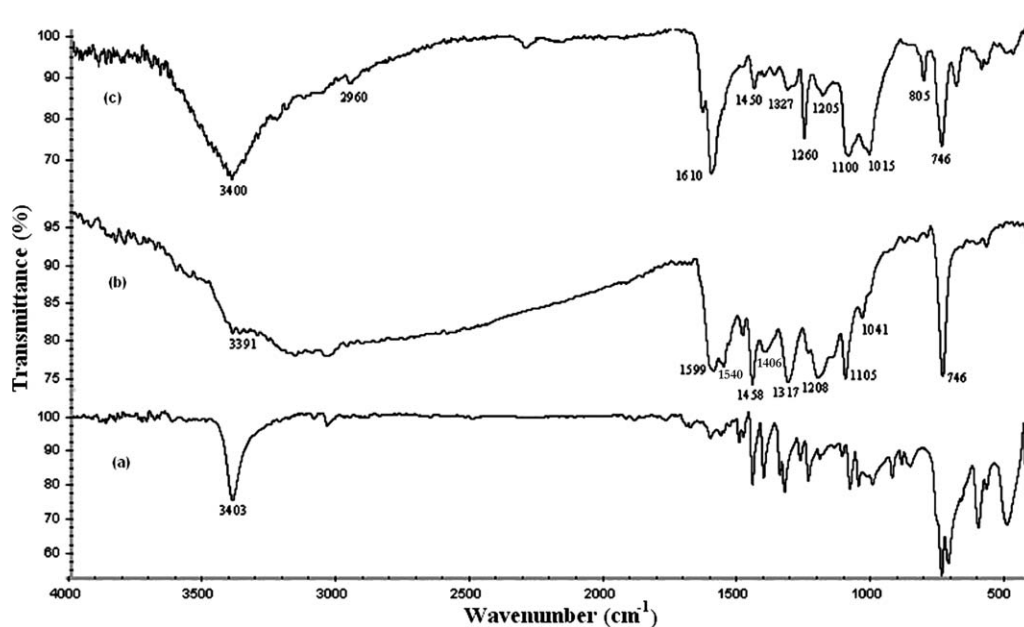


Figure 3 FTIR spectra of (a) Indole, (b) PIN, (c) PDMS/PIN (8%) composite.

nature.⁴¹ The composites including 49, 35, and 8% of PIN were found to have bipolaron nature.⁴²

FTIR results

Figure 3 shows FTIR spectra of indole [Fig. 3(a)], PIN [Fig. 3(b)] and PDMS/PIN composite, with 8% of PIN [Fig. 3(c)], respectively. The bands at 3391, 1540, and 1406 cm^{-1} indicate the presence of the $N-H$ stretching in the spectra of PIN [Fig. 3(b)]. The band at 1599 cm^{-1} assigned to the vibration of the $C_2=C_3$ aromatic bonds typical of indoles. Two bands at 746 and 739 cm^{-1} show $C-H$ vibration of indole [Fig. 3(a)]. However, PIN has one band at 746 cm^{-1} showing a hydrogen bond at the end of the chain. Also the band at 1540 cm^{-1} can be related $N-H$ stretch and C_2-C_3 deformation. The bands at 1458 and 1208 cm^{-1} attributed to the benzene and aromatic rings of PIN structure. FTIR spectrum of PIN strongly suggests that the monomers are linked via the positions 2 and 3 of the pyrrole ring.^{43,44}

The polymerization mechanism of indole shows two different postulated mechanisms concerning the propagation reactions (coupling between two radical-cations or between a radical-cation and a neutral species) to give conducting polymer.⁴³

PDMS/PIN composite indicates the bands coming from both PIN and PDMS [Fig. 3(c)]. There are four characteristic bands at 2960, 1260, 1100–1015, and 805 cm^{-1} for PDMS. The peak at 2960 cm^{-1} belongs to aliphatic $C-H$ stretching and the band at 1260 cm^{-1} has $Si-CH_3$ vibration. The peak at 1100 and 1015 cm^{-1} , $Si-O-Si$ asymmetric stretching; and at 805 cm^{-1} , $Si-CH_3$ rocking.⁴⁵ However the similar bands

to those of PIN are seen, the values of the bands shifted due to interaction between PIN and PDMS.

UV-vis results

The UV-vis spectra of PIN and PDMS/PIN composites are shown in Table II. The UV-vis spectrum of PIN shows two peaks at 268 and 308 nm; the 268-nm peak corresponds to $\pi-\pi^*$ transitions of the polymer chain⁴⁶ and the peak 308 nm belongs to conjugation and $\pi-\pi^*$ transitions of benzene ring.⁴⁷ The UV-vis spectra of PDMS/PIN composites show one peak. The first absorption is assigned to the conjugation of monomers.⁴⁶

Thermal analysis results

Thermal analysis (DSC and TGA) results were illustrated in Figure 4(a–d). The decomposition temperatures obtained from TGA thermograms of PIN and the three PDMS/PIN composites are also tabulated in Table III. As seen from Table III, PIN shows two step weight loss while the PDMS/PIN composites

TABLE II
UV-Vis Results of Samples

Polymer, composites	λ_1 (nm)	λ_2 (nm)
PIN	268	308
PDMS/PIN (91%)	268	–
PDMS/PIN (74%)	268	–
PDMS/PIN (49%)	268	–
PDMS/PIN (35%)	268	–
PDMS/PIN (8%)	268	–

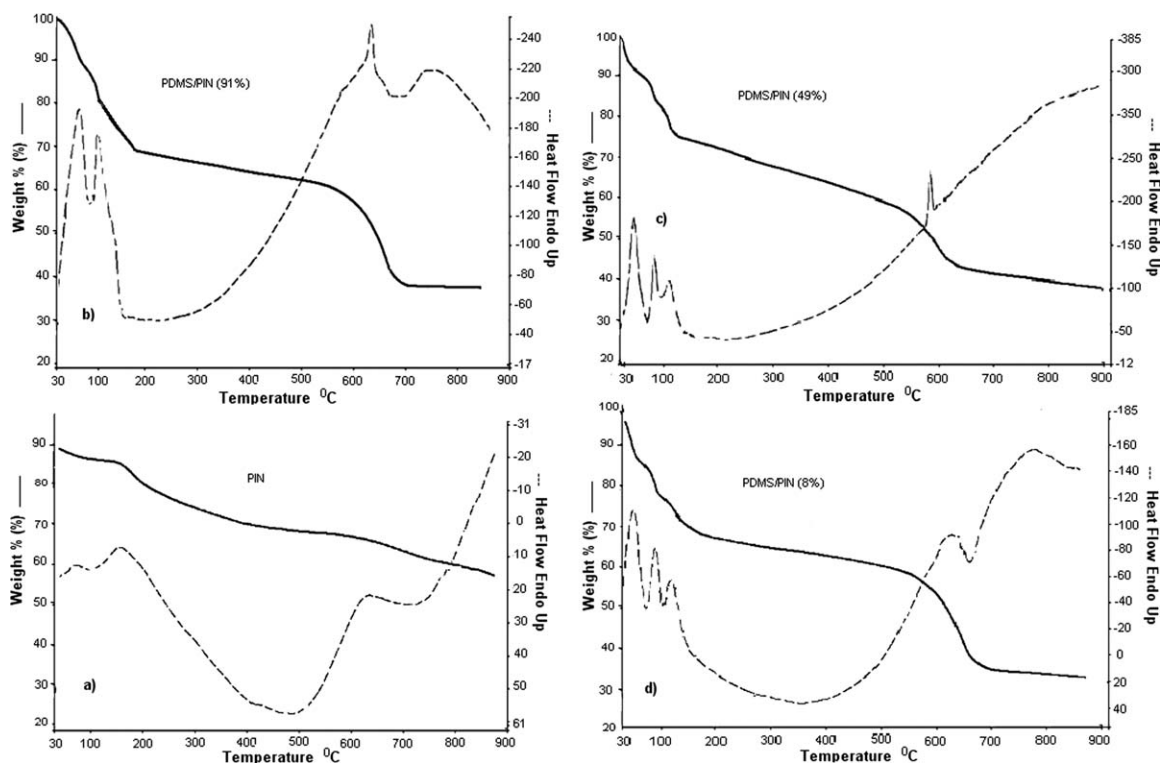


Figure 4 TGA and DSC curve of (a) PIN, (b) PDMS/PIN (91%) composite, (c) PDMS/PIN (49%) composite, (d) PDMS/PIN (8%) composite.

indicate decomposition with one-step weight loss. The weight loss between 30 and 150°C indicates the absorbed volatile molecules and low molecular weight segments in polymer matrix.⁴⁸ According to the initial decomposition temperatures (T_i), PDMS/PIN (91%) composite had the highest decomposition temperature with 600°C, whereas that of PDMS/PIN (8%) composite had the lowest decomposition temperature with 560°C. TGA curve of the PIN, first weight loss between 150 and 210°C, attributes removal of the dopant anions from the polymer structure and the second step at 600–700°C, shows degradation of the polymer.⁴⁹ The degradation of

the synthesized PDMS/PIN composites carried out between 560 and 680°C. As a result, the prepared PDMS/PIN composite systems were more thermally stable than polyindole. Moreover, from the decomposition temperatures it can be observed that the polymer and composites were thermally stable.

The endothermic transitions obtained from DSC curves (Fig. 4 dotted line) are given in Table IV. DSC curve of PIN shows endothermic phase changes at 65, 150, and 610°C. The endothermic peak obtained at 65°C can be described as the glass transition temperature (T_g) of PIN.⁵⁰ At 150°C is due to the removal of the dopant anions from the polymer structure and the last step at 610°C, shows the degradation of the polymer structure.⁵¹ The DSC curves of PDMS/PIN composites show endothermic phase transition between 60 and 620°C. First step

TABLE III
TGA Results of the Samples

Polymer, composites	Decomposition temperature (°C)			Residue at 900°C wt (%)
	T_i	T_m	T_f	
PIN	150	185	210	55
PDMS/PIN (91%)	600	650	700	40
PDMS/PIN (74%)	565	610	660	39
PDMS/PIN (49%)	570	620	640	38
PDMS/PIN (35%)	570	625	670	36
PDMS/PIN (8%)	560	620	680	32

T_i , Initial decomposition temperature; T_m , Maximum decomposition temperature; T_f , Final decomposition temperature.

TABLE IV
DSC Results of the Samples

Polymer, composites	Glass transition temperature (T_g) (°C)		
	Endothermic (°C)	Endothermic (°C)	Endothermic (°C)
PIN	65	150	610
PDMS/PIN (91%)	–	620	–
PDMS/PIN (74%)	–	610	–
PDMS/PIN (49%)	–	600	–
PDMS/PIN (35%)	–	605	–
PDMS/PIN (8%)	–	620	–

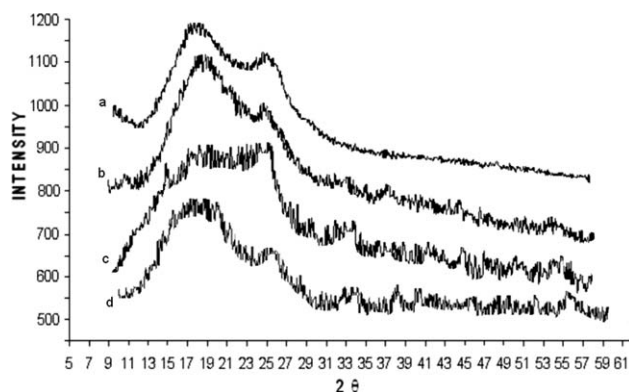


Figure 5 XRD patterns of (a) PIN, (b) PDMS/PIN (91%) composite, (c) PDMS/PIN (8%) composite, (d) PDMS/PIN (49%) composite.

(60–125°C) could be attributed to the loss of small units such as solvents and monomers in the composites. The last endothermic transitions observed between 600 and 620°C correspond to the degradation of the PDMS/PIN composites. The endothermic transitions are seen in all polymers and the endothermic transition temperatures of the composites were higher than PIN. Finally, DSC results exhibit adequate thermal stability.

X-ray diffraction results

Figure 5 represent the XRD scans of PIN [Fig. 5(a)] and PDMS/PIN (91%) [Fig. 5(b)], PDMS/PIN (8%) [Fig. 5(c)], and PDMS/PIN (49%) [Fig. 5(d)], respectively. The diffraction peak of PIN observed at scattering angles of $2\theta = 18^\circ$ and $2\theta = 25^\circ$ seem to be reflection of the polymer backbone.⁵² XRD of PDMS/PIN composite with 91% PIN, is more shallow peak between of $2\theta = 19^\circ$ and $2\theta = 25^\circ$. Interestingly, the XRD pattern of PIN suggesting a typical amorphous structure modified the XRD to a slightly amorphous pattern of PDMS/PIN composite by

introducing amorphous characteristics.⁵³ X-ray diffraction pattern of polyindole was recorded and it shows amorphous nature.⁵⁴ PDMS is also amorphous homopolymer.³⁶ XRD of PDMS/PIN composite with 8% PIN is more wide and shallow peak between $2\theta = 14^\circ$ – 28° . And XRD of PDMS/PIN composite with 49% PIN is more wide and shallow peak between $2\theta = 15^\circ$ – 23° and little shallow peak at $2\theta = 25^\circ$.

Elemental analysis results

The experimental and calculated compositions obtained from elemental analysis of the PIN and PDMS/PIN composites are given in Table V. The theoretical values of elemental analysis show good agreement with the experimental values. There is about 2% difference between calculated and experimental data. This can also attribute to the drastic synthesis conditions utilized. These results also support that PIN and PDMS/PIN composites were successfully synthesized.⁵⁵ Further, it was revealed that PIN contents in the composites were between 8 and 91 wt %.

Inductively coupled plasma-optic emission spectroscopy results

ICP-OES results (Table VI) show amount of Fe (mg g^{-1}) in the PIN and PDMS/PIN composites including different percentages of PIN structures. As seen in Table VI, the amount of Fe in the polymer structures increased with increasing PIN into the structure of composites. According to the obtained results it can be concluded that Fe may be absorbed in the PIN or can form a complex with PIN. This result indicates that the Fe in the polymer structure affects the conductivity.⁵⁶ The results indicate that, residual amounts of Fe was present in PIN and PDMS/PIN composites due to FeCl_3 oxidizing agent, which could not be removed by washing process.⁵⁵

TABLE V
Elemental Analysis Results of PIN, PDMS, and PDMS/PIN Composites

Polymers, composites	Element	C (%)	N (%)	H (%)
Experimental	PIN	82.94	11.89	4.75
Theoretical		82.05	11.97	5.98
Experimental	PDMS/PIN (89%)	74.93	10.77	5.94
Theoretical	PDMS/PIN (91%)	77.60	10.89	6.17
Experimental	PDMS/PIN (72%)	66.78	8.58	6.12
Theoretical	PDMS/PIN (74%)	69.15	8.86	6.54
Experimental	PDMS/PIN (48%)	55.38	5.78	6.87
Theoretical	PDMS/PIN (49%)	56.75	5.86	7.08
Experimental	PDMS/PIN (34%)	45.17	3.95	6.94
Theoretical	PDMS/PIN (35%)	49.81	4.19	7.38
Experimental	PDMS/PIN (7%)	34.86	1.10	7.21
Theoretical	PDMS/PIN (8%)	36.41	1.98	7.94
Experimental	PDMS	32.86	–	8.12
Theoretical		32.43	–	8.12

TABLE VI
ICP-OES Results of PIN and PDMS/PIN Composites

Polymer, composites	Fe (mg g ⁻¹)
PIN	19.28
PDMS/PIN (91%)	17.41
PDMS/PIN (74%)	18.76
PDMS/PIN (49%)	15.88
PDMS/PIN (35%)	12.67
PDMS/PIN (8%)	10.21

Scanning electron microscopy results of the powder composites

SEM photographs of PIN and PDMS/PIN composite powders, with 8, 49, and 91% of PIN, have been seen in Figure 6. The SEM micrograph of PIN [Fig. 6(a)] demonstrated a granular, sponge-like porous structure and also showed a tendency for particle aggregation.²² The SEM photograph of PDMS/PIN composite including 91% of PIN [Fig. 6(b)] shows granular and porous structure. It can be seen that morphological structures of composites are different from PIN. PDMS/PIN composite including 49% of

PIN has a layered and granular structure [Fig. 6(c)]. PDMS/PIN composite, with 8% of PIN shows sponge-like structure [Fig. 6(d)]. The surface properties of the composites illustrated different morphology from each other. The SEM photographs of PDMS/PIN composites demonstrate that composite formed a granular, sponge-like porous structure and more homogenous particle size distribution than PIN.

SEM results of the composite films

Composite films prepared in DMF, DMSO, and 1,1,2,2-tetrachloroethane solvent mixture showed relatively homogeneous morphology with high torsion resistance from the SEM of polymer films with different PDMS/PIN weight ratio as shown in Figure 7. The PDMS/PIN (74%) composite [Fig. 7(d)] film was less uniform and less homogenous compared to the other polymer films. As shown in Figure 7(a–c) with a higher content of PDMS the film surface appeared smooth, and the conductivities of these samples decreased. The most

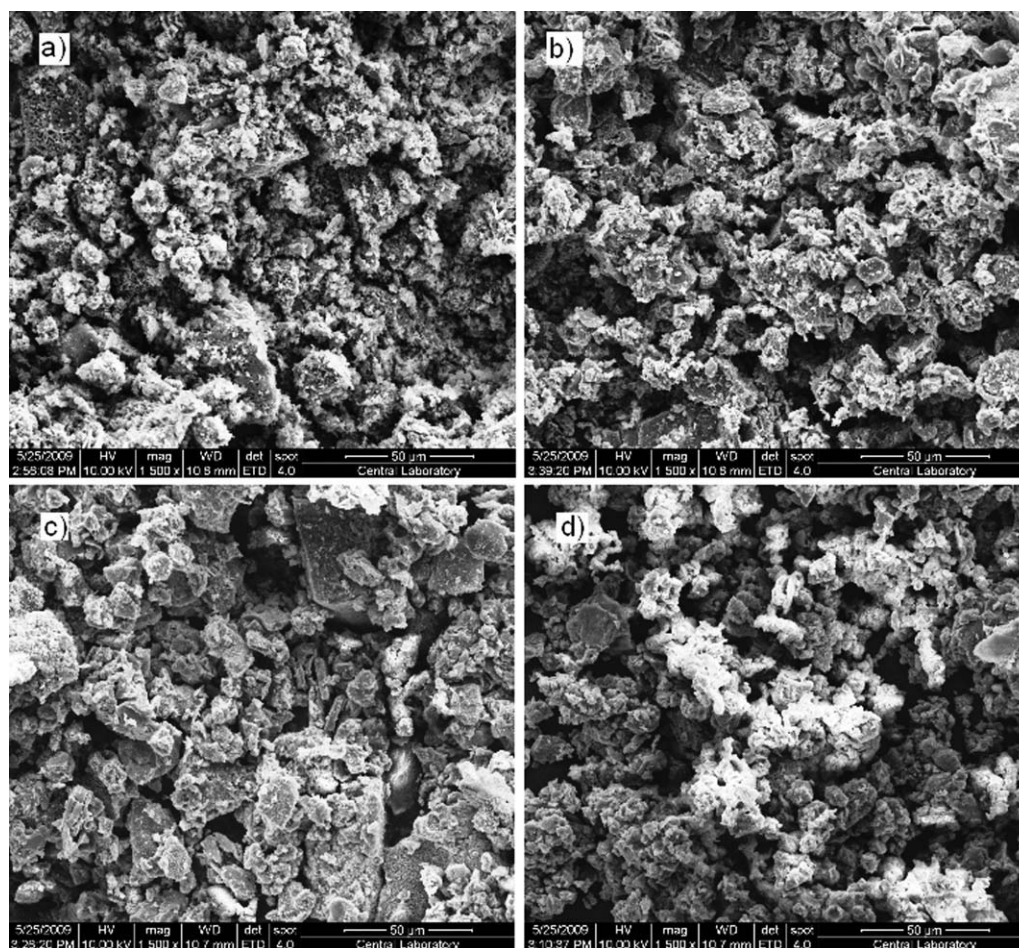


Figure 6 SEM photograph ($\times 1500$) of (a) PIN, (b) PDMS/PIN (91%) composite, (c) PDMS/PIN (49%) composite, (d) PDMS/PIN (8%) composite.

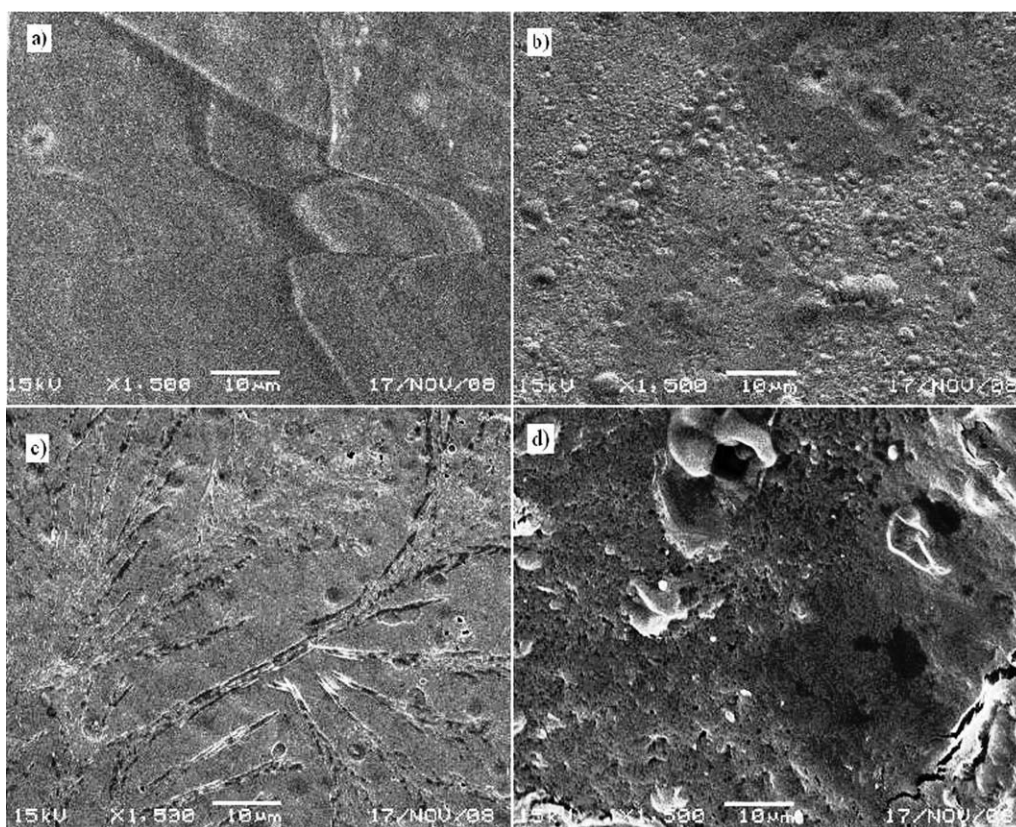


Figure 7 SEM photographs ($\times 1500$) of (a) PDMS/PIN (8%) composite film, (b) PDMS/PIN (35%) composite film, (c) PDMS/PIN (49%) composite film, (d) PDMS/PIN (74%) composite film.

homogenous and uniform film was prepared from PDMS/PIN (8%) composite [Fig. 7(a)].

Film photographs

Photographs of the freestanding composite films are given in Figure 8. Because of the poor solubility, PIN films from these media were not very good, especially their mechanical properties, usually in the form of powder. But freestanding films of PDMS/PIN composites were flat and could be peeled from the Petri dishes easily. Generally, by increasing the amount of PIN in the composite, the strength of the films decreased. PDMS/PIN composite, with 91% of PIN film could not be prepared as a uniform single layer. The most homogenous and uniform films were prepared from PDMS/PIN composites, with 8, 35, and 49% of PIN [Fig. 8(a–c), respectively].

Stress–strain results

The stress–strain experiments were carried out to determine the mechanical stability of films.²³ Stress–strain tests of PDMS/PIN composites including 8, 35, 49, and 74 wt % of PIN could carry out. The data of the stress–strain experiments are shown in Table VII. By increasing the PDMS percentage in films,

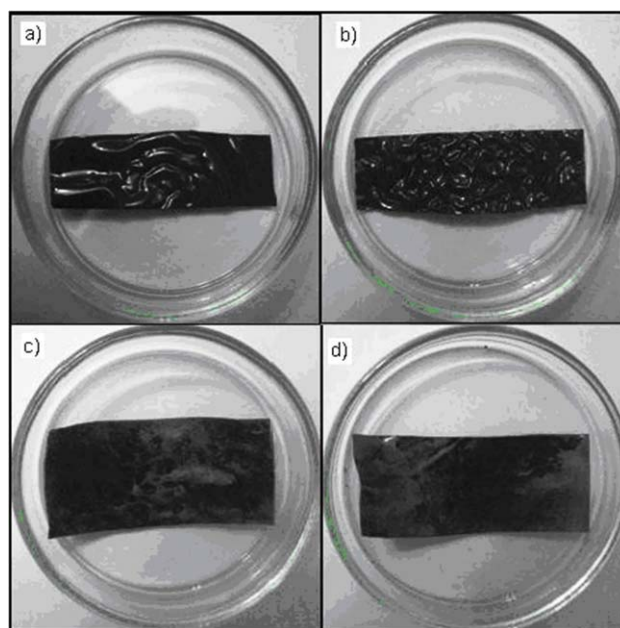


Figure 8 Film photographs of (a) PDMS/PIN (8%) composite, (b) PDMS/PIN (35%) composite, (c) PDMS/PIN (49%) composite, (d) PDMS/PIN (74%) composite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE VII
Results of Stress–Strain Experiments of Samples

Polymer, composites	Length (mm)	Width (mm)	Maximum load (N)	Maximum extension (mm)	Energy (J)
PIN	–	–	–	–	–
PDMS/PIN (91%)	–	–	–	–	–
PDMS/PIN (74%)	40	15	26	155	1.71
PDMS/PIN (49%)	40	15	52	85	2.05
PDMS/PIN (35%)	40	15	76	130	7.40
PDMS/PIN (8%)	40	15	98	185	9.27

value of thickness, maximum load, maximum extension, and energy of composite films are increased. Among the composites PDMS/PIN (8%) composite film had the highest maximum load, maximum extension, and absorbed energy values. It can be seen from Table VII that PDMS modify mechanical properties of PIN.

CONCLUSIONS

PDMS/PIN composites were successfully synthesized by chemical polymerization, and their spectral, thermal, optical characterizations, and some physical properties were investigated. From the XRD results the amorphous structure of PIN increased in the presence of PDMS matrix for composites. Freestanding films of PDMS/PIN composites were successfully prepared by casting. Chemical, structural, and morphology studies indicated that PIN/PDMS composites with better quality could be obtained from PDMS. According to FTIR spectra, the existence of the N–H bond implied that coupling between the monomer units occurred at the C₂ and C₃ positions. PDMS/PIN composites indicated good mechanical properties and thermal stability. From conductivity, FTIR, TGA-DSC, stress–strain, X-ray, and SEM results, we can say that the *in situ* composite system is more useful for application fields of PDMS and PIN materials. These features are expected to introduce more applications for conducting PIN in future.

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