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Polyaniline and polypyrrole modified conductive nanocomposites of polyfuran and polythiophene with montmorillonite clay

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Nanocomposites of polyfuran (PF) and polythiophene (PTP) with montmorillonite clay (MMT) were prepared and modified by loading of polyaniline (PANI) and polypyrrole (PPY) moieties via polymerization of aniline (ANI) and pyrrole (PY) in aqueous dispersions of PF-MMT and PTP-MMT nanocomposites. Formation of PANI and PPY and their subsequent incorporation in the PF-MMT and PTP-MMT composites was confirmed by FTIR absorption studies. X-ray diffraction (XRD) patterns of PANI and PPY modified PF-MMT and PTP-MMT composites showed that PF-MMT and PTP-MMT intercalates were still present in the modified composites. Scanning electron microscopic analysis revealed distinctive morphological patterns of the various composite particles. The dc conductivity values of PANI and PPY modified PF-MMT and PTP-MMT composites were in the order of 10^{-2} S/cm in either system – a value much improved compared to the same for both of the unmodified PF-MMT (10^{-7} S/cm) and PTP-MMT (10^{-5} S/cm) nanocomposites respectively.

1. Introduction

During recent past considerable research attention has been paid globally to synthesis and evaluation of speciality polymer based organic-inorganic hybrid composite materials. In particular, nanocomposites of speciality polymers with various metal oxides and MMT clay were studied in details [1–5]. Synergism of individual characteristics of the components of these nanocomposites resulted in the manifestation of distinctive properties as well. In these composites, speciality polymers such as PANI, PPY, PTP, PF, and polyN-vinylcarbazole (PNVC) were chosen as the organic components. Several nanodimensional metal oxides such as SiO_2 [6, 7], SnO_2 [8], MnO_2 [9], Fe_2O_3 [10], ZrO_2 [11], Fe_3O_4 [12], MMT [2, 4], Al_2O_3 [13, 14], several inorganic host matrixes like V_2O_5 xerogels [15–18], FeOCl [19, 20], various grades of carbon black (CB), acetylene black (AB), buckminsterfullerene (BMF) [21] and carbon nano tubes (CNTs) [22] were used as the inorganic components.

Literature on MMT based nanocomposites of speciality polymers like PNVC, PANI and PPY has grown tremendously over these years. Ray and Okamoto [4] reviewed polymer-layered silicate nanocomposites in details. Bio-degradibility characteristics of various MMT based nanocomposites were reported in recent times [4, 23, 24]. Among these various polymer-MMT nanocomposite systems, conductive composites comprising PANI/PPY and MMT received considerable attention [25–27]. These composites could be effectively utilized as 'electrorheological fluids (ER)' (suspension of a polarizable moieties in an insulator continuous phase) or as 'smart fluids' due to their high dc conductivity values (10^{-1} S/cm). Relevantly, PANI-MMT and PPY-MMT nanocomposites were synthesized via emulsion and inverted emulsion polymerization techniques using PDS as the oxidant for the polymerization of ANI and PY monomers respectively. In this context, very recently, Biswas and Ballav prepared PTP-MMT [28], PANI-MMT [29], PPY-MMT [29] and PF-MMT [30] nanocomposites via intercalative polymerization of the respective monomers by MMT alone without using any extraneous oxidants such as ammonium perdisulfate (PDS), or $FeCl_3$.

In contrast to PANI-MMT and PPY-MMT nanocomposites, the dc conductivity values of PF-MMT and

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PTP-MMT composites are rather low (10^{-7} and 10^{-5} S/cm respectively). In the present work attempts were made to improve the conductivity values of these composites by incorporation of conducting PANI and PPY moieties on these preformed PF-MMT and PTP-MMT nanocomposites. To our knowledge, the procedure developed in this work is indeed novel and the prepared PANI and PPY modified PF-MMT or PTP-MMT composites are evidently new materials of interest combining the distinctive properties of either speciality polymer components. Salient aspects of this research are highlighted in this article.

2. Experimental

2.1. Materials

Furan (Fluka, Switzerland), thiophene (Aldrich, USA), pyrrole (Lancaster, UK) and aniline (Lancaster, UK) were vacuum distilled (cold water circulated reflux condenser) and stored in dark before use. Na^+-MMT caly (Aldrich, USA) was heated to ~100°C for 3 h prior to use. Ammonium perdisulfate (Merck, Germany) was used as such. All other reagents were of AR grade and used after necessary purification by standard procedures.

2.2. Preparation of PF-MMT nanocomposite

A known amount of furan (F) was injected to a conical flask containing a weighed quantity of MMT and fitted with a reflux condenser through which ice-cold water (0°C) was circulated. The temperature of the mixture was raised to 50°C and the colour of the reaction mixture gradually changed from white–pink–brown and finally to dark-gray. The system was kept under this condition for 3 h. Excess MeOH was added to remove any unreacted monomer and the whole mass was filtered off. The dark-gray mass thus collected was dried at 50°C under vacuum for 5 h to constant weight and subsequently characterized as the PF-MMT nanocomposite [30].

2.3. Preparation of PTP-MMT nanocomposite

MMT clay pre-dried as described before was weighed into a round-bottomed Pyrex flask fitted with a reflux condenser. A known weight of thiophene (TP) was syringed into the flask and the mixture allowed to interact for 24 h. at the boiling point of TP. The reaction was exothermic and any un-reacted TP vapour was refluxed back into the flask continuously. The colour of MMT clay gradually changed from white–yellow–orange–brown–brownish black and finally too black followed by a gradual rise in the temperature of the TP-MMT clay system. The whole mass was then washed with MeOH to remove any un-reacted monomer and dried at 50°C under vacuum for 5 h. The dried mass was subsequently characterized as the PTP-MMT nanocomposite [28].

2.4. Preparation of PANI-(PF-MMT) and PANI-(PTP-MMT) nanocomposites

Weighed amounts of PF-MMT and PTP-MMT composites prepared as above were taken in two 1×10^{-4} m^3 conical flasks each containing 0.25×10^{-4} m^3 2M HCl solution. A known volume of ANI was added to these systems and stirred for 15 min. Thereafter, a known quantity of PDS was added to these mixtures and stirring continued for further 45 min. Excess MeOH was added, filtered and the residues were repeatedly washed with acetone to remove oligomers of PANI [31]. Finally the dark green coloured masses were dried under vacuum at 50°C for 1 h and characterized as PANI-(PF-MMT) and PANI-(PTP-MMT) nanocomposites respectively.

2.5. Preparation of PPY-(PF-MMT) and PPY-(PTP-MMT) nanocomposites

Weighed amounts of PF-MMT and PTP-MMT composites prepared as above were taken in two 1×10^{-4} m^3 conical flasks each containing 0.25×10^{-4} m^3 distilled water. A known volume of PY was added to these systems with continued stirring for 15 min. Thereafter, a known quantity of PDS was added to these mixtures and stirring continued for further 45 min. Excess MeOH was added, filtered and the residues were repeatedly washed with acetone. Finally the dark black coloured masses were dried under vacuum at 50°C for 1 h and characterized as PPY-(PF-MMT) and PPY-(PTP-MMT) nanocomposites respectively.

2.6. Characterization and property evaluation

FTIR spectra of various the composites were taken on a JASCO 680 Plus model instrument. XRD pattern of the composites was obtained from a Philips X Ray diffractometer (Cu LFF; 40 kV; 20 mA). Dispersions of the composite samples were sputter-coated with gold layer and a Hitachi S 415 A model scanning electron microscope was used to take the micrographs. Dc conductivity values were measured by the conventional four-probe method using pressed pellets.

3. Results and discussion

3.1. General features of polymerization vis-à-vis composite formation

Table I represents some typical data on the aqueous polymerization of ANI and PY vis-à-vis composites formation thereof. Conversions (%) to PANI and PPY varied from 20–65 and 20–85 using a [Ox]/[Monomer] mole ratio 1.5:1.0 at room temperature in aqueous medium in either systems and % loading of PANI and PPY per g of composite varied from 10–40 and 10–50 respectively.

It should be noted in this context that with an [Ox]/[Monomer] mole ratio of 1.5:1 at room temperature, the homopolymerization of ANI and PY proceeds

TABLE I. Some typical data on the polymerization vis-à-vis composite formation

	Reactants				Products		
	Composites (g)		Monomers (g)				
Entry No.	PF-MMT	PTP-MMT	ANI	PY	Binary composite (g)	% PANI/g of composite	% PPY/g of composite
1	0.5	–	0.3	–	0.57	12	–
2	0.5	–	0.5	–	0.70	28	–
3	0.5	–	1.0	–	1.10	54	–
4	–	0.5	0.3	–	0.56	11	–
5	–	0.5	0.5	–	0.71	29	–
6	–	0.5	1.0	–	1.10	54	–
7	0.5	–	–	0.3	0.58	–	14
8	0.5	–	–	0.5	0.75	–	33
9	0.5	–	–	1.0	1.15	–	56
10	–	0.5	–	0.3	0.59	–	15
11	–	0.5	–	0.5	0.80	–	37
12	–	0.5	–	1.0	1.20	–	58

usually to very high conversions (>90% in each system). The fall in the% conversion to these polymers in presence of preformed PF-MMT and PTP-MMT nanocomposites is thought to be due to the interaction of oxidant with the basic PF-MMT and PTP-MMT moieties which would in turn reduce the% conversion to PANI and PPY respectively.

3.2. FTIR spectral characteristics

Table II presents detailed FTIR absorption peaks (cm^{-1}) observed for various composite systems. PF-MMT and PTP-MMT composites prepared by our earlier method showed characteristic FTIR absorptions of PF (625: C–H out of plane vibration; 797: $\alpha\alpha'$ coupling of the carbon backbone; 1018–1158: C–H bending and stretching; 1535: C=C stretching; 1590–1635: vibration mode of furan ring; 1750: C=O stretching mode) and PTP (695: C–H out of plane vibration of 2,5 disubstituted thiophene ring; 787: $^{\delta}$C–H out of plane vibration; 834: 2,4 coupling; 1070: C–H in plane bending; 1270: stretching vibration of thiophene ring; 1435: $^{\nu}$C=C stretching; 1713: >C=O stretching) homopolymers along with the characteristic FTIR absorptions of MMT clay in either system [32]. The observed peaks and their assignments fully matched with the observation of Glenis et al. [33] and Chanunpanich et al. [34] for PF and PTP systems respectively. Incorporation of PANI and PPY moieties in the PF-MMT and PTP-MMT composite systems was endorsed by the appearance of characteristic FTIR absorptions of PANI (1475–1602: N–H bending; 1309: C–N stretching) and PPY (1043–1338: C–H vibration of 2,5 disubstituted pyrrole ring; 798: C–H deformation of 2,5 disubstituted pyrrole ring) homopolymers respectively [29]. Characteristic FTIR absorptions of MMT clay were also found to be present in PANI-(PF-MMT), PANI-(PTP-MMT), PPY-(PF-MMT) and PPY-(PTP-MMT) composites respectively.

TABLE II. FTIR absorption peaks (cm^{-1}) of various composites

Composites	FTIR peaks observed (cm^{-1})
PF-MMT	413.6, 443.6, 469.5, 523.1, 1048.6 (MMT) 668, 800, 1630, 1726 (PF)
PTP-MMT	415.6, 427.6, 470.1, 528.1, 1052.5 (MMT) 695.2, 799, 1461, 1721 (PTP)
PANI-PF-MMT	418.5, 422.3, 464.8, 1041.8 (MMT) 696.6, 802.2, 838.9, 1240.5, 1304.6, 1472.4, 1559, 1763 (PF and PANI)
PANI-PTP-MMT	418.5, 422.3, 464.8, 1041.8 (MMT) 621.9, 802.2, 1240.9, 1303.1, 1479.6, 1563 (PTP and PANI)
PPY-PF-MMT	467.6, 522.6, 1048.6 (MMT) 686.1, 797.9, 928.1, 1454.1, 1552, 1626, 1703 (PF and PPY)
PPY-PTP-MMT	403, 417.9, 469.1, 524.1, 1045.2 (MMT) 695.2, 797.9, 925.1, 1460.8, 1720.1 (PTP and PPY)

3.3. Thermogravimetric stability studies

Table III shows data on thermal stability characteristics (TGA and DTA) of various composite systems. In general the overall thermal stability trends were as follows: PF-MMT ≈ PANI-PF-MMT ≈ PPY-PF-MMT and PTP-MMT < PPY-PTP-MMT < PANI-PTP-MMT respectively. Previous results on TGA of the PF-MMT and the PTP-MMT composite revealed that both composites were thermally more stable compared to the corresponding homopolymers PF and PTP. However, in the present study upon incorporation of PANI or PPY homopolymers thermal stability of the PF-MMT composite did not improve significantly. In contrast, PANI or PPY modified PTP-

TABLE III. Thermogravimetric stability data of various MMT based composite systems

Systems	% weight loss up to 1000°C	DTA exothermic peaks (°C)
PF-MMT	70	408
PANI-PF-MMT	69	373 (shoulder), 480
PPY-PF-MMT	68	350, 475 (shoulder), 525, 533
PTP-MMT	80	395
PANI-PTP-MMT	49	300, 385 (shoulder), 503
PPY-PTP-MMT	69	338 (shoulder), 378

TABLE IV. XRD data for various composites

Composites	2θ ($\times 10^{-10}$ m)	References
PF-MMT	4.45 (19.84), 12.6 (7.01), 26.66 (3.34), 27.95 (3.18)	30
PTP-MMT	5.36 (16.47), 8.81 (10.02), 12.33 (7.17), 17.68 (5.01)	28
PANI-PF-MMT	5.28 (16.70), 8.98 (9.84), 12.3 (7.19), 17.94 (4.94), 19.91 (4.45), 20.93 (4.24), 21.89 (4.05)	This study
PANI-PTP-MMT	6.74 (13.10), 9.10 (9.71), 12.60 (7.01), 16.45 (5.38), 17.98 (4.93), 19.90 (4.45), 20.67 (4.29), 24.13 (3.68)	”
PPY-PF-MMT	4.35 (20.29), 8.97 (9.85), 17.98 (4.92), 19.90 (4.45), 20.94 (4.23), 23.81 (3.73)	”
PPY-PTP-MMT	5.6 (15.76), 9.10 (9.70), 19.92 (4.45), 21.02 (4.22), 21.54 (4.12), 26.68 (3.33)	”

MMT composite showed much improved thermal stability compared to that of PTP-MMT composite.

Manifestation of exothermic peaks in the DTA scans of various PANI or PPY modified PF-MMT or PTP-MMT composites DTA scans were indicative of the oxidative degradation of the respective polymer backbones as observed earlier in PANI or PPY modified PNVC-Al_2O_3 composite system [14].

3.4. X-ray diffraction pattern analysis

Table IV presents the XRD data for PF-MMT and PTP-MMT composites reported earlier along with the same for PANI and PPY based binary composite systems prepared in this study. Fig. 1 shows the XRD scans of (a) PANI-(PF-MMT), (b) PANI-(PTP-MMT), (c) PPY-(PF-MMT), and (d) PPY-(PTP-MMT) composites respectively. Intercalation of PF and PTP in between the layers of MMT was confirmed by the appearance of peaks at $2\theta = 4.45$ (19.84×10^{-10} m) and $2\theta = 5.36$ (16.47×10^{-10} m) in the XRD scans of PF-MMT [30] and PTP-MMT [28] composites respectively. Interestingly, intercalated PF-MMT and PTP-MMT structures appeared to be almost undisturbed upon incorporation of PANI or PPY moieties as evident from Fig. 1. Characteristic XRD peaks of PANI and PPY were also found to be present in the PANI-(PF-MMT), PANI-(PTP-MMT) and PPY-(PF-MMT), PPY-(PTP-MMT) composite systems respectively which matched convincingly with the data of PANI-MMT and PPY-MMT nanocomposites respectively reported by Kim et al. [25, 26].

3.5. Scanning electron microscopic characteristics

Fig. 2 shows scanning electron micrographs of (a) PTP-MMT, (b) PANI-(PTP-MMT), (c) PF-MMT, and (d) PPY-(PF-MMT) respectively. In our earlier observations, SEM images of PF-MMT and PTP-MMT [30, 28] composites showed existence of lumpy aggregates of varying sizes formed from the agglomeration of small composite particles. The observed morphological patterns were reminiscent of typical ‘raspberry morphology’ reported by Armes et al. [6], Stejskal et al. [7, 8] and Biswas and coworkers [9, 11, 13, 14] for various metal oxides – polymer nanocomposite systems. In the present study, formation of spherical particles is clearly visible in the SEM images of various binary composite systems and these morphological features could be due to the encapsulation of PF-MMT and PTP-MMT composite particles by the precipitating PANI or PPY moieties.

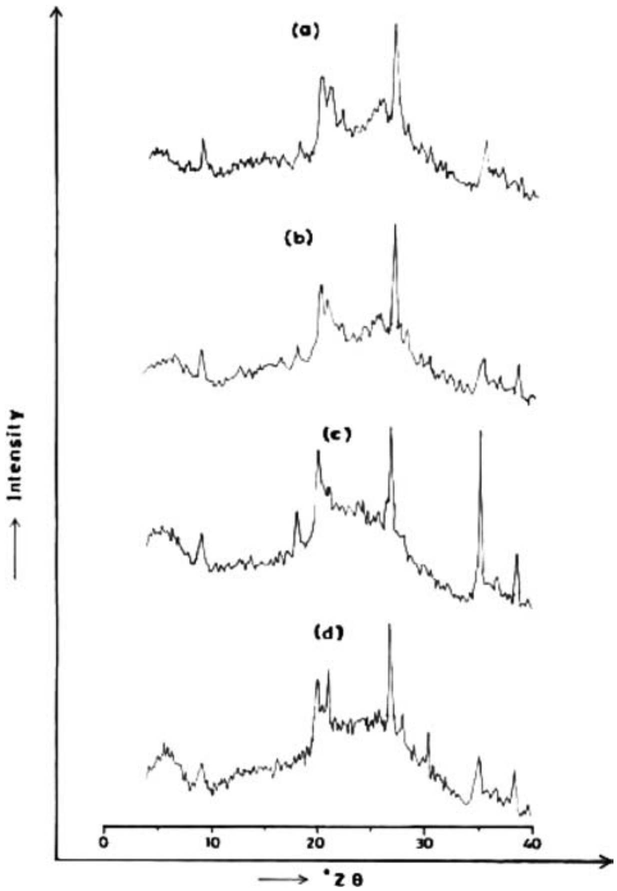

Figure 1 XRD scans of (a) PANI-(PF-MMT) composite, (b) PANI-(PTP-MMT) composite, (c) PPY-(PF-MMT) composite, and (d) PPY-(PTP-MMT) composite respectively.

3.6. Conductivity characteristics

Table V represents dc conductivity values of various MMT based composite systems of speciality polymers such as PANI, PPY, PF and PTP. Entries 1 and 2 indicate that the dc conductivity values of PF-MMT and PTP-MMT composite were in the order of 10^{-7} and 10^{-5} S/cm respectively. Even after doping with $FeCl_3$ the conductivities improved 10 fold in either system. However, upon incorporation of PANI or PPY moieties in the PF-MMT and PTP-MMT composites, the conductivity values improved 10^5 and 10^3 fold respectively (entries 3, 7 and 5, 9 of Table V). The observed features are consistent with those reported recently by Maity and Biswas for binary poly-

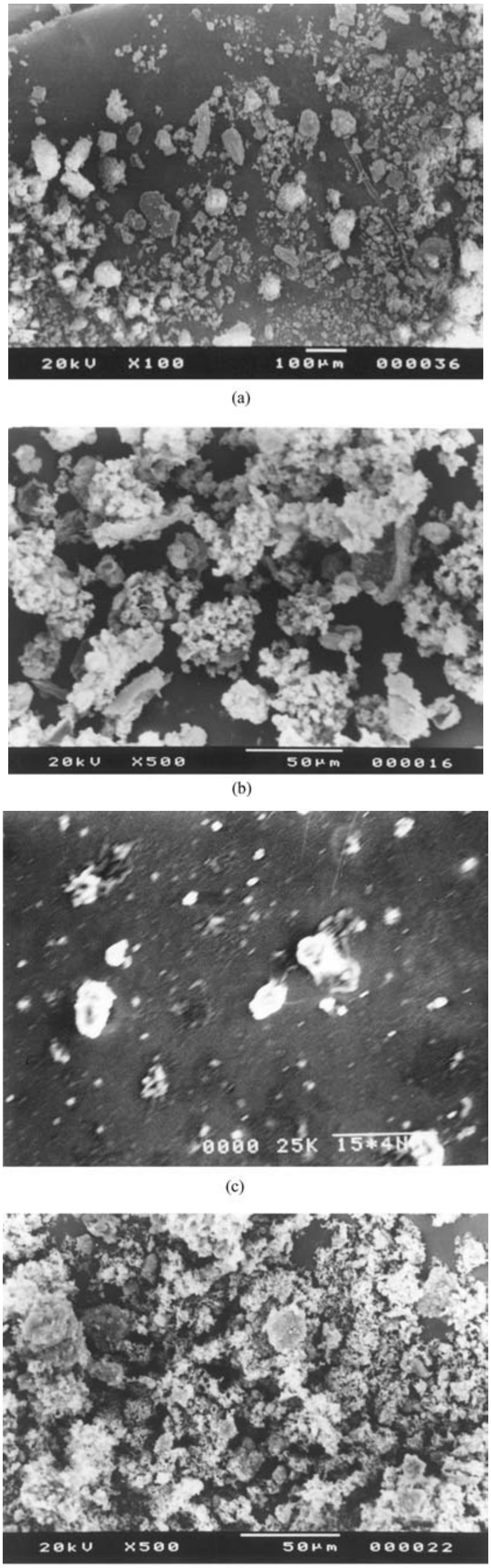

Figure 2 SEM images of (a) PTP-MMT composite, (b) PANI-(PTP-MMT) composite, (c) PF-MMT composite, and (d) PPY-(PTP-MMT) composite respectively.

TABLE V. Dc conductivity data of various composite materials

Entry No.	Materials	Oxidant systems used	Conductivity values (S/cm)	References
1	PF-MMT	–	10^{-7}	30
2	PTP-MMT	–	10^{-5}	28
3	PANI-(PF-MMT)[a]	PDS	10^{-2}	This study*
4	PANI-(PF-MMT)[b]	”	10^{-3}	”
5	PPY-(PF-MMT)[c]	”	10^{-2}	”
6	PPY-(PF-MMT)[d]	”	10^{-3}	”
7	PANI-(PTP-MMT)[e]	”	10^{-2}	”
8	PANI-(PTP-MMT)[f]	”	10^{-3}	”
9	PPY-(PTP-MMT)[g]	”	10^{-2}	”
10	PPY-(PTP-MMT)[h]	”	10^{-3}	”
11	PANI-MMT	–	10^{-5}	29
12	PANI-MMT	PDS	10^{-1}	25
13	PPY-MMT	–	10^{-6}	29
14	PPY-MMT	PDS	10^{-1}	26

*Vide, [a]entry 3, [b]entry 2, [c]entry 6, [d]entry 5, [e]entry 9, [f]entry 8, [g]entry 12, [h]entry 11 of Table I.

mer based nanocomposite systems like PANI and PPY modified PNVC-Al_2O_3 and PAN-SiO_2 nanocomposites [14, 35] and also by several others most relevant being the observation of Omastova et al. [36, 37]. The latter reported that the electrical conductivity of composites prepared by mixing PMMA with PPY coated PMMA particles was enhanced by more than four orders of magnitude when the amount of the loaded PPY moieties exceeded the threshold concentration of PPY (30 w%). In the present study the results clearly show that the conductivity values of the PF-MMT and PTP-MMT composites were enhanced depending upon the w% of PPY being loaded in the composites. Beyond the threshold concentration of PPY (14 w%) the conductivity (S/cm) was increased progressively with increasing (w%) PPY loading (14 (10^{-4}), 33 (10^{-3}) and 56 (10^{-2})) which appear to be quite justified.

4. Conclusion

Loading of PANI or PPY homopolymers onto PF-MMT (10^{-7} S/cm) and PTP-MMT (10^{-5} S/cm) composites improved the dc conductivity values 10^5 and 10^3 fold respectively. The procedure involved in situ polymerization of ANI or PY monomers in aqueous dispersions of preformed PF-MMT and PTP-MMT nanocomposites by PDS oxidant.

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