Conducting Composites of Poly(*N*-vinylcarbazole), Polypyrrole, and Polyaniline with 13X-Zeolite

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ABSTRACT: *N*-vinylcarbazole (NVC) was polymerized by 13X zeolite alone in melt (65°C) or in toluene (110°C) and a poly(*N*-vinylcarbazole) (PNVC)-13X composite was isolated. Composites of polypyrrole (PPY) and polyaniline(PANI) with 13X zeolite were prepared via polymerization of the respective monomers in the presence of dispersion of 13X zeolite in water ($CuCl_2$ oxidant) and in $CHCl_3$ ($FeCl_3$ oxidant) at an ambient temperature. The composites were characterized by Fourier transform infrared analyses. Scanning electron microscopic analyses of various composites indicated the formation of lumpy aggregates of irregular sizes distinct from the morphology of unmodified 13X zeolite. X-ray diffraction analysis revealed some typical differences between the various composites, depending upon the nature of the polymer incorporated. Thermogravimetric analyses revealed the stability order as: 13X-zeolite > polymer-13X-zeolite > polymer. PNVC-13X composite was essentially a nonconductor, while PPY-13X and PANI-13X composites showed direct current conductivity in the order of 10^{-4} S/cm in either system. However, the conductivity of PNVC- 13X composite could be improved to 10^{-5} and 10^{-6} S/cm by loading PPY and PANI, respectively. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 913–921, 2006

Key words: poly(*N*-vinylcarbazole); polyaniline; polypyrrole; 13X zeolite; composite; conductivity

INTRODUCTION

During recent past, a good deal of research attention was paid to control and modify the physical, structural, and electronic properties of potential polymers to counteract the conjugation interacting defects in conducting polymers.[1,2] Attempts have been made to encapsulate various polymers in heterogeneous hosts such as zeolites and layered materials. Synthetic methods for growing various conducting polymers in Y-zeolite have been reported by Bein et al.[3–6] and Roque et al.[7]

Choi et al.[8] reported that polymerization of pyrrole (PY) monomer in Cu^{II} ion-exchanged MCM-41 channels resulted in the manifestation of better properties of the composite, and self-aggregation of polypyrrole (PPY) moieties could be eliminated in this system. Densakulprasert et al.[9] synthesized various polyaniline (PANI)-zeolite (Y, 13X and A1MCM-41) composite systems using Cu^{2+} ion as the oxidant, which could act as sensors to CO and N_2 gas. Relevantly, PANI-13X composite showed highest electrical conductivity. Sreedhar et al.[10] prepared PANI sulfate–zeolite composite by emulsion polymerization technique, and the composite was found to be effective in curing expoxy resins. Chang et al.[11] reported PANI-zeolite composite via intercalation of aniline (ANI) or ANI–HCl salt in HZ, HS, and HY zeolites followed by its subsequent oxidation to PANI by using PDS oxidant. Interestingly, among various composites thus prepared, PANI-HZ and PANI-HS composites showed variable-range hopping, tunneling, and insulating properties.

In the background of the foregoing survey, we have developed suitable procedures to polymerize NVC, PY, and ANI in the presence of 13X zeolite in bulk and in aqueous and nonaqueous solvent, and have been able to isolate the corresponding polymer-13X composites. The procedures developed by us have not been explored earlier, and we feel that these can be extended to a variety of polymer zeolite-based composite systems. We wish to highlight in this article the details of the preparation and evaluation of some bulk properties of 13X-based composites of PNVC, PANI, and PPY. Barring PNVC-13X composite, PPY-13X and PANI-13X composites exhibited appreciable, direct current (dc) conductivity despite the presence of nonconducting 13X moieties in the systems. As for the PNVC-13X composite, we were able to improve the dc conductivity value by 10^{10}-fold via incorporation of conducting PPY or PANI moieties on the PNVC-13X composite.

EXPERIMENTAL

MATERIALS

N-vinylcarbazole (NVC; BASF, Germany) was recrystallized from n-hexane and kept in dark prior to use. Aniline (Lancaster, UK) and pyrrole (Lancaster, UK) were distilled under reduced pressure and stored at 5°C in dark prior to use. 13X zeolite powder (Fluka, Switzerland) was heated to 100°C for 2 h in vacuum prior to use. Cupric chloride (Merck, Germany) and anhydrous $FeCl_3$ (Merck, Germany) were used as such. All other reagents and solvents were of analytical grade (AR) and were used after necessary purification by standard procedures.

Polymerization of NVC by 13X and preparation of PNVC-13X composite

NVC and 13X, both in solid phase

A known weight of NVC was homogeneously mixed with a particular amount of 13X in a conical flask and heated to melting temperature (65°C) for 1 h. Tetrahydrofuran (THF) was added to the reaction mixture and the whole mass was added to excess MeOH. The separated mass was filtered and washed with boiling MeOH to remove any unreacted monomer. The white residue was dried at 60°C in vacuum for 3 h to obtain a constant weight, and the mass was subsequently characterized as a PNVC-13X composite.

NVC in toluene and 13X in solid phase

A known weight of NVC was dissolved in 50 mL of toluene in a 50-mL conical flask and a weighed amount of 13X was added to it. The reaction mixture was refluxed at 110°C for 5 h and thereafter it was added to an excess amount of MeOH. The precipitated white mass was filtered off and the residue was washed with boiling MeOH followed by acetone to remove any adhering substances. The mass was dried at 60°C in vacuum for 3 h to obtain a constant weight and subsequently characterized as a PNVC-13X composite.

Polymerization of PY in the presence of 13X in aqueous medium and preparation of PPY-13X composite

A known weight of 13X zeolite was added to 50 mL H_2O in a conical flask. To this dispersion a known volume of PY was injected. The system was kept under stirring at an ambient temperature. Thereafter, a known weight of $CuCl_2$ was added and was kept under stirring for 6 h. The precipitated mass was filtered off and the residue was washed successively with H_2O, MeOH, and acetone and finally dried at 60°C in vacuum.

Polymerization of PY in the presence of 13X in $CHCl_3$ medium and preparation of PPY-13X composite

A known weight of 13X zeolite was added to 50 mL $CHCl_3$ in a conical flask. To this dispersion a known volume of PY was injected. The system was kept under stirring at an ambient temperature. Thereafter, a known weight of $FeCl_3$ was added and the system was kept under stirring for 6 h at an ambient temperature. The precipitated mass was filtered off and washed successively with H_2O, MeOH, and acetone and finally dried at 60°C in vacuum.

Polymerization of ANI in presence of 13X in aqueous medium and preparation of PANI-13X composite

A known weight of 13X zeolite was added to 50 mL H_2O in a conical flask. To this dispersion a known volume of ANI was injected. The system was kept under stirring at an ambient temperature. Thereafter, a known weight of $CuCl_2$ was added and was kept under stirring for 6 h. The precipitated mass was filtered off and the residue was washed successively with H_2O, MeOH, and acetone and finally dried at 60°C in vacuum.

Modification of PNVC-13X composite via incorporation of PANI and PPY

Preparation of PPY-(PNVC-13X) composite

A known weight of preformed PNVC-13X composite was added to 50 mL H_2O in a conical flask and stirred slowly for 1 h. To this dispersion a known volume of ANI was injected and thereafter a known amount of $CuCl_2$ was added. The system was kept under stirring for 6 h at an ambient temperature. The resulting black mass was centrifuged and washed successively with H_2O, MeOH and finally acetone and dried at 60°C in vacuum.

Preparation of PANI-(PNVC-13X) composite

A known weight of preformed PNVC-13X composite was added to 50 mL H_2O in a conical flask and stirred slowly for 1h. To this dispersion a known volume of PY was injected and thereafter a known amount of $CuCl_2$ was added. The system was kept under stirring for 6 h at an ambient temperature. The resulting black mass was centrifuged and washed successively with H_2O, MeOH and finally acetone and dried at 60°C in vacuum.

Characterization and property evaluation

The FTIR spectra of the PNVC-13X, PPY-13X, PANI-13X, PPY-(PNVC-13X), and PANI-(PNVC-13X) com-

TABLE I
Some Typical Data on Polymerization[a] of NVC, ANI, and PY vis-à-vis Composite Formation with 13X-Zeolite

Entry no.	Reactants (g)				Product	
	NVC	PY	ANI	13X	Composite (g)	% loading[b] of polymer/g of composite
1	0.5	—	—	0.7	0.80	13
2	0.5	—	—	1.0	1.20	17
3	0.5	—	—	1.5	1.76	15
4	1.0	—	—	0.5	1.20	58
5	1.0	—	—	1.0	1.80	45
6	1.0	—	—	1.5	2.40	37
7	—	1.5	—	1.0	2.10	52
8	—	1.5	—	2.0	3.10	35
9	—	0.4	—	0.6	1.02	39
10	—	0.7	—	0.6	1.50	60
11	—	—	1.5	1.0	2.10	52
12	—	—	1.5	1.5	2.60	42

[a] Polymerization conditions: total volume = 50 mL; room temperature; entries 1–3, solution polymerization of NVC; entries 4–6, bulk polymerization of NVC; entries 7 and 8, aqueous polymerization of PY by $CuCl_2$; entries 9 and 10, polymerization of PY by $FeCl_3$ in $CHCl_3$; and entries 11 and 12, aqueous polymerization of ANI by $CuCl_2$.
[b] % loading of polymer/g of composite was calculated by the following relation: {(column 6 − column 5)/column 6} × 100.

posites were taken on a JASCO-680 plus model instrument using pressed KBr pellet. Powder X-ray diffraction patterns were obtained from Rigaku Miniflex (Cu/30 kV/15 mA) model instrument. 13X powder, PNVC-13X, PPY-13X, PANI-13X, PPY-(PNVC-13X), and PANI-(PNVC-13X) composites were taken on a mica foil and then sputter coated with gold layer. Thereafter, a JSM 5200 model scanning electron microscope was used to take the micrographs. Thermogravimetric (TGA/DTA) studies were performed on a Shimadzu DT-40 model instrument. Direct current (dc) conductivity values were measured by the conventional four-probe technique using pressed pellets.

RESULTS AND DISCUSSION

General features of polymerization and composite formation

The characteristic features of the studied systems include the following: (i) polymerization of NVC was achieved by 13X alone (without using any extraneous oxidant) in bulk (65°C, melting temperature of NVC) and in toluene (110°C); (ii) 13X alone was unable to polymerize PY and ANI; (iii) attempts to polymerize NVC, ANI, or PY in aqueous dispersion of 13X using a strong oxidant ($FeCl_3$ or PDS) resulted in the formation of respective homopolymers only, but no stable composites of the polymers with 13X could be recovered perhaps because of the collapse of the 13X network; (iv) among a number of Lewis acids (mainly $3d$-transition metal based oxidant systems) and PDS, only $CuCl_2$ oxidant was effective for the polymerization of ANI and PY in aqueous system; and (v) the pH for $CuCl_2$-13X-H_2O suspension was ~4, while the same for $FeCl_3$-13X-H_2O system was ~1.3. This high

acidity could be the reason for nonrecovery of the polymer-13X zeolite composites in $FeCl_3$ oxidant system.

Tables I and II present some typical data on polymerization of NVC, PY, and ANI vis-à-vis composite formation of the respective polymers with 13X zeolite and on the preparation of PPY and PANI modified PNVC-13X composite systems, respectively. Entries 1–6 (Table I) indicate that % conversion to PNVC and its subsequent loading in the PNVC-13X composite varied between 30 and 90 and 20–70, respectively, depending upon the amount of 13X taken in the initial feed. Likewise, entries 7–10 (Table I) and entries 11, 12 (Table I) indicate that % loading of PPY and PANI per gram of PPY-13X and PANI-13X composites varied between 33 and 60 and 40–50, respectively.

A comparison of the data of entries 7,8,11,12 of Table I and entries 1,4,5,6,9,10 of Table II indicate that the % of PPY and PANI formed in the latter system was low. This could possibly be due to the interaction of Cu^{2+} ions with electron-rich PNVC-13X moieties that reduce the % conversion to PPY and PANI.

FTIR absorption characteristics

Table III summarizes FTIR absorption peaks (cm^{-1}) observed for various 13X zeolite based composite systems (PNVC-13X, PPY-13X, and PANI-13X) along with their assignments. These data matched with the characteristic FTIR absorptions reported for PNVC, PPY, and PANI homopolymers.[12,13] FTIR spectra of PPY-(PNVC-13X) and PANI-(PNVC-13X) composites showed absorption peaks characteristic of PPY, PNVC and PANI, PNVC, respectively. Thus formation of these polymers and their presence in the respective

TABLE II
Some Typical Data on Polymerization[a] of PY and ANI vis-à-vis Composite Formation with PNVC-13X-Zeolite

Entry no.	Reactants (g)				Product	
	PNVC-13X	PY	ANI	Composite (g)	% PPY loading[b]/ g of composite	% PA NI loading[c]/g of composite
1	2.4	—	1.5	4.5	—	22
2	2.4	—	2.0	6.0	—	38
3	2.4	—	2.5	7.5	—	44
4	1.8	—	1.5	2.7	—	33
5	1.2	—	1.5	2.2	—	45
6	2.4	1.5	—	3.4	29	—
7	2.4	2.0	—	4.0	40	—
8	2.4	2.5	—	4.4	45	—
9	1.8	1.5	—	2.7	33	—
10	1.2	1.5	—	2.2	45	—

[a] Polymerization conditions: total volume = 50 ml; room temperature; entries 1–5 and entries 6–10, polymerization of PY and ANI in the presence of aqueous dispersion of PNVC-13X by $CuCl_2$.
[b] % PPY loading/g of composite was calculated by the following relation: {(column 6 − column 2)/column 6} × 100.
[c] % PANI loading/g of composite was calculated by the following relation: {(column 7 − column 2)/column 7} × 100.

13X zeolite based composite was endorsed by FTIR spectral analysis.

Scanning electron micrographic characterization

Figure 1 represents the scanning electron micrographs (SEM) of (a) 13X, (b) PNVC-13X, (c) PANI- 13X, (d) PPY-13X, (e) PPY-(PNVC-13X) and (f) PANI-(PNVC-13X), respectively. In general, the SEM image of 13X zeolite showed the presence of small, nearly spherical particles with irregular sizes (1.5–3.0 μm). In contrast,

SEM images of PNVC-13X, PANI-13X and PPY-13X composites indicated the formation of lumpy agglomerates of much larger particles with nonuniform sizes (5–15 μm for PNVC-13X, 4–10 μm for PPY-13X composites and 3–6 μm for PANI-13X, respectively). SEM images of PPY-(PNVC-13X) and PANI-(PNVC-13X) composites showed the presence of densely packed agglomerates of particles (diameters in the range of 6–20 and 60–200 μm, respectively) with a tendency to form larger lumps in either systems compared to that of PNVC-13X, PPY-13X, and PANI-13X composite

TABLE III
FTIR Absorption Characteristics

| Observed peaks (cm^{-1})					Assignment of peaks
PNVC-13X	PPY-13X	PANI-13X	PPY-(PNVC-13X)	PANI-(PNVC-13X)	
724			720	734	Ring deformation of substituted aromatic structure (PNVC)
745			750	740	CH_2 rocking vibration (PNVC)
1158			1145	1150	Out of plane deformation of vinylidene gr(PNVC)
1383			1385	1380	CH_2 deformation of vinylidene gr (PNVC)
1415–1484			1400–1475	1410–1480	Ring vibration of NVC moiety (PNVC)
1630			1635	1640	C═C stretching vibration of vinylidene gr(PNVC)
3089			3065	3075	Aromatic C—H stretching vibration (PNVC)
	1047		1045		C—H vibration of 2,5-disubtituted pyrrole(PPY)
	1530		1537		2,5-substituted pyrrole (PPY)
	2316		2315		Aromatic ring vibration of pyrrole (PPY)
	3309		3315		H-bonded N—H stretching vibration (PPY)
		1243–1318		1240–1320	C≡N stretching vibration (PANI)
		1484–1635		1480–1640	N—H bending vibration (PANI)
		3425		3420	NH stretching vibration (PANI)
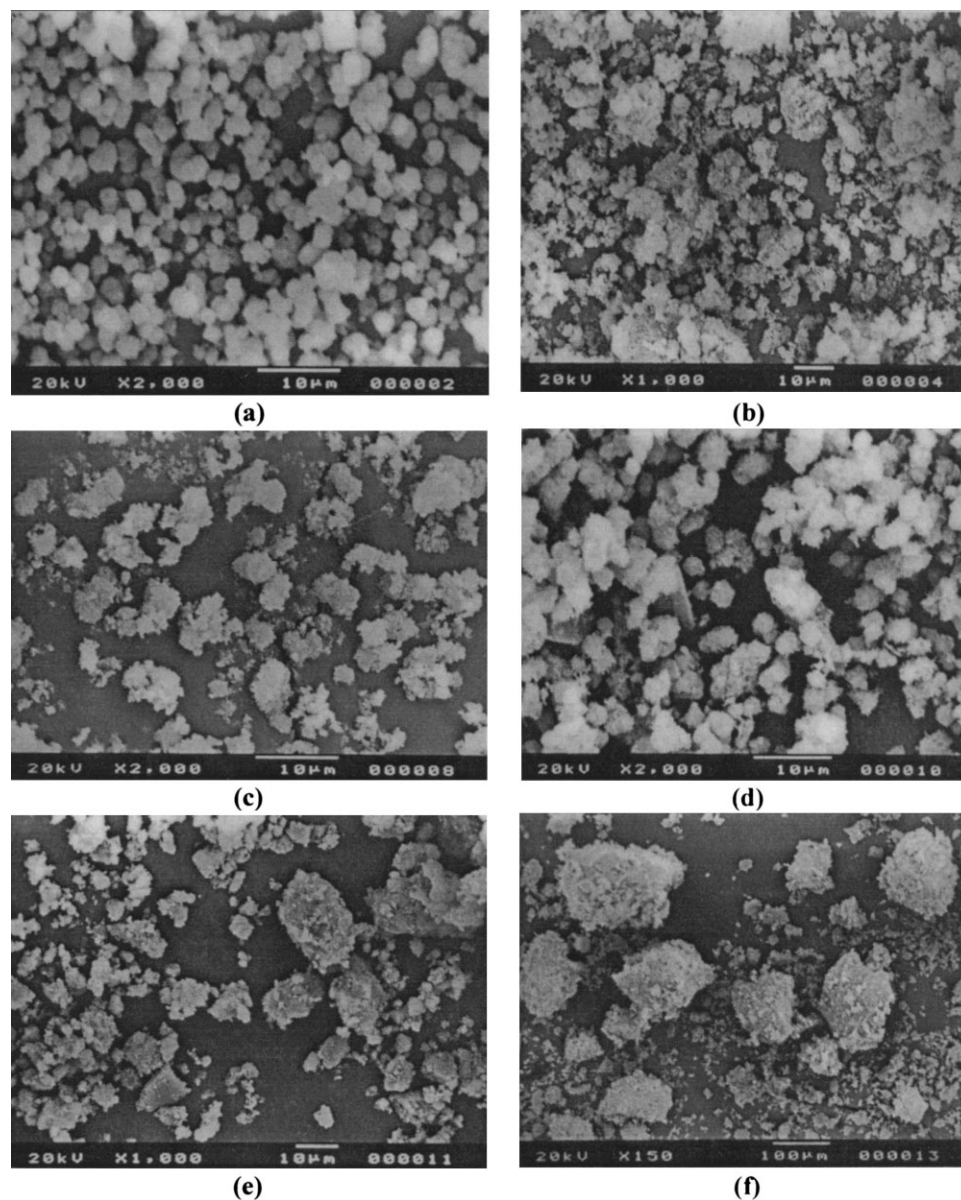
Figure 1 SEM image of (a) 13X-zeolite, (b) PNVC-13X zeolite composite, (c) PPY-13X zeolite composite, (d) PANI-13X zeolite composite, (e) PPY-(PNVC-13X) composite, (f) PANI-(PNVC-13X) composite.

particles. The formation of such aggregates could result through cementation of ultra-fine 13X particles with precipitating polymeric moieties, which is consistent with SEM images of various polymer-metal oxide composite systems recently reported by us.[14–20]

X-ray diffraction pattern characteristics

Figure 2 presents the XRD scans (2θ versus intensity plot) of (a) 13X, (b) PNVC-13X, (c) PPY-13X, and (d) PANI-13X composites, respectively. Interestingly, Figure 2(a) and (b) are almost similar in nature, which suggests the retention of 3D-network of 13X zeolite[1] in the PNVC-13X composite. This could possibly be due

to onset of polymerization of NVC monomer at the active sites present on the surface rather than those in the channels of 13X zeolite powder. In contrast, however, Figure 2(c) and (d) indicates that the XRD patterns were somewhat different. Being small molecules unlike NVC, the polymerization of these monomers might occur in the channels of 13X zeolite and the growth of the polymers might damage the 3D network of 13X zeolite structure.

Thermal stability characteristics

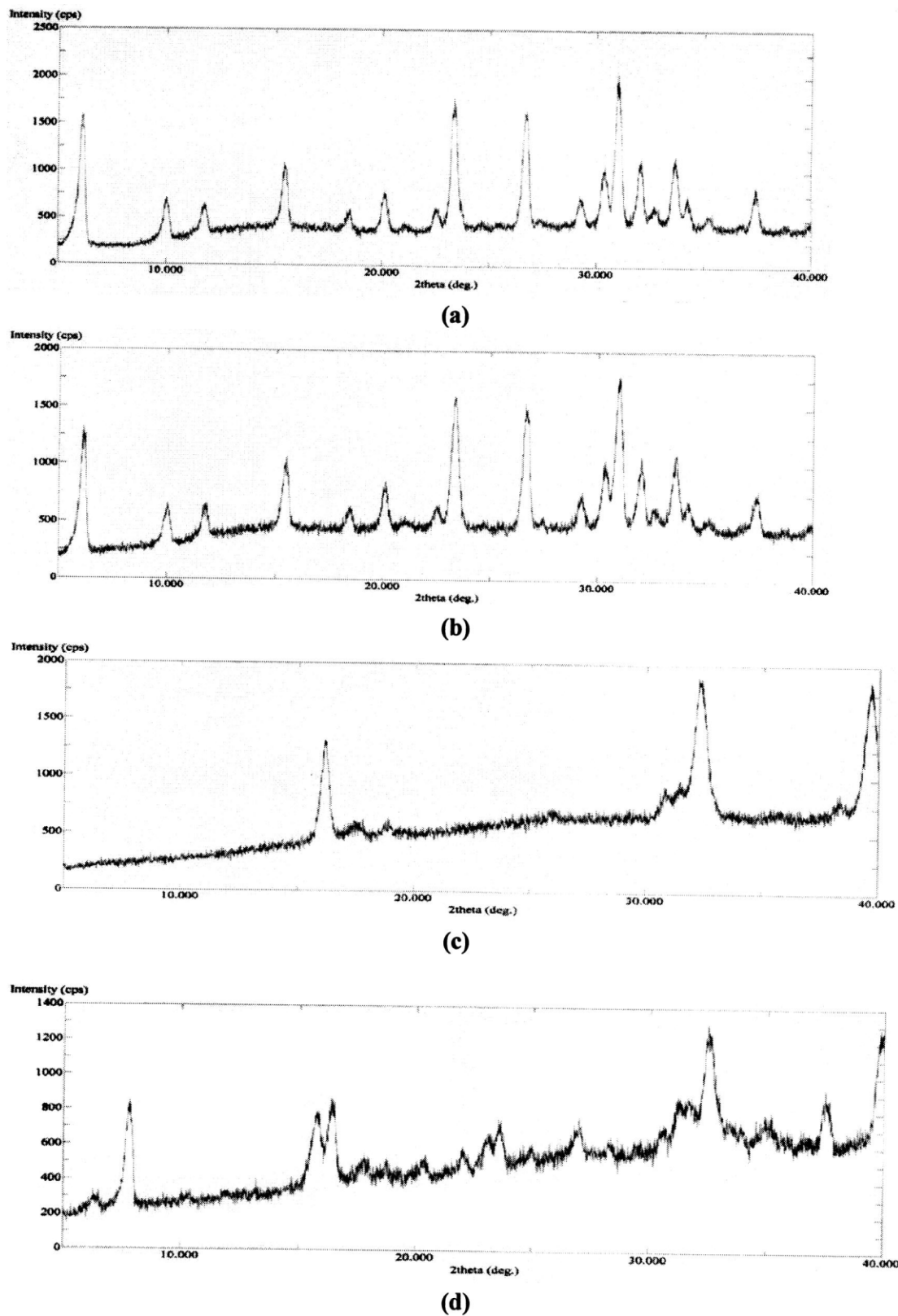Table IV presents the % weight loss versus temperature (°C) data for 13X zeolite and of various polymer-

Figure 2　XRD scan for (a) 13X zeolite, (b) PNVC-13X zeolite composite, (c) PPY-13X zeolite composite, (d) PANI-13X zeolite composite.

TABLE IV
Thermogravimetric Stability Data of 13X-Zeolite, 13X-Polymer Composites, and Respective Homopolymers

Materials	% weight loss (temperature, °C)			
	200	400	600	800
13X	0	20	20	20
PNVC-13X	0	40	40	40
PPY-13X	0	55	60	60
PANI-13X	0	60	65	65
PNVC	0	20	100	—
PPY	0	40	60	90
PANI	0	39	100	—

13X zeolite composites including those of the respective homopolymers. The trends in the thermogravimetric stability are as follows: 13X zeolite > PNVC-13X composite > PNVC homopolymer; 13X zeolite > PPY-13X composite > PPY homopolymer and 13X zeolite > PANI-13X composite > PANI homopolymers. This observation is in line with those reported in the case of polymer-metal oxide and polymer-clay (MMT) composite systems.[12,14–20]

Conductivity characteristics

Table V summarizes the dc conductivity values for PNVC-13X, PPY-13X, and PANI-13X composites and the PPY and PANI modified PNVC-13X composites with a few other composites reported for these polymers. PNVC-13X composite is essentially a nonconductor; however, loading of conducting PPY and PANI moieties improved the conductivity values of PNVC-13X significantly. This feature is consistent with our earlier, recent observations on such conductivity modification of a variety of nonconducting polymer composite systems.[14,17,19,21] The dc conductivity values of PPY-13X and PANI-13X composites were in the order of 10^{-4} S/cm in either system. Interestingly, the dc conductivity values of PPY-13X composites pre-

pared in $CHCl_3/FeCl_3$ system showed 10-fold improved conductivity value compared with that prepared in $H_2O/CuCl_2$ system. Toshima et al.[22] also reported variation of conductivity of PPY samples prepared in various solvents and oxidants.

Mechanistic aspects

The bulk polymerization of NVC by 13X without any extraneous oxidant is consistent with the results of earlier studies by Biswas and Maity.[23] The formation of colorless PNVC in the system indicated that charge-transfer polymerization was unlikely. Initiation of NVC was by interaction between the active Lewis acids sites as well as Bronsted acid sites[23] present in 13X zeolite and the π-electrons of vinyl group in NVC, schematically shown in Scheme 1(a). Relevantly, a similar pathway was believed to be operative in NVC-MMT and NVC-3d-transition metal polymerization systems, and propagation would follow a carbocationic pathway.[2,12,24] Termination would be by H transfer or by impurities like adventitious water.[25] The oxidative polymerizations of PY and ANI by Cu (II) oxidant followed the pathway as shown in Scheme 1(b,c), respectively.[22,26]

CONCLUSIONS

Polymerization of NVC was achieved by 13X zeolite alone and a PNVC-13X composite was isolated. Composites of PPY and PANI with 13X zeolite were prepared by aqueous/nonaqueous polymerization of the respective monomers in presence of dispersions of 13X zeolite powder. SEM images of various composites indicated formation of composite particles in the micrometer range. PNVC-13X composite was essentially nonconductor, while PPY-13X and PANI-13X composites showed conductivity values in the order of 10^{-3} and 10^{-4} S/cm, respectively. Conductivity of the PNVC-13X composite could be significantly improved

TABLE V
Conductivity Data for Various PNVC, PPY, and PANI Based Composites

Entry no.	Materials	Oxidant systems	Conductivity (S/cm)	Reference
1	PNVC-13X	Bulk (65°C) and toluene (110°C)	$<10^{-8}$	This study
2	PPY-13X	$CuCl_2/H_2O$	10^{-4}	This study
3	PPY-13X	$CHCl_3/FeCl_3$	10^{-3}	This study
4	PANI-13X	$CuCl_2/H_2O$	10^{-4}	This study
5	PPY-(PNVC-13X)	$CuCl_2/H_2O$	10^{-5}	This study
6	PANI-(PNVC-13X)	$CuCl_2/H_2O$	10^{-6}	This study
7	PANI-Al_2O_3	$FeCl_3/H_2O$	10^{-3}	[17]
8	PPY-Al_2O_3	$FeCl_3/H_2O$	10^{-4}	[14]
9	PANI-(PNVC-Al_2O_3)	$FeCl_3/H_2O$	10^{-3}	[17]
10	PPY-(PNVC-Al_2O_3)	$FeCl_3/H_2O$	10^{-4}	[14]
11	PANI-(PAN-Al_2O_3)	K_2CrO_4/HCl	10^{-3}	[21]
12	PPY-(PAN-Al_2O_3)	K_2CrO_4/HCl	10^{-4}	[21]
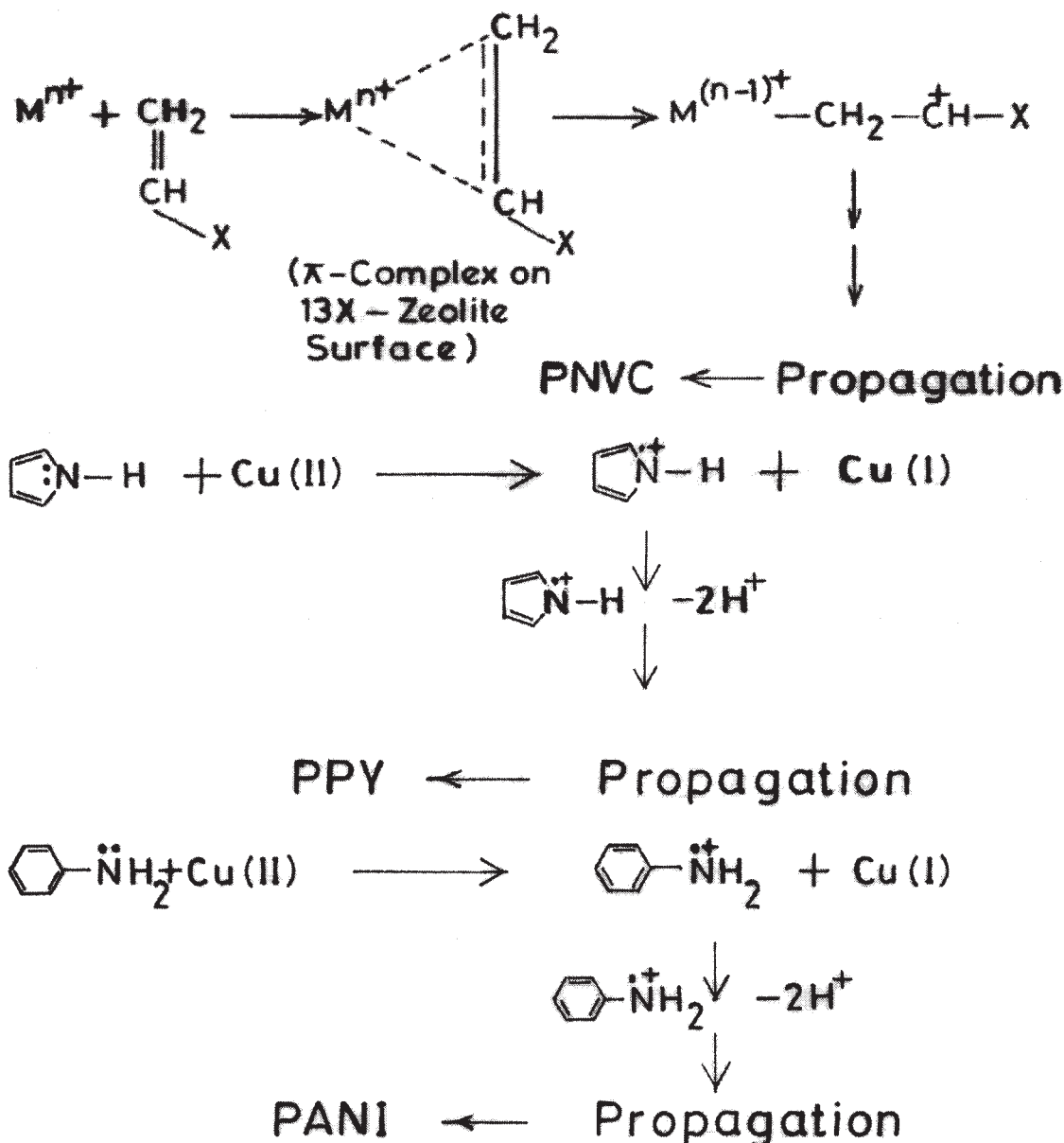
Scheme 1 (a) A possible mechanism for the polymerization of NVC catalyzed by 13X- zeolite, (b) a possible mechanism for the polymerization of PY catalyzed by Cu (II), and (c) a possible mechanism for the polymerization of NVC catalyzed by Cu (II), respectively.

upon incorporation of the conducting PPY or PANI moieties.

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