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Heavy Metal Separation from Industrial Effluent and Synthetic Mixtures using Newly Synthesized Composite Material

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Abstract

A stable composite cation exchange adsorbent for the treatment of heavy metal ions has been synthesized by sol-gel method and characterized by FTIR, XRD, SEM, TGA and TEM analysis. Ion-exchange capacity, pH titration, elution behavior and distribution studies have been also carried out to determine the primary ion-exchange characteristics of the composite. The material shows exchange capacity of 1.49 meq g^{-1} (for Na^+). The composite material exhibits improved ion-exchange capacity, chemical and thermal stability. It can be used up to 300°C with 87.5% retention of initial ion-exchange capacity. The pH titration data reveals bifunctional behaviour of composite. On the basis of distribution coefficient (Kd), the material has been selective for Cd(II), Ba(II), Hg(II) and Pb(II) ions. A number of important and analytically difficult quantitative separations of metal ions have been achieved using columns packed with this exchanger. The composite cation exchanger has been applied for the treatment of sewage water and synthetic mixture successfully.

Keywords: Nano-composite; Ion exchange; Selectivity; Separation

Introduction

Any undesirable change in the water when happens, we call it a water pollution. There are so many causes of this water pollution. Presence of toxic metal ions like lead, mercury, cadmium, etc. is one of them, which has become a major environmental problem that is gradually affecting our ecosystem adversely and getting accumulated in living organisms, due to this human beings are suffering from large number of diseases. The toxic heavy metals are released from anthropogenic activities such as metallurgical, galvanizing, metal finishing, electroplating, mining, power regeneration, electronic devices manufacturing and tannery industries [1]. These metals, when present in the water more than the permissible limit, become unsafe to the health. So, it is indispensable to remove these heavy metals from the water before they are supplied for potable and others useful purposes.

The number of procedures have been introduced and developed for the removal of these heavy metals e.g. solvent extraction, adsorption, preconcentration, reverse osmosis and ion exchanger [2]. Ion exchangers have several advantages over other methods because it is relatively clean, energy efficient and show selectivity for certain ions even in the solution of low concentration of the target ion. Furthermore, it has better exchange capacity, high removal efficiency, fast kinetics [3,4] and can also be utilized in metal recovery process, which are of economical importance [5]. A number of organic and inorganic ion-exchangers have been developed but there are certain limitations existing with them. The main drawbacks associated with organic ion-exchangers are of poor mechanical strength and low thermal stability under high radiation conditions while inorganic ion-exchangers are not suitable for column applications. Hence, in order to overcome all the above limitations, composite ion-exchangers have been synthesized by incorporating organic polymer with the matrix of inorganic precipitate. The composite materials show better exchange capacity, granulometric properties, reproducibility, and chemical stability along with thermal stability and also possess better selectivity for heavy metals compared to pure inorganic and organic materials. These composite ion-exchange materials can also be used as a catalyst [6,7] ion-exchanger [8-10], ion selective electrode [11] and also finds a large number of applications in pollution control and water treatment [12]. In continuation of the previous work [13], we herein describe synthesis, characterization and analytical applications of polyaniline Zr(IV) molybdophosphate composite

Experimental

Materials and methods

The reagents used for the synthesis of composite were Zirconium oxychloride CDH (India), sodium molybdate CDH (India), orthophosphoric acid, N-methylaniline and ammonium persulfate from Merck (India). All other reagents and chemicals were of analytical grade. A digital pH meter of Elico (EL-10, India) was used in pH measurements. Fourier Transform-IR Spectrophotometer (Perkin Elmer (1730, USA) was used to record FTIR spectra using KBr disc method. Thermal Analysis (TGA/DTA) was carried out by (DTG-60 H; C305743 00134 Schimadzu, Japan) at a rate of 10°C min^{-1} in nitrogen atmosphere. An X' Pert PRO analytical Diffractometer (PW-3040/60 Netherlands, Holland with Cu-Kα radiation λ=1.5418A°) was used for X-ray diffraction (XRD) measurement. The morphology of composite material was characterized by scanning electron microscopy, SEM (SEM; LEO, 435 VF). Transmission Electron Microscopy (TEM) analysis was carried out by Jeol H-7500 Microscope. Atomic Force Microscopy (AFM, Veeco; Digital Instruments; Innova) was used with a typical resonance frequency of ca 300 Hz. The UV-Visible spectrophotometric experiments were carried out using a Shimadzu UV-1601 spectrophotometer. To determine the ion-exchange capacity, 1.0 g dry cation exchanger in H^+ form was packed in a column (1.0 cm

internal diameter) fitted with glass wool at the bottom. The nitrates of metal ions were used as eluent to elute the H^+ ions completely from the cation exchanger column. The effluent was titrated against a standard solution of 0.1 M NaOH.

Preparation of reagents

Solutions of Zirconium oxychloride (0.25 M), orthophosphoric acid (0.25 M), sodium molybdate (0.25 M) solutions were all prepared in demineralised water (DMW) while a 10% solution (v/v) of aniline and 0.1 M potassium persulphate were prepared in a 1 M HCl solution.

Preparation of polyaniline Zr(IV) molybdophosphate

Poly N-methylaniline gel was synthesized using the same method [14]. The inorganic precipitate of Zr(IV) molybdophosphate was prepared by mixing 0.25 M solutions of orthophosphoric acid, sodium molybdate, and Zr(IV) oxychloride steadily with continuous stirring at 25°C for 1 h whereby a yellow gel type slurry was obtained. The pH of the solution was maintained by adding a dilute solution of HCl or HNO_3. The resulting yellow precipitate formed was kept overnight in the mother liquor for digestion. The poly N-methylaniline Zr(IV) molybdophosphate composite material was prepared by mixing of inorganic precipitate and polyaniline gel (in 1:1 volume ratio) with continuous stirring for 1 h at 25°C. The resultant d ark green gel obtained was kept for 24 h at room temperature for digestion. The supernatant liquid was decanted, and the gel was filtered under suction. The excess acid was removed by washing with DMW, and the material was dried in an oven at 50°C. The dried material was grounded into small granules, sieved and converted into H+ form by treating with 1.0 M nitric acid solution for 24 h with occasional intermittent shaking and replacing the supernatant liquid with fresh acid. The excess acid was removed after several washings with DMW and finally dried in an oven at 50°C. By applying the above-mentioned chemical route, a number of samples of poly N-methylaniline Zr(IV) molybdophosphate composite

were synthesized under different conditions of mixing volume ratios of reactants. On the basis of good yield, highest ion exchange capacity (for K+ ions), sample A-9 was selected for detail studies. The proposed structure of poly N-methylaniline Zr(IV) molybdophosphate is shown in Figure 1.

Ion exchange capacity

To determine ion exchange capacity, one gram of the exchanger (H+ form) was taken into a glass column (0.5 cm, internal diameter) plugged with glass wool at the bottom. The length of bed was approximately 1.5 cm in height. Alkali and alkaline earth metal nitrates (0.1 M) were used to elute H+ ions from the cation-exchanger. The flow rate of column is maintained at 1.0 mL min^{-1}. The collected effluent was titrated against standard solution of 0.1 M NaOH.

pH titration

In order to determine the nature of the ionogenic group, pH titrations were performed in various alkali and alkaline earth metal chlorides and their corresponding hydroxides using Topp and Pepper method [15]. In this method 0.5 g of the exchanger (H+ form) is taken in each of several 250 mL conical flasks which were followed by the addition of equimolar solution of 0.1 M solutions of alkali or alkaline earth metal chlorides and their corresponding hydroxides in different volume ratios. The final volume is adjusted to 50 mL to maintain the ionic strength.

Effect of eluent concentration

To find out the optimum concentration of the eluent for complete elution of H+ ions, a fixed volume (250 mL) of $NaNO_3$ solution of different concentrations were passed through the columns, containing 1.0 g of the exchanger (H+ form) with a flow rate of 0.5 mL min^{-1}. The effluents were titrated with a standard solution of 0.1 M NaOH to find the H+ ions eluted out.

Figure 1: Scheme showing the preparation of poly N-methylaniline Zr(IV) molybdophosphate

Elution behavior

A column containing 0.5 g of exchanger (H$^+$ form) was eluted with 1.0 M sodium nitrate solution. The effluent was collected in 10.0 mL fractions. Each fraction of 10.0 mL was titrated against a standard solution of sodium hydroxide.

Selectivity (sorption) studies

The distribution coefficient (Kd) of metal ions was determined by batch method in different solvents of analytical interest. Distribution coefficient is actually used to access the overall ability of the material to remove the ions of interest. The various portions of (300 mg each) of poly N-methylaniline Zr(IV) molybdophosphate (H$^+$ form) were taken in Erlenmeyer flasks and titrated with 30 mL of different metal nitrate solution in the required medium and subsequently shaken for 6 h in temperature controlled shaker at 25°C to attain the equilibrium. The concentration of metal ion before and after the equilibrium was determined by titration against a standard solution of 0.01 M di-sodium salt of EDTA. The distribution coefficients (Kd) were calculated using the equation:

$$\frac{milli\ equivalent\ of\ metal\ ions\ /\ gm\ of\ ion-exchanger}{milli\ equivalent\ of\ metal\ ions\ /\ mL\ of\ solution} \left(mLg^{-1} \right)$$

$$K_d = I - F/F \times V/M\ mL\ g^{-1}$$

where I is volume of EDTA used for metal ion solution without treatment with exchanger. F is the volume of EDTA consumed by metal ion left in solution phase after treatment. The sorption of metal ions involves the ion exchange of the H$^+$ ions in exchanger phase with that of metal ions in solution phase

For example:

$$2R-H^+ + M2^+ \rightleftharpoons R2-M + 2H^+$$

Exchangephase   Solutionphase   Exchangephase   Solutionphase

where R=Poly N-methylaniline Zr(IV) molybdophosphate

Quantitative separations of metal ions in synthetic binary mixtures

Quantitative separations of some selective metal ions were achieved on columns of poly N-methylaniline Zr(IV) molybdophosphate. 1 g of exchanger (H$^+$ form) was packed in a glass column (0.5 cm, internal diameter) with a glass wool support at the bottom. The column was washed thoroughly with demineralised water and the mixture of two metal ions (each with initial concentration 0.1M) was loaded on it and allowed to pass through the column at a flow rate of 1 mL min^{-1} till the solution level was just above the surface of the composite material. The process was repeated twice or thrice to ensure the complete absorption of metal ions on the bed. The separation of metal ions is achieved by collecting the effluent in 10 mL fractions and titrating against the standard solution of 0.01M di-sodium salt of EDTA.

Selective separation of metal ion from a synthetic mixture

Selective separation of Cd^2+ and Hg^2+ from the synthetic mixtures containing (Fe^{3+}, Cu^{2+}, Al^{3+}, Sr^{2+}, Ca^{2+}, Zn^{2+}, Ba^{2+}) and (Ca^{2+}, Sr^{2+}, Fe^{3+}, Cu^{2+}, Cd^{2+}, Hg^{2+}) was achieved on poly N-methylaniline Zr(IV) molybdophosphate columns. The amount of the Cd^{2+} and Hg^{2+} ions in the synthetic mixture was varied keeping amount of the other metal ions constant.

Results and Discussion

The number of samples of poly N-methylaniline Zr(IV) molybdophosphate were prepared by sol-gel method for the development of composite ion exchanger (Table 1). The ion-exchange capacity of the synthesized material depends upon the pH and mixing ratio of the reagents. It is clear from Table 1 that with increasing pH (0.6-2.0) of the reaction mixture, ion-exchange capacity of composite material decreases (because at higher pH, the hydrolysis of composite material occurs). In order to screen the working ability of the composite exchanger, ion-exchange capacity for some monovalent and divalent cations were determined (Table 2) which follows the sequence Li$^+$<Na$^+$<K$^+$ and Mg^{2+}<Ca^{2+}<Sr^{2+}<Ba^{2+}, respectively. The ion exchange capacity for alkali and alkaline earth metal ions increases with decrease in hydrated ionic radii [16].

Hence, it may be suggested that the ions with smaller hydrated radii enter the pores of the exchanger more easily, resulting in higher sorption [17]. The interesting feature shown by the material is that ion-exchange capacity for alkaline earth metal ions was found to be greater than alkali metal ions.

It was observed from Figure 2 that for complete elution of H$^+$ ions (from 1.0 g of exchanger), the optimum concentration of the eluent was found to be 1.0 M. It was also observed (Figure 3) that the rate of exchange (H$^+$ ions) is quite fast in the beginning and only 120 mL of NaNO$_3$ solution (1.0 M) is sufficient for complete elution of H$^+$ ions from the column containing 1.0 g exchanger. From this experiment it is found that the efficiency of the column is quite satisfactory.

The pH titration curves for alkali and alkaline earth metals show two inflection points which indicate bifunctional strong cation exchange behavior (Figures 4 and 5). Initially alkali and alkaline earth metal chlorides are added in the absence of base. There is a sharp decrease in pH due to the release of hydrogen ions.

S. no	A mol L^{-1}	B mol L^{-1}	C % v/v	Mixing ratio v/v/v	Temp.	pH	Appearance of bead	Exchange capacity	Yield (g)
A-1	0.1	0.1	–	01:01	25 ± 2 °C	0.6	Yellow	0.5	1.82
A-2	0.1	0.1	–	01:02	25 ± 2°C	1	Yellow	0.85	2.35
A-3	0.1	0.1	–	02:01	25 ± 2°C	1.4	Yellow	0.72	1.88
A-4	0.1	0.1	–	01:01	25 ± 2°C	1.7	Yellow	0.71	2.08
A-5	0.1	0.1	20	01:01:01	25 ± 2°C	0.6	Green	0.00	0.00
A-6	0.1	0.1	20	01:01:01	25 ± 2°C	1	Green	1.63	2.71
A-7	0.1	0.1	20	01:01:01	25 ± 2°C	1.4	Green	1.37	2.93
A-8	0.1	0.1	20	02:01:01	25 ± 2°C	1.7	Green	1.31	3.42
A-9	0.1	0.1	20	01:02:01	25 ± 2°C	2	Green	1.51	3.64
A. Zr(IV) oxychloride, B. Sodium molybdate, C. Stock solution of 20% polyN-methylaniline									

Table 1: Conditions for the synthesis of poly N-methylaniline Zr(IV) molybdophosphate cation exchanger.

Exchanging ions	Ionic radii (A°)	Hydrated Ionic radii (A°)	Ion exchange capacity (meq g-1)
Li⁺	0.68	3.4	1.12
Na⁺	0.97	2.76	1.49
K⁺	1.33	2.32	1.35
Mg²⁺	0.78	7	1.06
Ca²⁺	1.43	5.9	0.91
Sr²⁺	1.27	6.3	0.98
Ba²⁺	1.43	5.9	1.1

Table 2: Ion-exchange capacity of various exchanging ions on poly N-methylaniline Zr(IV) molybdophosphate cation exchanger.
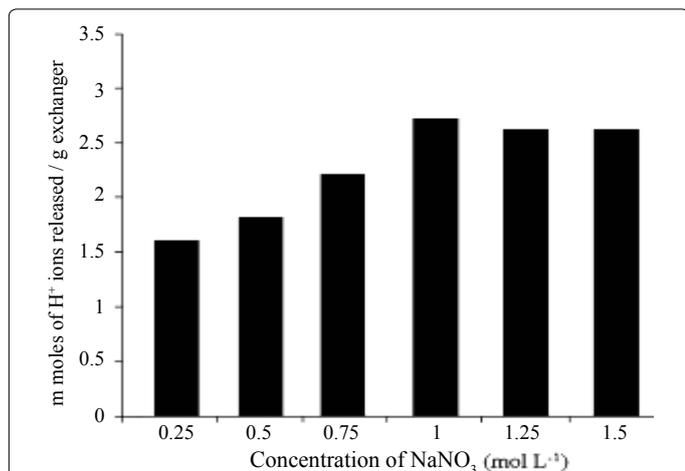

Figure 2: Effect of eluent concentration on ion-exchange capacity of poly N-methylaniline Zr(IV) molybdophosphate.
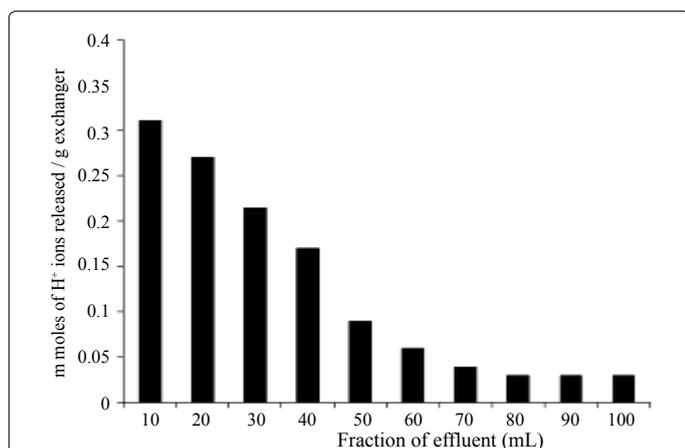
Figure 3: The elution behaviour of poly N-methylaniline Zr(IV) molybdophosphate nano-composite.

The effect of heating on the composite material at different temperatures for 1 h (Table 3) indicates that the ion-exchange capacity and physical appearance of the exchanger changed as temperature increased. The material appears to be thermally stable up to 300°C as it retains significant ion-exchange capacity. From this observation it is clear that the composite cation -exchanger possessed potential thermal stability and ion-exchange capacity.

TGA and DTG curves (Figure 6) of the nano-composite show a weight loss of mass (about 11%) continuous up to 150°C, which is due

to the removal of water molecules [18]. The weight loss in the region of 200-300°C accounts for the loss of interstitial water molecules due to the condensation of -OH groups. Further, weight loss from 320°C to 500°C may be due to complete decomposition of the organic part of the exchanger. A gradual decrease in weight loss beyond 500°C may be due to the formation of metal oxide. The main purpose of the TGA experiment is to study the thermal degradation and stability of composite material.

The Figure 7 depicts the FTIR spectrum of poly N-methylaniline Zr(IV) molybdo phosphate composite which indicates the characteristic absorption peaks shown by the composite material. The three composites show broad absorption peaks at ν 3450-3467 cm⁻¹ for (OH) and ν 32450-3390 cm⁻¹ for (NH). The strong absorption at ν
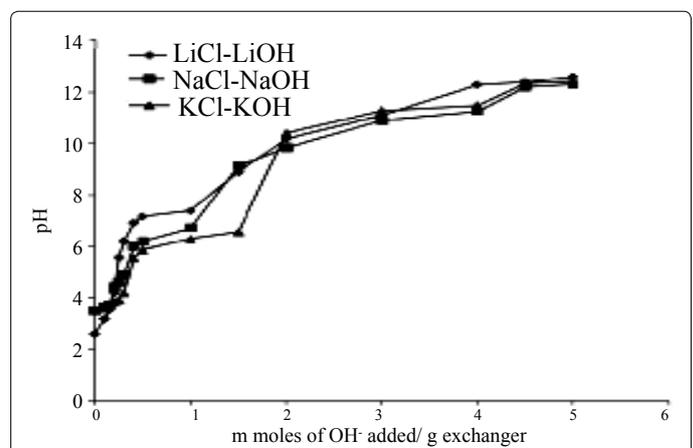
Figure 4: pH-titration curves of poly N-methylaniline Zr(IV) molybdophosphate with various alkali metal hydroxides.
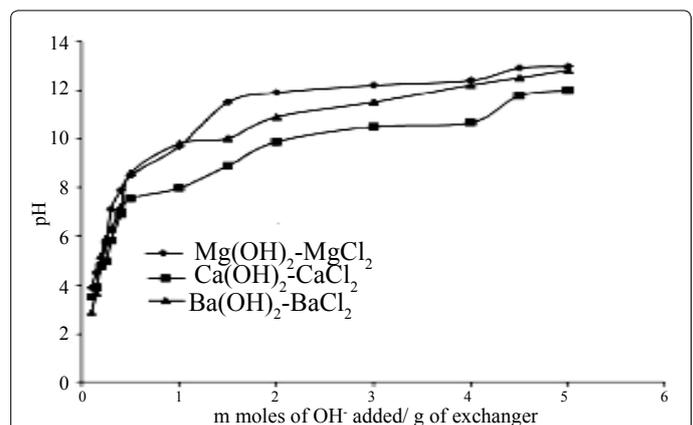
Figure 5: pH-titration curves of poly N-methylaniline Zr(IV) molybdophosphate with various alkaline earth metal hydroxides.

Temperature (°C)	Color	IEC (meq g⁻¹) for Na⁺	% retention of IEC
50	Green	1.49	100
100	Green	1.38	94.6
200	Dark Green	1.33	90.3
300	Black	1.28	87.5
400	Black	1.14	78.7
500	Muddy color	0.91	69.4

Table 3: Effect of temperature on the ion-exchange capacity of poly N-methylaniline Zr(IV) molybdophosphate cation exchanger on heating time for 1 h.
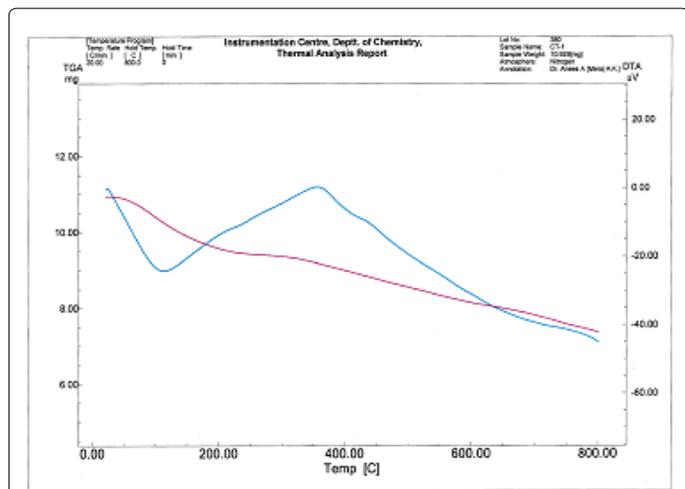
Figure 6: TGA curve of poly N-methylaniline Zr(IV) molybdophosphate cation exchanger.
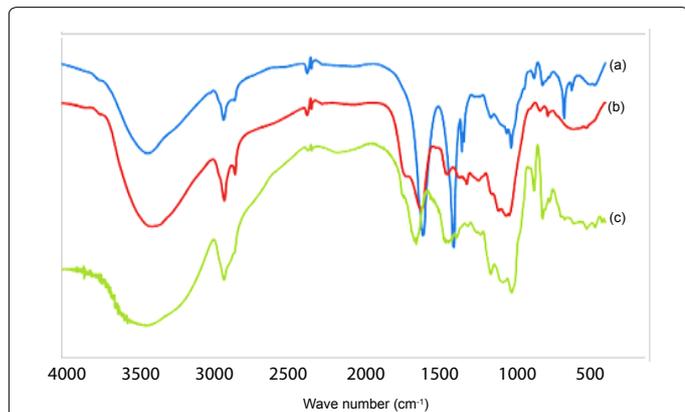

Figure 7: FTIR spectrum of poly N-methylaniline Zr(IV) molybdophosphate.

1590-1641 cm^{-1} (C=C), 3050-3100 (Aromatic), 1220-1245 (P=O) and the weak absorption peaks at ν 530 cm^{-1} and 460 cm^{-1} corresponds to (Zr-N) and (Zr-O).

A typical powder X-ray diffraction (XRD) pattern of nano-composite at room temperature is shown in Figure 8. The analysis shows that the nano-composite is formed in single phase with tetragonal crystal symmetry. All the peaks are very well matched with the crystal structures and no indication of a secondary phase is found. The lattice parameters calculated from the XRD pattern are given in Table 4, which are quite close to the values reported in the literature [19-21]. The maximum deviation that occurred between the observed and calculated values of interplanar spacing (d) remains below 0.0011 Å. In order to calculate the particle size (D) of the nano composite, Scherer's formula was used which is as follows:

$$D = \frac{0.9\lambda}{B\cos\theta} \tag{1}$$

Where λ is the X-ray wavelength (1.5418 Å), B is the full width at the half-maximum (FWHM) of the most intense peak and θ is the diffraction angle.

The particle size of the nano composite was found to be ~19.2 nm, estimated from the line width of the (101) XRD peak. Well-crystallized

diffraction peaks were observed, from which the calculated d-values are in good agreement with those given in the standard data (JCPDS, 36-1451) for nano composite (Table 4). This suggests that nano-composite has crystallized in a tetragonal symmetry. Well-crystallized diffraction peaks were also observed for the nano-composite from which cell parameters, lattice type and d-values were calculated (Table 4) which were also in good agreement with those given in the standard data (JCPDS, 36-1451). The reason for uniform values of particle size of nano-composite is the preparation mode.

The surface morphology of composite A1-A4 was investigated by using SEM micrographs. The SEM images for the composite A1-A4 is with a width of 14 mm and magnification of 1000X (Figure 9).

The signals that derive from SEM depict information about the external morphology (texture), crystalline structure, homogeneity, thickness and orientation of materials making up the nano-composites. The SEM study of nano composites A1-A4 confirm the semi-crystalline
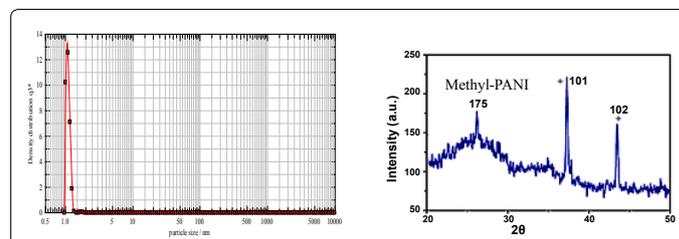

Figure 8: XRD parameters of the nano-composite of poly N-methylaniline Zr(IV) molybdophosphate.

Crystal system Method	Tetragonal Powder
Cell parameters	a=6.8841 b=6.8841 c=13.421 α=90.000 β=90.000 γ=90.000
2θ min-max	19.573-73.298
Particle size	19.2 nm
Lattice Type	p

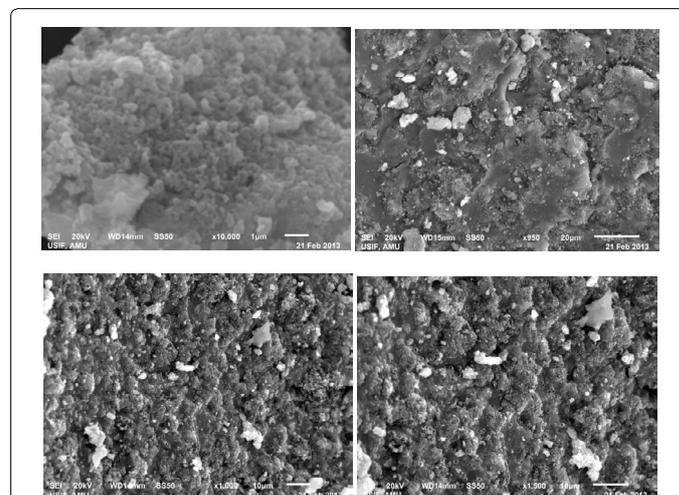**Table 4:** The XRD parameters of the nano-composite prepared in this study.

Figure 9: SEM of poly N-methylaniline Zr(IV) molybdophosphate at 1000X and 14 nm width.

nature of the material and it was found that the morphology of the exchanger gets changed after it binds with an inorganic part [Zr(IV) molybdophosphate] with the matrix of poly N-aniline as shown in Figure 9. As evident from TEM (Figure 10) the particle size in the range of 19.2 nm and with the average particle size of 19.6 nm, 19.2 nm and 19.4 nm.

In order to explore the metal-ion separation potential of the composite cation exchange material, distribution coefficient values (Kd) for different metal ions were checked in different solvent systems (Table 5). It has been observed that on decreasing the concentration of different solvents, the uptake of metal ions was sharply increased (Table 5). The data obtained in Table 5 indicate that metal ions such as Cd(II), Ba(II), Pb(II) and Hg(II) ions showed high uptake (high values of distribution coefficient).

On the basis of higher Kd values, the material was found to be selective for Cd(II), Ba(II), Hg(II), and Pb(II) ions. Some analytically important separations (e.g. $Zn^{2+}–Pb^{2+}$, $Ca^{2+}–Hg^{2+}$, $Fe^{3+}–Hg^{2+}$, $Zn^{2+}–Ba^{2+}$, $Ni^{2+}–Ba^{2+}$, $Fe^{3+}–Cd^{2+}$ and $Ca^{2+}–Pb^{2+}$) of metal ions were carried on the columns of poly N-methylaniline Zr(IV) molybdophosphate composite exchanger (Table 6). The possible reason for 100% recovery of Fe^{3+} may be due to the weak attachment or adsorption of metal ions on the adsorbent.

The Tables 7 and 8 show results of selective separation of Cd^{2+} and Hg^{2+} from the synthetic mixtures of other metals. It is clear that the separations are quite sharp and recovery is quantitative and reproducible. The practical utility of the composite material was demonstrated by separating Cd(II) and Hg(II) from synthetic mixtures.
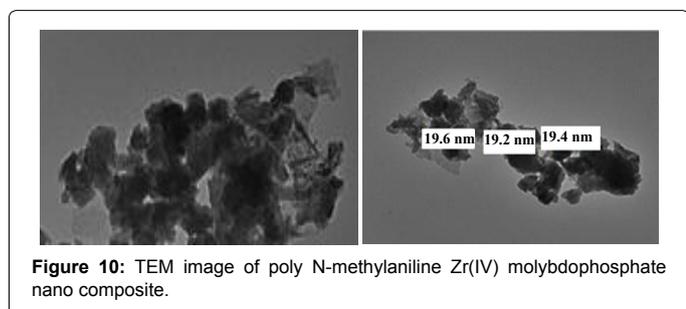

Figure 10: TEM image of poly N-methylaniline Zr(IV) molybdophosphate nano composite.

Metal ion	DMSO 0.4M	DMSO 0.2M	DMSO 0.1M	HNO₃ 0.4M	HNO₃ 0.2M	HNO₃ 0.1M	AcOH 0.4M	AcOH 0.2M	AcOH 0.1M
Ba^{2+}	1107	1233	1390	1703	1922	2093	1911	2073	2153
Bi^{3+}	481	531	605	108	221	387	161	302	418
Mg^{2+}	133	177	222	231	246	381	200	300	389
Zn^{2+}	187	300	371	172	208	243	210	236	254
Ca^{2+}	123	139	153	105	120	138	133	182	237
Cd^{2+}	383	471	604	533	718	961	1042	1193	1311
Cu^{2+}	214	259	374	160	216	351	102	190	232
Ni^{2+}	177	257	382	197	281	369	129	234	391
Hg^{2+}	881	1072	1390	833	1062	1241	718	919	1127
Al^{3+}	133	312	419	176	251	439	202	251	372
Pb^{2+}	243	301	381	431	652	791	1243	1351	1507
Fe^{3+}	98	152	285	87	173	209	100	202	314
Th^{4+}	325	409	502	204	344	471	217	298	346
Sr^{2+}	100	121	141	102	110	122	97	112	128

Table 5: Distribution coefficients (mL g^{-1}) K_d of metal ions on poly N-methylaniline Zr(IV) molybdophosphate cation exchanger in different solvent systems.

S. No.	Metal ion separation (mg)	Amount loaded (mg)	Amount found (mg)	% recovery	Volume of eluent used	Eluent used (mL)
6	Fe^{3+}	5.86	5.34	91	70	2 M HCl
	Cd^{2+}	19.31	18.27	94	60	4 M HCl
4	Zn^{2+}	6.34	6.15	97	60	4 M HNO₃
	Ba^{2+}	15.23	12.03	81	90	2 M HCl
2	Ca^{2+}	4.233	3.92	92	60	0.1 M HCl
	Hg^{2+}	20.51	18.76	91	90	0.4 M AcOH
1	Zn^{2+}	5.821	5.21	89	50	0.1 M AcOH
	Pb^{2+}	20.11	19.33	96	70	0.2 M HCl
5	Ni^{2+}	5.94	5.86	98	60	0.1 M HNO₃
	Ba^{2+}	14.36	13.71	95	80	2 M HCl
7	Ca^{2+}	4.83	4.47	92	70	0.1 M AcOH
	Pb^{2+}	19.28	18.65	97	80	0.4 M HCl
3	Fe^{3+}	5.53	5.79	100	70	2 M HCl
	Hg^{2+}	20.31	19.25	95	70	4 M HCl

Table 6: Quantitative separation of metal ions from a binary mixture using polyN-methylaniline Zr(IV)molybdophosphate cation exchanger columns at room temperature.

S. No	Amount of Cd^{2+} loaded (mg)	Amount of Cd^{2+} found (mg)	% recovery	Eluent Used	Eluent Volume (mL)
1	1.68	1.61	96	0.1M HNO₃	80
2	4.25	4.16	97	0.1M HNO₃	95
3	6.69	6.53	97	0.1M HNO₃	105

Table 7: Selective separations of Cd^{2+} from a synthetic mixture of, Sr^{2+}, Cu^{2+}, Fe^{3+}, Ca^{2+}, Al^{3+}, Zn^{2+} and Cd^{2+} on poly N-methylaniline Zr(IV) molybdophosphate cation exchanger columns.

S. No	Amount of Hg^{2+} loaded (mg)	Amount of Hg^{2+} found (mg)	% recovery	Eluent Used	Eluent Volume (mL)
1	2.43	2.25	92	0.1 M HCl	80
2	5.71	5.53	97	0.1 M HCl	95
3	7.12	7.03	98	0.1 M HCl	105

Table 8: Selective separations of Hg^{2+} from a synthetic mixture of, Sr^{2+}, Cu^{2+}, Fe^{3+}, Ca^{2+}, Cd^{2+} and Hg^{2+} on poly N-methylaniline Zr(IV) molybdophosphate cation exchanger columns.

Conclusions

Novel synthesized semi-crystalline nano-composite cation exchanger shows selective behavior toward heavy metal ions and can withstand fairly high temperature. Thermally stable, it retains significant ion exchange capacity up to 300°C. It can be used for the quantitative separation of metal ions from binary mixture of analytical importance. The material can be explored further for the removal and recovery of important metal ions from industrial effluents. PolyN-methylaniline Zr(IV) molybdate composite cation exchanger thus, exhibits the characteristics of a promising ion-exchanger as well as separating material.

Acknowledgements

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