

Research Article

Application of Composite Cation Exchanger for Remediation of Pollutants from Aqueous Environment

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Abstract

A composite ion exchanger Polyaniline- Cerium(IV) molybdsilicate was synthesized by ex-situ polymerisation method and was characterized by various techniques. The exchanger shows high ion exchange capacity of 2.51 meqg^{-1} as well as good chemical and thermal stability. The pH titration studies revealed bifunctional behavior and cationic nature of the exchanger. The distribution studies of various metal ions showed that the material was highly selective for Bi(III), Hg(II) and Th(IV) ions which are toxic environmental pollutant. On the basis of differential selectivity towards metal ions some of the important binary separations are achieved using composite exchanger. Also the utility of the exchanger in environmental remediation was achieved by removing the highly hazardous organic dye pollutants such as methylene blue and crystal violet from aqueous solution under batch conditions. Results of the present investigation shows that the material is a promising adsorbent for heavy metal ions and a good heterogeneous catalyst for degradation of dyes.

Keywords: Composite exchanger, Polyaniline, Heavy metal sorption, Dye degradation

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Introduction

Discharge of pollutants such as heavy metal ions and organic dyes into the water, even in small concentrations, become a great threat to the human and aquatic life. Series of heavy metals releasing from various industries on the environment poses various detrimental health effect. Besides this, heavy metals are non renewable resources and therefore recovery of metal ions is as important as removal from waste streams [1]. Disposal of dye effluents mainly from textile, paint and varnishes and paper and pulp industries are of great concern because of their toxic and carcinogenic nature arises due to the non biodegradability of the dyes. Inorder to minimize the harmful effect of these effluents to the human and environment, various treatment technologies such as adsorption, ion exchange, chemical precipitation, electro dialysis, etc, were developed [2]. Among these methods ion exchange and adsorption are acceptable because of its cost effectiveness, ecofriendly nature and easy reusability.

Synthetic composite [3-5] ion exchangers are attractive because of its synergic properties of both organic and inorganic moieties. The flexibility of the organic part possess specific chemical reactivity and the presence of inorganic backbones make them thermally and mechanically more stable.

In the present study, the synthesis of novel heteropolyacid salt of cerium and its composite with polyaniline were explained. The utility of ion exchanger Polyaniline-Cerium(IV) molybdsilicate towards the removal of toxic pollutants such as heavy metal and dyes were carried out.

Experimental**Materials and methods**

Sodiumsilicate(LobaChemie,India),ceric(IV)ammoniumnitrate(E.Merck),ammoniummolybdate(E.Merck),aniline(E. Merck) were used for the synthesis of the exchanger. Methylene blue and Crystal Violet were obtained from Merck

and the dye solutions were prepared by dissolving the dye in deionised water. All other reagents and chemicals used were of analytical grade.

ELICO LI613 pH meter was used for pH measurements and an electric thermostat oven was used for heating the sample at various temperatures. UV-Visible spectrophotometer model JASCO V660 was used for spectrophotometric measurements. FTIR spectrometer model Thermo Nicolet Avtar370 for IR studies, X-ray Diffractometer Bruker AXS D8 Advance for X-ray diffraction studies, Perkin Elmer Diamond TG/DTA Analysis System for thermal analysis and an electric shaking machine for shaking were also used. JEOL Model JSM - 6390LV for Scanning Electron Microscopic analysis and chemical composition was determined using EDS.

Synthesis of Polyaniline- Cerium(IV) molybdosilicate (PANI-CeMoSi)

The precipitate of Cerium(IV) molybdosilicate was prepared by adding solution of Ceric(IV) ammoniumnitrate to hot acidic solution of mixture of ammonium molybdate and sodium silicate. Polyaniline gels were prepared by mixing of the acidic solutions of 10% aniline and 0.1M ammonium persulphate in different volume ratios with continuous stirring by a magnetic stirrer, keeping the temperature below 10°C for half an hour. The green gels of polyaniline were added to the yellow inorganic precipitates of Cerium(IV) molybdosilicate and mixed thoroughly with constant stirring. The resultant green colored gels were kept for 24 hrs at room temperature (25 ± 2 °C) for digestion. The supernatant liquid was decanted and gels were filtered under suction. The excess acid was removed by washing with demineralized water (DMW) and the material was dried in an air oven at 50°C. The dried products were immersed in DMW to obtain small granules. They were converted to H⁺ form by treating with 1.0 M HNO₃ for 24 hrs with occasional shaking, intermittently replacing the supernatant liquid with fresh acid. The excess acid was removed after several washings with DMW and the material was dried at 50°C.

Characterisation

Ion exchange capacity (IEC)

Ion exchange capacity [6] of the material was determined by column method. One gram of the exchanger in H⁺ form was taken in the column. The H⁺ ions were eluted by percolating 100 ml of 1M sodium chloride solution at the rate of 5-6 drops per minute. The eluent was collected and titrated against standard sodium hydroxide solution. IEC in milliequivalents of H⁺ ions per gram was calculated from the titre values by using the equation

$$IEC = \frac{av}{w}$$

Where 'a' is the molarity, 'v' is the volume of alkali used during titration and 'w' is the weight of the exchanger taken.

Chemical stability

The chemical stability of the exchanger was assessed in mineral acids like HCl, HNO₃ and H₂SO₄, bases like NaOH and KOH and organic solvents like acetic acid, acetone, ethanol and diethyl ether. For this 500 mg of the exchanger was kept separately in 50 ml of different solvents at room temperature for 24 Hrs. The change in colour and weight were noted.

Specific surface area

The specific surface area of synthesized composite exchanger was computed from the Sear's method [7]. In this method 0.5g of the powdered exchanger was acidified with 0.1M HCl to pH=3. It was then mixed with 10.0g of the NaCl and the final volume was made to 50ml with distilled water. It was then titrated against standard 0.1M NaOH to achieve pH=9. The Specific surface area was calculated using Sear's equation.

$$S = 32V-25$$

Where S is the specific surface area of the exchanger material in m²/g, V is the volume of the standard 0.1M NaOH solution required to raise the pH from 4 to 9.

Effect of temperature on IEC

The effect of temperature on IEC was studied by heating several 100 mg sample of the exchanger at different temperatures for three hours in an air oven and Na⁺ ion exchange capacity in meqg⁻¹ was determined by column method after cooling them to room temperature.

pH titration

Topp and Pepper [8] method was used for pH titration using NaOH/NaCl, KOH/KCl systems. 500 mg of exchanger was equilibrated with varying amounts of metal chloride and metal hydroxide solutions. pH of each solution was measured after attainment of equilibrium and plotted against milliequivalents of OH⁻ ions.

Distribution studies (K_d)

Distribution studies were carried out for various metal ions in demineralised water by batch method. The distribution before and after equilibrium were determined volumetrically using EDTA as the titrant. The K_d values were obtained from the formula;

$$K_d = \frac{I-F}{F} \times \frac{V}{w}$$

Where 'I' is the initial volume of EDTA used, 'F' is the final volume of EDTA used, 'v' is the volume of the metal ion solution and 'w' is the weight of the exchanger.

Analytical applications

Quantitative separation of metal ions from synthetic binary mixtures

Separations of some metal ions of analytical utility were achieved on the column of the exchangers by knowing the effect of electrolyte concentration on distribution coefficients. This was studied by equilibrating known amount of exchanger with metal ion solution in electrolytes of varying concentrations (0.1 M, 0.01 M, 0.001 M NH₄NO₃ and HNO₃. The column (30cm x 0.69cm internal diameter) on which the separations were to be carried out was filled uniformly with the exchanger (5g). First of all distilled water was added to pack the granules so that no air bubbles get stuck. Then the mixture of the metal ion solutions of concentration about 0.005 M (10 ml) was slowly added. The process was repeated for maximum sorption. The exchanged metal ions were eluted using suitable eluent. The rate of flow in all separations was 0.2 ml per minute. The concentration of metal ion in the solution was determined by collecting known volume of effluent at regular intervals. Binary separations were carried out on the column.

Quantitative separation of metal ions from industrial waste water

Industrial wastewater was collected from textile industry and phosphate fertilizer plant. Samples were first filtered to remove any solid particles and then it was neutralised. The colour producing substances were removed by adsorption using charcoal. The samples were chemically treated for the detection and separation of any heavy element present. The granular ion exchanger having greater selectivity towards heavy metals were packed in a column, 100 ml of the sample was passed through the column. The flow rate was maintained at 0.3-0.5 mL/min. Finally the ions were separated using eluents and determined quantitatively.

Application of PANI-CeMoSi as catalyst for the degradation of Dyes

The catalytic activity of PANI-CeMoSi on the degradation of methylene blue (MB) and crystal violet (CV) dye were evaluated in laboratory conditions. For this a stock solution (40 ppm) of MB and CV dye were prepared in deionised water and diluted as and when required. The catalytic degradation was observed by the addition of 200 mg of PANI-CeMoSi to the dye solution at room temperature without any irradiation and external catalyst. Decolourisation of

media was measured by taking optical density at different time intervals using UV-Vis DRS. The degradation efficiency was calculated using Beer's law as;

$$\% \text{ Degradation} = \frac{\text{Initial absorbance} - \text{final absorbance}}{\text{Initial absorbance}} \times 100$$

Results and Discussions

Characterisation

The optimum conditions used for the synthesis and properties of the CeMoSi inorganic ion exchanger and its composite with PANI are summarized in the **Table 1**. The sample, CeMoSi-5 having a mixing volume ratio of $\text{Ce}^{4+}:\text{Mo}^{6+}:\text{Si}^{4+}$: 1:2:2 obtained as Yellowish granular solid shows highest ion exchange capacity (1.14 meq/g). Thus CeMoSi-5 was selected for making composite with PANI and the ion exchange capacity was found to be increased after composite formation. Ion exchange capacity of PANI- CeMoSi was 2.51 meq/g. This increase in IEC probably due to increase in number available of cavities after composite formation and thus enhances the easy mobility of H^+ ions.

Table 1 Condition for the preparations of various samples of CeMoSi and PANI-CeMoSi Composite cation exchange material

Samples	Mixing Volume Ratio					pH	Appearance	Na^+ IEC(meq/g)
	A	B	C	D	E			
PANI	-	-	-	1	1	-	Dark Green Powder	0.24
CeMoSi -1	1	1	1	-	-	1	Yellowish granular	0.75
CeMoSi -2	1	2	1	-	-	1	''	0.91
CeMoSi -3	2	1	1	-	-	1	''	0.82
CeMoSi -4	1	1	2	-	-	1	''	0.63
CeMoSi -5	1	2	2	-	-	1	Dark yellow crystal	1.14
CeMoSi -6	2	2	1	-	-	1	''	0.67
PANI-CeMoSi	1	2	2	1	1	1	Greenish granular	2.51

A-0.1M $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$ in DMW B-0.1M $\text{Na}_2\text{Mo}_4.2\text{H}_2\text{O}$ in DMW C-0.1M $\text{Na}_2\text{SiO}_3.9\text{H}_2\text{O}$ in DMW D- Aniline in 1M HCl E- $(\text{NH}_4)_2\text{S}_2\text{O}_8$ in 1M HCl.

A study on chemical stability of PANI-CeMoSi exchangers and its inorganic counterpart were done in various solvent systems and all materials were found to be quite stable in different concentrations of mineral acids such as 10.0M HNO_3 , 5.0M H_2SO_4 and 10.0M HCl, 0.05M solutions of bases and organic solvents like ethanol, acetone, CCl_4 , DMSO, DMF etc.

Specific surface area determined through Sear's method shows substantially high surface area of 81.2 and 85.5 m^2/g for CeMoSi and PANI-CeMoSi respectively.

Ion exchange capacity of the material was also affected by heating. On heating at different temperatures for 3 Hr, the ion exchange capacity and physical appearance of the dried sample were changed as the temperature increased (**Figure 1**). The material CeMoSi was found thermally stable up to 500°C as the sample retained 75% of its initial ion exchange capacity without any change in its color and appearance whereas in the case of its PANI-CeMoSi composite, only 49% of initial capacity was retained up to 500°C even though its ion exchange capacity was higher than those of CeMoSi. Decrease in ion exchange capacity with increasing temperature is attributed to the condensation of structural hydroxyl groups.

pH titration studies of CeMoSi and PANI-CeMoSi using NaCl/NaOH, KCl/KOH systems shows (**Figure 2**) nearly bifunctional behaviour indicating the presence of two ionizing group. Both exchangers appear to be strong cation exchangers as indicated by a low pH (~ 2-3) of the solution when no OH⁻ ion was added. On the other hand, weak acidic groups are undissociated and thus inactive at this low pH. As the volume of NaOH added to the system is increased, more OH⁻ ions are consumed suggesting in the increase of the rate of ion exchange in basic medium due to the removal of H^+ in the external solution. The rate of $\text{H}^+ - \text{K}^+$ exchange was found faster than those of $\text{H}^+ - \text{Na}^+$ exchanges.

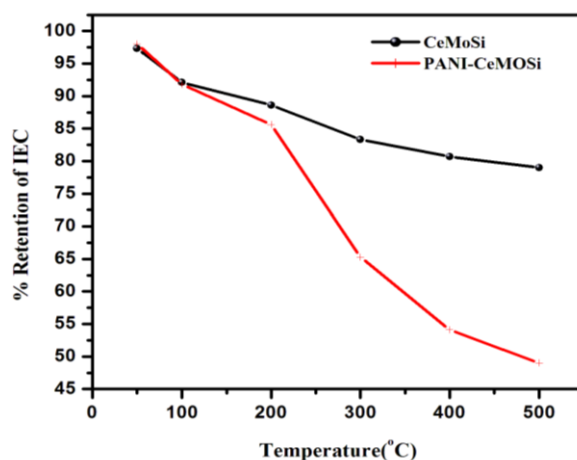


Figure 1 Effect of temperature on IEC

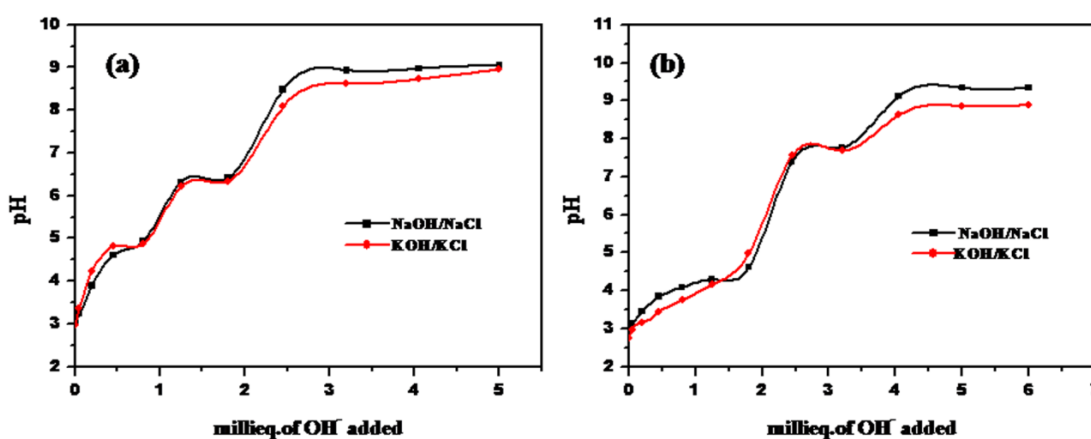


Figure 2 pH titration curves of (a) CeMoSi and (b) PANI-CeMoSi

Figure 3a and 3b depicts the FTIR spectra of CeMoSi and PANI- CeMoSi. For CeMoSi a broad band in the region $\sim 3382\text{ cm}^{-1}$ which is assigned to symmetric and asymmetric $-\text{OH}$ stretching, while the band at $\sim 1610\text{ cm}^{-1}$ is attributed to H-O-H bending vibrations of water molecules. The band observed at $\sim 1097\text{ cm}^{-1}$ may be due to M-O-H bending vibrations and this indicates the presence of structural hydroxyl protons in CeMoSi. Bands at $\sim 556\text{ cm}^{-1}$ and $\sim 817\text{ cm}^{-1}$ are contributed by Ce-O [9] and Mo-O. In addition to that, the band recorded at and $\sim 917\text{ cm}^{-1}$ corresponded to Si-O-Si stretching vibration and also due to OH-OH interaction. In the FTIR of PANI- CeMoSi a peak observed at 1238 cm^{-1} was attributed to stretching vibration of C-H in the benzenoid ring and that observed at 1469 cm^{-1} was attributed to C=N stretching vibration of quinoid ring. Also the band at 1564 cm^{-1} may be due to the presence of benzenic-quinonic N atom [10]. This may indicate that PANI- CeMoSi containing considerable amount of aniline. Other peaks observed are characteristics of inorganic counter part.

Figure 4a shows the Tg-DTG curves of PANI-CeMoSi. It decomposes mainly in three stages: first stage occurs around $50\text{--}200^\circ\text{C}$ of mass loss 18% is due to loss of external water molecules. Second stage of weight loss observed around $250\text{--}650^\circ\text{C}$ may be characteristics of condensation and decomposition of organic part of the material. Abrupt weight loss of above 700°C is the third stage which occurs due to formation of metal oxides.

The XRD pattern of PANI-CeMoSi (Figure 4b) composite also shows number of sharp and strong peaks at various 2θ values. Average crystallite size of composite was calculated using Scherrer equation and are found to be 2.46 nm .

The SEM image of PANI-CeMoSi (Figure 4c) composed of intense and loose aggregation of small granular particles and pores with non linear channels. This granular morphology after composite formation was probably due to the reason that one CeMoSi interacted with more than one nitrogen atom in the PANI chain. The granular nature makes them suitable for column operations and porous nature increases the surface area, which increases rate of ion exchange and catalytic activity. Also the presence of porosity in the material may results in transportation of ions

across the channel which in turn increases the exchange of ions between the crystal and external solutions.

In the EDX spectra (**Figure 4d**) of PANI-CeMoSi presence of Ce, Mo, Si and O elements of CeMoSi and in addition to that peak corresponding to C and N also present which confirms the composite formation.

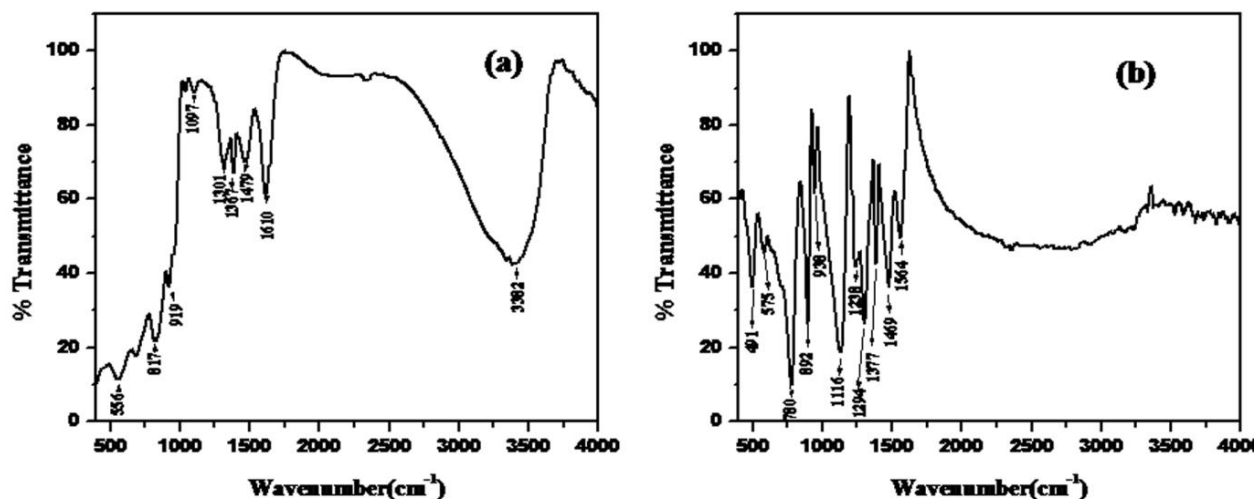


Figure 3 FTIR of (a) CeMoSi and (b) PANI-CeMoSi

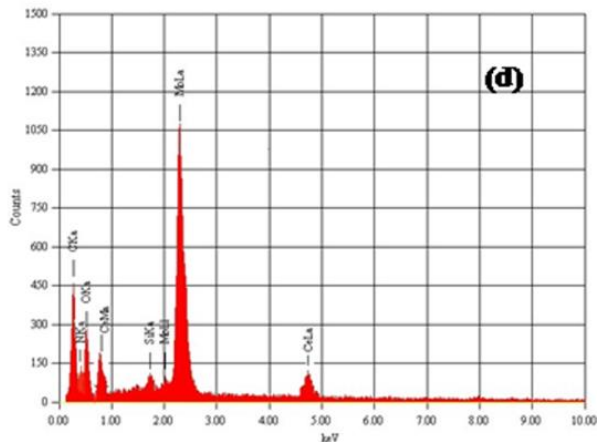
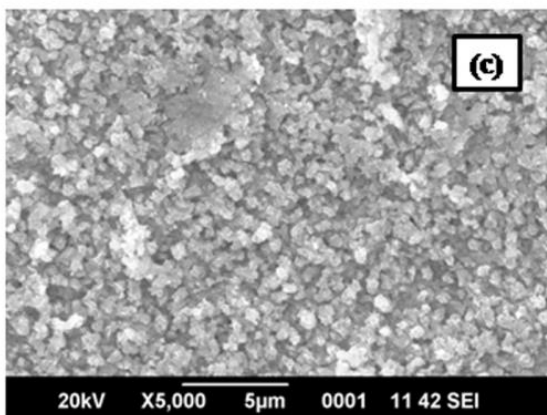
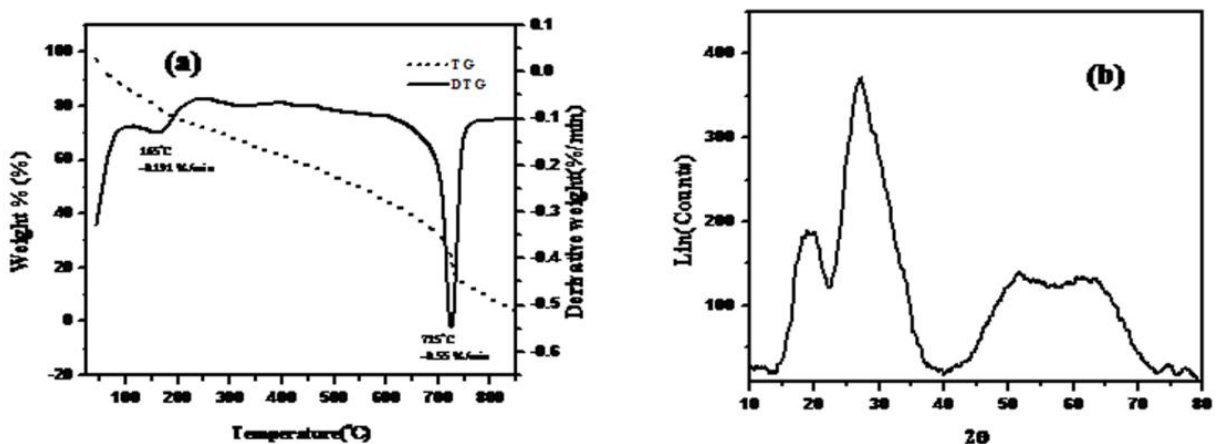


Figure 4 (a) TG-DTG (b) XRD (c) SEM (d) EDX of PANI-CeMoSi

A tentative mixed oxide formula suggested for PANI-CeMoSi composite based on IR, TGA and EDS data was $[(\text{CeO}_2).(\text{SiO}_2).(\text{MoO}_3)_4.-(\text{C}_6\text{H}_5\text{NH})_5. \cdot n\text{H}_2\text{O}]$, where n is the number of water molecules and was found to be ~ 5 determined using Alberti-Torroca formula.

Distribution studies of different metal ions on PANI-CeMoSi (**Table 2**) shows that the exchanger has very high affinity towards Bi^{3+} ion in comparison to other metal ions studied and no selectivity is shown towards Ca^{2+} . The selectivity was found to be in the order $\text{Bi}^{3+} > \text{Hg}^{2+} > \text{Th}^{4+} > \text{Mn}^{2+} > \text{Al}^{3+} > \text{Pb}^{2+} > \text{Cu}^{2+} > \text{Ni}^{2+} > \text{Zn}^{2+} > \text{Co}^{2+} > \text{Cd}^{2+} > \text{Mg}^{2+} > \text{Ca}^{2+}$.

Table 2 Distribution coefficients in water and other Electrolytes for PANI-CeMoSi

Cations	Distribution coefficients (K_d)						
	DMW	HNO ₃			NH ₄ NO ₃		
		0.001M	0.01M	0.1M	0.001M	0.01M	0.1M
Bi^{3+}	432.4	345.23	303.45	297.01	402.76	368.24	311.24
Th^{4+}	292.16	254.13	162.34	102.00	272.66	201.11	154.66
Cd^{2+}	21.32	15.42	9.74	NS	17.23	10.45	3.45
Pb^{2+}	121.72	112.12	82.54	58.47	118.45	96.33	62.00
Co^{2+}	27.4	20.11	15.77	8.33	25.33	17.42	10.09
Zn^{2+}	38.44	22.31	17.42	11.22	35.01	27.44	20.98
Mg^{2+}	17.28	10.67	5.52	NS	14.32	10.58	8.86
Mn^{2+}	276.92	234.59	189.05	143.23	262.44	193.23	101.22
Cu^{2+}	102.56	87.42	54.55	37.42	95.30	64.74	58.87
Ni^{2+}	40.6	35.47	32.05	26.22	38.28	23.44	20.12
Al^{3+}	270.84	189.65	130.22	121.72	215.22	201.44	160.25
Hg^{2+}	411.4	362.02	326.73	292.01	394.01	372.22	301.16
Ca^{2+}	NS	NS	NS	NS	NS	NS	NS

NS: No Sorption

Analytical applications

Quantitative separation of metal ions from synthetic binary mixtures

The difference in the K_d values of metal ions indicated the separation possibilities of certain metal ions of analytical importance and which has been demonstrated by achieving some difficult binary separations (**Table 3**) such as Hg^{2+} - Mg^{2+} and Bi^{3+} - Cd^{2+} on PANI-CeMoSi. The separation was based on sequential elution of ions through the column using different eluting agents such as NH_4NO_3 and HNO_3 . The sequential elution of ions from the column depends up-on the stability of metal-eluting ligand. The weakly retained metal ions eluted first and the stronger one at the last. The separation was quite sharp and recovery was quantitative and reproducible.

Table 3 Binary separations of metal ions on PANI-CeMoSi

Separations achieved	$\alpha = K_{d1}/K_{d2}$	Eluent	Metal ion (mg)		Efficiency (%)
			Loaded	Eluted	
Bi^{3+} - Mg^{2+}	25.02	0. 1M NH_4NO_3	5.21	5.19	99.61
		0.5M HNO_3 +0.1M NH_4NO_3	1.33	1.31	98.45
Hg^{2+} - Mg^{2+}	23.81	0. 1M NH_4NO_3	5.01	4.98	99.40
		0.5M HNO_3 +0.1M NH_4NO_3	1.34	1.32	98.37
Bi^{3+} - Cd^{2+}	20.28	0. 1M NH_4NO_3	5.18	4.92	94.94
		1.0M HNO_3 +0.1M NH_4NO_3	2.76	2.73	98.37
Hg^{2+} - Cd^{2+}	19.29	0. 04M NH_4NO_3	5.02	5.00	99.67
		1.0M HNO_3 +0.1M NH_4NO_3	2.77	2.72	96.23
Th^{4+} - Mg^{2+}	16.93	0. 05M NH_4NO_3	5.31	5.28	99.49
		0.5M HNO_3 +0.1M NH_4NO_3	1.34	1.29	96.98
Al^{3+} - Cd^{2+}	12.73	0.1M NH_4NO_3	3.12	3.05	97.75
		1.0M HNO_3 +0.1M NH_4NO_3	2.76	2.72	98.59

Quantitative separation of metal ions from industrial wastewater

The specific selectivity of PANI-CeMoSi composite exchanger towards bismuth, mercury and thorium was the promising feature of the materials, where mercury and thorium are key environmental pollutant. In order to demonstrate the practical utility of the materials, quantitative separations of mercury from wood industry effluents and thorium from phosphate fertilizer plant have been achieved on the materials using column method (**Table 4**). Results tabulated showed that the polyaniline composite is very efficient in removing these metal ions from industrial wastewater.

Table 4 Quantitative separation of metal ions from industrial wastewater effluents on PANI-CeMoSi column

Sample	Metal ions	Conc. of metal ion in water sample loaded (mg)	Estimated conc. of metal ion eluted(mg)	Removal %
Wood industry effluents				
Sample-1	Hg ²⁺	3.42	2.99	87.43
Sample-2		5.13	4.89	95.42
Sample-3		4.21	4.10	97.38
Phosphate fertilizer plant				
Sample-1	Th ⁴⁺	5.62	5.21	92.70
Sample-2		2.87	2.46	85.71
Sample-3		4.54	4.32	95.15

PANI-CeMoSi as catalyst for dye degradation

The analytical application of the material was studied by removing the hazardous organic pollutants in waste water by degradation of methylene blue and crystal violet on the ion exchange surfaces. The drastic decrease in the intensity of corresponding absorbance peaks (**Figure 5**) of MB and CV reveals the effectiveness of the exchanger for removing these dyes from aqueous solution. PANI-CeMoSi composite exchanger removes 98.3 and 96.4 percentage of MB and CV dye within 2 Hrs under stationary conditions.

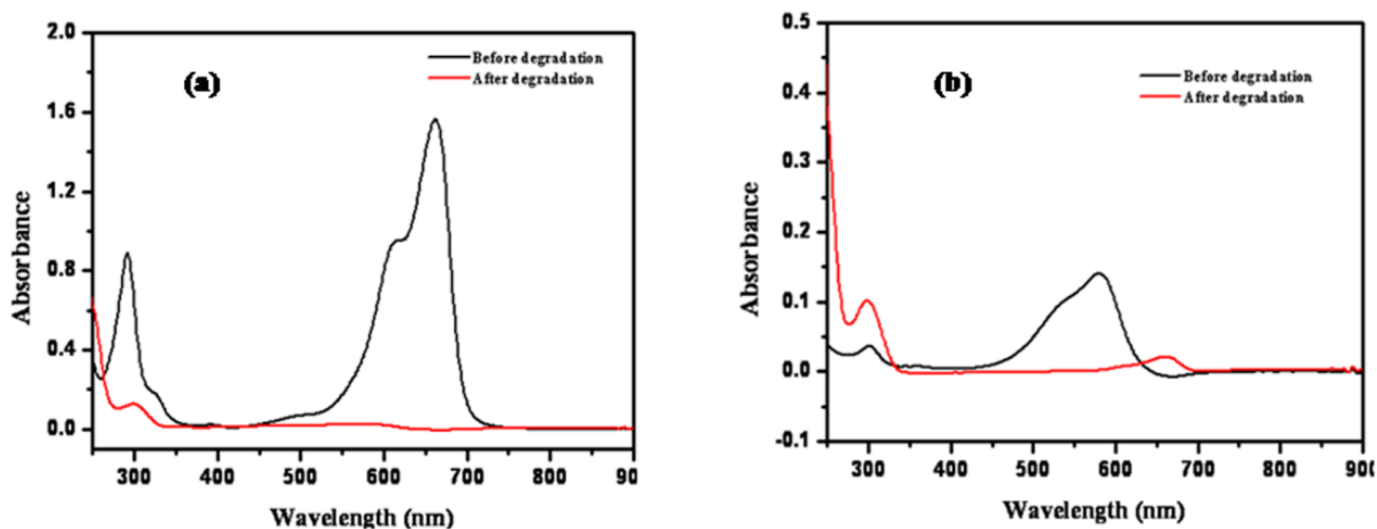


Figure 5 UV-Visible Spectra of degradation of (a) MB and (b) CV on PANI-CeMoSi

The removed ion exchangers from the dye solutions after equilibrium were collected and dried. Absorbance recorded for these samples show close agreement with that of pure samples of exchangers (**Figure 6**). This shows that no dye molecules were present on the surface of the catalysts. Hence the decolourisation of the dye solution is certainly due to the degradation of the dye molecules by the exchangers. These results indicate the strong catalytic activity of the new exchangers towards MB and CV dye degradation.

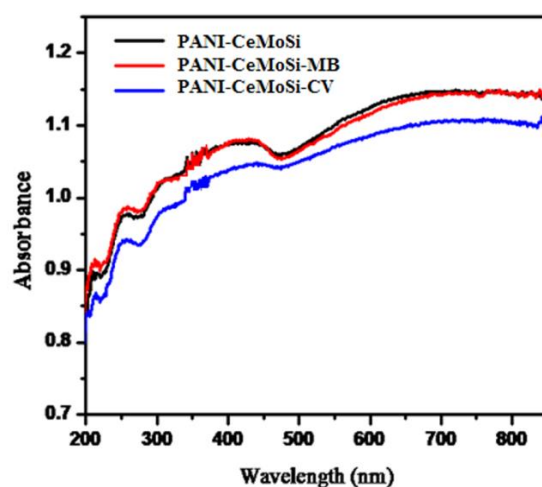


Figure 6 UV-DRS of PANI-CeMoSi before and after treatment with MB and CV

Conclusion

The present study revealed the ability of the ion exchange material to remove heavy metal ions and dyes from aqueous solution. The results of the study show that the material is a good cation exchanger with synergic properties. Almost 90% of Hg^{2+} and Th^{4+} , toxic environmental pollutant can be removed efficiently by using the exchanger. Also UV-Visible DRS data shows that the ion exchange material is an efficient catalyst for the degradation of MB and CV dyes and above 95 % of these dyes can be removed within 2 Hrs of treatment with exchanger. Thus entire studies showed the potential applicability of material towards environmental remediation.

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