Removal of Toxic Metal Ions by Using Composite Cation-Exchange Material

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ABSTRACT: Polyanilinezirconium(IV) arsenate composite cation exchange material was synthesized under different experimental conditions by the incorporation of polyaniline into the matrices of inorganic precipitate (zirconium(IV)arsenate). The experimental parameters such as concentration, mixing volume ratio, and pH were established for the synthesis of the material. Ion-exchange material that was synthesized at pH 1.0 showed an ion exchange capacity of 1.33 meq g^{-1} for Na$^+$ ions. The composite material exhibits improved ion-exchange capacity along with chemical and thermal stability. The exchanger was characterized based on FTIR, TGA, XRD, and SEM analysis. The X-ray diffraction study shows semi-crystal-line nature of the material. The distribution coefficient studies (K_d) of metal ions on the material were performed in diverse solvent systems. Based on K_d values the material was found to be selective for Pb(II) and Hg(II) ions. Some analytically important binary separations of metal ions in synthetic mixtures viz. Ba^{2+}-Pb^{2+}, Pb^{2+}-Ni^{2+}, Cd^{2+}-Hg^{2+}, Ni^{2+}-Hg^{2+}, Zn^{2+}-Pb^{2+}, Ca^{2+}-Bi^{3+}, Al^{3+}-Hg^{2+}, and Ca^{2+}-Pb^{2+} were achieved on the columns of polyanilinezirconium(IV) arsenate cation exchanger. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: composite adsorbent; synthesis; characterization and analytical applications

INTRODUCTION

Nowadays one of the most important issue is related to pollution and public health. The presence of heavy metals in the environment is of great concern because of their toxicological and physiological effects. These metals if present above specific concentration can be a serious health hazard leading to many disorders in the body of human beings and animals. Wastewater containing heavy metals as untreated or partially treated by-products of different industries enter rivers and the sea, and contaminate land. Among heavy metals, mercury, lead, and cadmium seem to be the most toxic. It is therefore very important to treat such water to remove these metal ions before it is supplied for human use. Mercury and Lead are responsible for causing poisoning through water, food, and smoking. Its vapor on inhalation enters the brain through the blood stream and causes severe damage to the central nervous system. Organic mercurial CH$_3$Hg$^+$ is the most toxic substance which can pass through the placental barrier and enter the fetal tissues. The intake of Hg(II) and Pb(II) through water affects nervous system causes nephropathy, sluggishness, and loss of skin color.[1]

Among ion-exchangers composite ion-exchange materials have been used for solving environmental and industrial problems. A number of organic and inorganic ion-exchange materials have been synthesized. The limitations existing with organic ion exchangers are of low mechanical strength and degradation of ion-exchange properties at higher temperature and radiation conditions, whereas in case of inorganic ion-exchanger it is very difficult to obtain granular form of material. Hence, to overcome all these drawbacks, hybrid ion-exchanger hanging with conjugates properties of polymer and intrinsic properties of inorganic exchanger have been introduced.[2–9]

To overcome all these drawbacks much attention has been paid to synthesized organic–inorganic composite ion-exchangers because of its excellent ion-exchange capacity, selectivity, chemical and thermal stability. Synthesis of composite ion-exchanger with controlled functionality and hydrophobicity open new area for organo-metallic chemists. The composite materials have been preferred due to its intercalation properties. They can be use as ion-exchanger,[10–13] ion selective electrode,[14–16] catalyst,[17] sorbent,[18] and in the host–guest chemistry[19,20] for separation of radioactive isotopes. They also find a large number of applications in pollution control and waste water treatment.[21] Zirconium based hybrid ion-exchanger

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have drawn much attention owing to its excellent ion-exchange capacity. The present work describes the synthesis, characterization, ion-exchange behavior, and analytical applications for the removal of heavy metal ions of analytical interest.

MATERIALS AND METHODS

Instruments

A digital pH meter Elico (EL-10, India) was used for pH measurements. Infrared (IR) spectra were recorded on a Fourier Transform-IR Spectrometer (FTIR) from Perkin Elmer (1730, USA) using KBr disc method. An X' Pert PRO analytical diffractometer (PW-3040/60 Netherlands, Holland with Cu Kα radiation λ = 1.5418 Å) was used for X-ray diffraction (XRD) measurement. Thermogravimetric analysis/differential thermal analysis (TGA/DTA) analysis by DTG—60 H; C305743 00134 (Schimadzu), scanning electron microscope (SEM; LEO, 435 VF) instrument was used for SEM images of the material at different magnifications. CHNO analysis was carried out on Carlo Erba EA1108 (Milan, Italy) elemental analyzer in sophisticated analytical instrument facility (Chandigarh, India). A temperature controlled shaker (MSW-275, India) was used for shaking. Muffle furnace (Narang Scientific works—India) was used for heating samples at different temperatures.

Reagents and solutions

All the reagents used were of analytical grade. Aniline, potassium persulfate, and zirconium(IV)oxychloride were procured from Loba Cheme (India). Solutions of zirconium(IV)oxychloride (0.2M) and sodium arsenate (0.2M) were prepared in demineralized water while solutions of 10% aniline (v/v) and potassium persulphate (0.1 mol L^{-1}) were prepared in HCl solution (1 M).

Synthesis of polyaniline

The polymerization of aniline was initiated by the addition of oxidizing agent, i.e., potassium persul-phate in 1 : 1 ratio under the controlled condition with continuous stirring for 2 h below 10°C[22] a green gel of polyaniline was obtained.

Synthesis of inorganic precipitate Zr(IV)arsenate

The method of preparation of the inorganic precipitate (Zr(IV)arsenate) was very similar to that of Alberti and Constantino with slight modification[23] by adding a aqueous solution of zirconium(IV)oxychloride into a solution of sodium arsenate in different molarities at the flow rate of 2.0 mL min^{-1}. Continuous stirring was done during mixing using a magnetic stirrer, white gel type slurries were obtained. The resulting white precipitate so formed was allowed to stand for 24 h in the mother liquor for digestion.
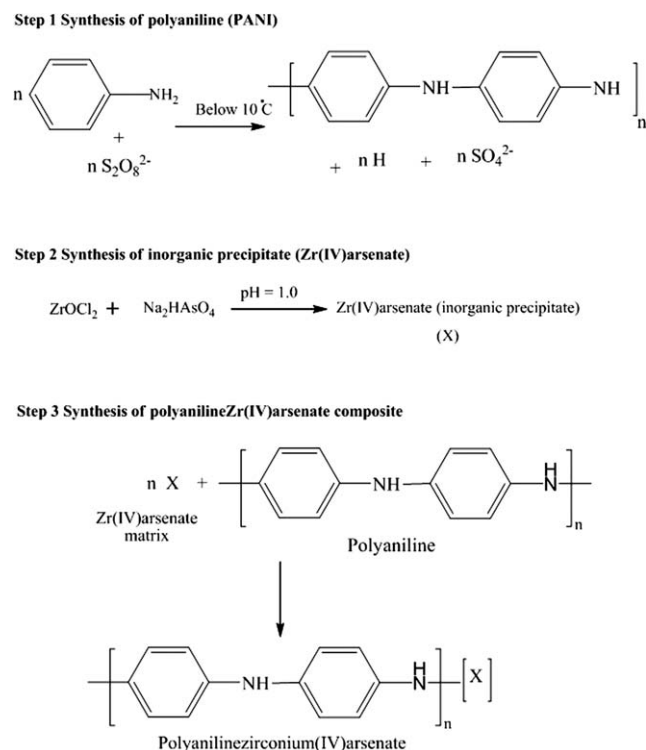
Synthesis of polyanilinezirconium(IV)arsenate composite

The composite cation-exchanger was prepared by the mixing of polyaniline gel into the inorganic precipitate of zirconium(IV) arsenate at the flow rate of 2.0 mL min^{-1} with continues stirring, the resultant mixture turned slowly into green slurries. The resultant slurries were kept for 24 h at room temperature. Now, the polyaniline-based composite gels were filtered off, subsequently washed with demineralized water to remove excess acid and any adhering trace of ammonium persulphate. The washed gel was dried at 50°C ± 2°C in an oven. The dried material was immersed in demineralized water to obtain small granules. They were converted to H$^+$ by treating with 1M nitric acid with occasional shaking. The excess acid was removed after several washings with demineralized water. The material was finally dried at 50°C ± 2°C. Thus a number of samples of "polyanilinezirconium(IV)arsenate" composite were prepared under different condition of pH range (1–1.5) and mixing ratio of reactants (Table I). Based on the improved ion-exchange capacity along with percentage yield, sample S-5 was selected for further studies (Table I). The

TABLE I
Conditions for the Synthesis of Different Samples of Polyaniline Zirconium(IV) Arsenate

S. no.	(A)	(B)	Mixing ratio (v/v)	Mixing volume ratios (v/v) 0.1 M K$_2$S$_2$O$_8$	10% Aniline	Temperature	pH	IEC for Na$^+$ ions	Yield (g)
S-1	0.10	0.10	1 : 1	1	1	25°C ± 2°C	1.0	0.27	1.82
S-2	0.20	0.20	1 : 1	1	1	25°C ± 2°C	1.0	0.73	3.02
S-3	0.20	0.20	1 : 1	1	1	25°C ± 2°C	0.76	0.0	No ppt
S-4	0.20	0.20	1 : 1	1	1	25°C ± 2°C	1.5	1.0	2.20
S-5	0.20	0.20	1 : 2	1	1	25°C ± 2°C	1.0	1.33	4.41

A, zirconium (IV)oxychloride (Mol L-1); B, Sodium arsenate (Mol L-1); IEC, ion-exchange capacity (meq g^{-1}).

Figure 1 Proposed scheme of polyanilinezirconium(IV) arsenate composite material.

proposed structure of polyanilinezirconium(IV) arsenate is shown in Figure 1.

Ion exchange capacity

To determine ion exchange capacity, 1 g 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 $\sim$ 1.5 cm in height. Alkali (Li^+, Na^+, K^+) and alkaline earth metal (Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}) nitrates solutions ($0.1M$) were used to elute H^+ ions from the exchanger. The flow rate of column is maintained at 2.0 mL min^{-1}. The effluent collected was titrated against standard solution of $0.1M$ sodium hydroxide using phenolphthalein as indicator.

pH titration

To determine the nature of the ionogenic group of the ion-exchanger pH titrations were performed in various systems LiCl-LiOH, NaCl-NaOH, and KCl-KOH. Top and Pepper[24] method was used for pH titration study. In this method 0.5 g of the exchanger (H^+ form) was taken in each of several 250-mL conical flasks which is followed by the addition of equimolar solution of alkali metal ($0.1M$). The final volume was adjusted to 50 mL to maintain the ionic strength.

Chemical dissolution

About 250 mg portion of the composite cation exchanger (H^+ form) was treated with 20 mL of different common acids (HCl, HNO_3, H_2SO_4, $HClO_4$), bases (NaOH, KOH, NH_4OH), sodium nitrate solution, and few organic solvents (DMSO, DMF, acetone, THF, $CHCl_3$, CCl_4) and also with demineralized water for 24 h with occasional shaking.

Chemical composition

Nearly 0.50 g of the sample was dissolved in 10 mL of hot concentrated H_2SO_4 solution. The sample solution was diluted to 50 mL. The amount of zirconium and arsenate were determined by standard spectrophotometric methods.[25] Carbon, hydrogen, and nitrogen contents of the material were determined by elemental analysis.

Effect of eluent concentration

Fixed volume (250 mL) of varied concentrations of sodium nitrate solution was used for complete elution of H^+ ions from the column. The collected effluent was titrated against standard solution of sodium hydroxide to determine the concentration of H^+ ions.

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.

Thermal stability

The effect of heating temperature on the ion-exchange capacity of the material was studied by heating the composite from 50 to 600°C for 1 h at each temperature. The decomposition for each heated sample was determined according to the recommended procedure.[26]

Selectivity (sorption) studies

The distribution coefficient (K_d) of metal ions were 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. Various portions of (5 mg each) of composite cation exchanger (H^+ form) were taken in Erlenmeyer flasks and titrated with 40 mL of different metal nitrate solution in the required medium and subsequently shaken for 6 h in temperature-controlled shaker at 25°C $\pm$ 2°C to attain the

| Exchanging ions	Ionic radii (Å)	Hydrated ionic radii (Å)	IEC
Li^+	0.68	3.40	1.25
Na^+	0.97	2.76	1.33
K^+	1.33	2.32	1.51
Mg^{2+}	0.78	7.00	1.22
Sr^{2+}	1.27	6.30	1.77
Ca^{2+}	1.43	5.90	1.95
Ba^{2+}	1.43	5.90	2.23

IEC, ion-exchange capacity (meq g^{-1}).

equilibrium. The metal ion concentration before and after the equilibrium was determined by EDTA titration. The distribution coefficients were calculated using the equation:

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

where I is the 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. V is the volume of the solution (mL), and M is the amount of ion exchanger taken (g). 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$^+$	+	M^{2+}	⇌	R$_2$-M	+	2 H$^+$
Exchanger phase		Solution phase		Exchanger phase		Solution phase
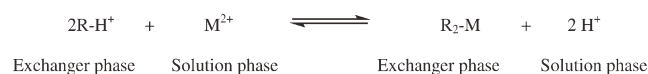
where R = polyanilinezirconium(IV)arsenate.

Quantitative separations of metal ions in synthetic binary mixtures

Quantitative separations of some selective metal ions were achieved on column of polyanilinezirconium

| Temperature (°C)	Color	IEC for Na$^+$ ions	Retention of IEC (%)
50	Black	1.33	100
100	Black	1.28	96
200	Black	1.26	94
300	Black	1.00	75
400	Brown	0.98	73
500	Grey white	0.82	62
600	White	0.77	58

IEC, ion-exchange capacity (meq g^{-1}).
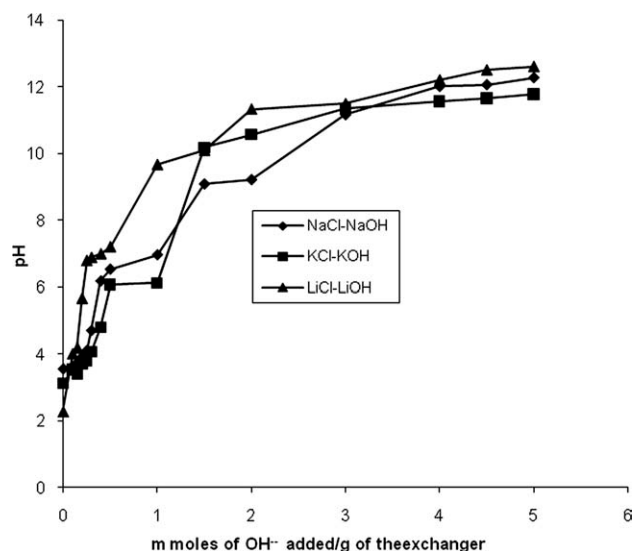
Figure 2 pH-titration curves of polyanilinezirconium(IV)arsenate with various alkali metal hydroxides.

(IV)arsenate. One gram of exchanger (H^+ form) was packed in a glass column (0.6 cm, internal diameter) with a glass wool support at the end. The column was washed thoroughly with demineralized 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 2.0 mL min^{-1} till the solution level was just above the surface of the composite material. The process was repeated two or three times to ensure the complete adsorption of metal ions on the bead. The separation of metal ions achieved by collecting the effluent in 10 mL fractions and titrating against the standard solution of 0.01 M di-sodium salt of EDTA.
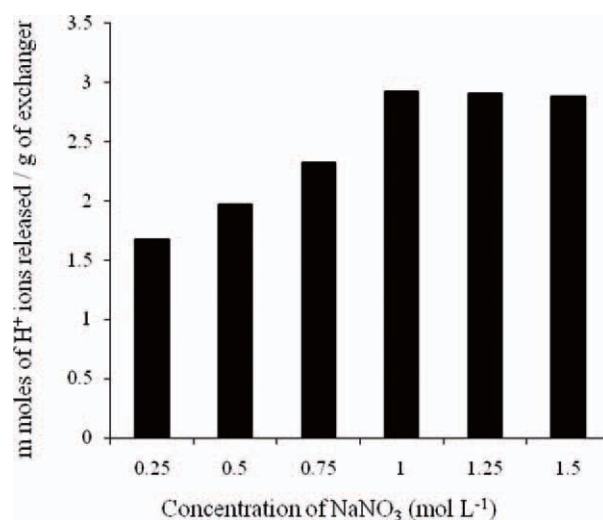
**Figure 3** Effect of eluent concentration on ion-exchange capacity of polyaniline zirconium(IV)arsenate.
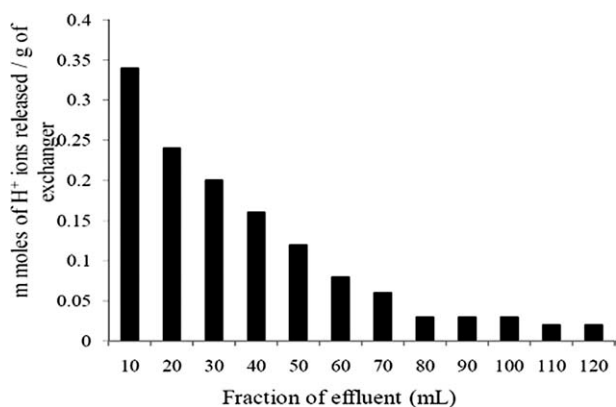
Figure 4 Elution behavior of polyanilinezirconium(IV) arsenate.

Selective separation of metal ion from a synthetic mixture

Selective separation of Pb^{2+} and Hg^{2+} from the synthetic mixtures containing (Ca^{2+}, Sr^2, Ni^{2+}, Pb^{2+}) and (Ca^{2+}, Ni^{2+}, Ba^{2+} Zn^{2+}, Hg^{2+}) was achieved on polyanilinezirconium(IV)arsenate columns. The amount of the Pb^{2+} and Hg^{2+} ions in the synthetic mixture was varied keeping amount of the other metal ions constant.

RESULTS AND DISCUSSION

It was evident from Table I, the volume ratio of reactants and pH of the reaction mixture affect the appearance and ion-exchange capacity of the material. Results show that at higher pH the ion-exchange capacity of composite material decreases. The optimum pH for the synthesis of polyanilinezirconium(IV)arsenate was found to be 1. It is informed in Table I that the cation exchange characteristic of the exchanger is due to the presence of ionogenic group of the inorganic part of the material to which counter ions are attached. The affinity for alkali metals follow the sequence (Table II) $K^+> Na^+> Li^+$
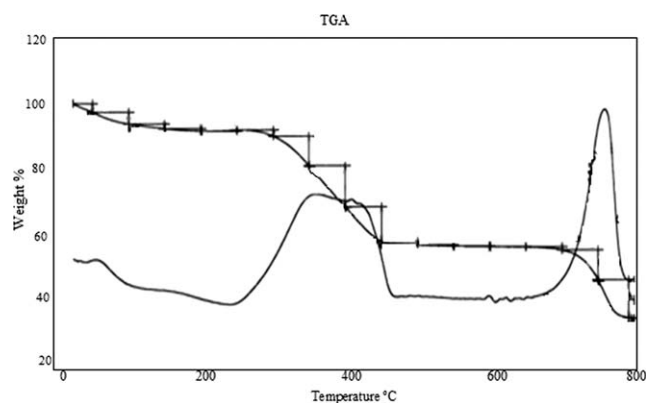

Figure 5 TGA-DTG curves of polyanilinezirconium(IV) arsenate.

while for alkaline earth metals $Ba^{2+}> Ca^{2+}> Sr^{2+}> Mg^{2+}$. It is in accordance with decreasing order of the hydrated ionic radii.[27] The ions with smaller hydrated ionic radii easily enter the pore of the exchanger resulting in higher sorption capacity.[28]

Ion-exchange capacity was also affected by heating the material. Decrease of ion-exchange capacity was observed by increasing the heating temperatures. It was found that the material retains 96% ion-exchange capacity up to 100°C, 94% at 200°C, 75% up to 300°C, and 58% at 600°C (Table III). The pH titration curve (Fig. 2) obtained under equilibrium conditions for each of the LiOH-LiCl, NaOH-NaCl, and KOH-KCl systems shows two inflection points which indicate the bifunctional strong cation exchange behavior. Sequence of uptake as evident from pH titrations curve has been found to be below pH 7: $K^+> Na^+> Li^+$ and between pH 7–11; $Na^+> K^+> Li^+$ (Fig. 2).

This can also be concluded that as the concentration of eluent is increased the elution of H^+ also become faster (Fig. 3). The elution behavior of the material depicts that the exchange is quite fast at the beginning and maximum H^+ ions are eluted out (Fig. 4) in the first 10 mL of the effluent. All these observations conclude the better column efficiency.

The material is stable in some organic solvents (DMSO, DMF, CH_3CN, m-cresol, THF), acids (HCl,
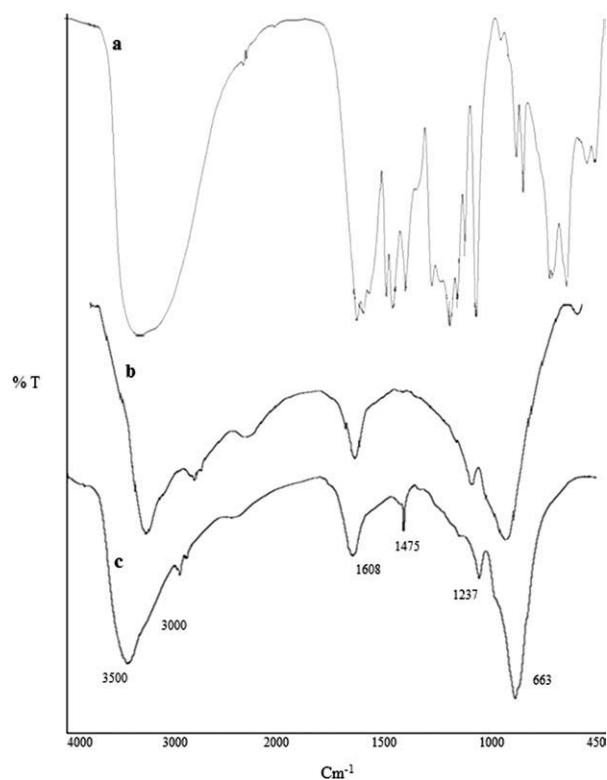
Figure 6 (a, b, c) FTIR spectra of polyaniline (a), zirconium(IV)arsenate (b) and polyaniline zirconium(IV)arsenate composite (c).
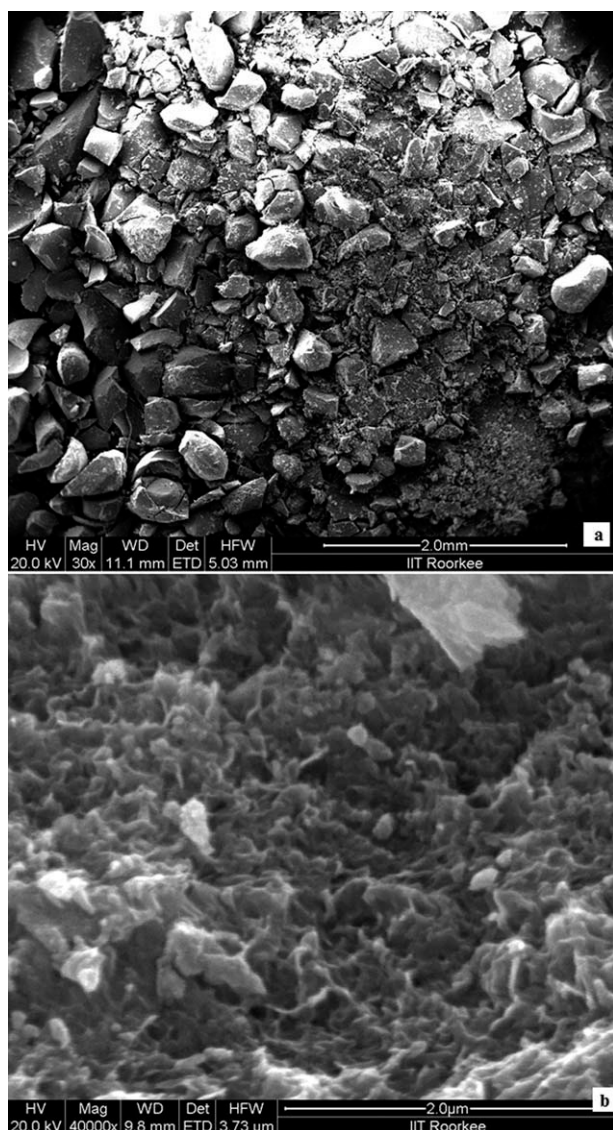
Figure 7 (a, b) SEM images of zirconium(IV)arsenate (a), polyaniline zirconium(IV) arsenate (b).

HNO_3, H_2SO_4) and bases, (NaOH, KOH) up to 2M, while it was found to be miscible in CCl_4. Thus exchanger is chemically resistant to most of the solvents and can be successfully used with diverse solvents in column operation. The material is reasonably stable in 2M solution of HCl, HNO_3, $HClO_4$, and H_2SO_4. It is slightly soluble in common alkali solutions while completely insoluble in organic solvents (DMSO, DMF, Acetone, THF, $CHCl_3$, and CCl_4). The chemical analysis of polyanilinezirconium(IV)arsenate revealed that the amount of different elements in the composite material was found to be: Zr(28.20%); As(56.49%); C (40.81%), H (5.41%), N (13.15%).

The thermogravimetric and derivative thermogravimetric curves (Fig. 5) show 8% weight loss of mass up to 100°C which is due to the removal of external water molecules.[29] Continuous weight loss (about

24%) in the region 300 to 425°C is observed due to elimination of interstitial water molecules along with the decomposition of organic constituents of the material. Further weight loss from 700 to 780°C is due to decomposition of inorganic part of the exchanger leading to the formation of metal oxides.

The FTIR spectra of polyanilinezirconium(IV) arsenate showed [Fig. 6(c)] a broad band in the region 3500–3200 cm^{-1} confirming the presence of —OH group,[30] in addition to stretching vibration of —NH— that indicates the presence of secondary amino group.[31] The peaks between $\sim$ 1475 and $\sim$ 1237 cm^{-1} indicate the presence of considerable amount of polyaniline in the material while the bands with a maximum at 1384, 1300, 1123, and 663 cm^{-1} are attributed to the in-plane bending of the —CH bands.[32] A sharp band at $\sim$ 1608 cm^{-1} is attributed to aquo H—O—H bending and C—C in plane stretching. A sharp peak at 850 cm^{-1} can be attributed due to the presence of metal-oxygen stretching vibration. The IR spectra of composite material have been compared with the individual spectra of polyaniline [Fig. 6(a)] and inorganic precipitate (Zr(IV)As) [Fig. 6(b)] and polyanilinezirconium(IV)arsenate [Fig. 6(c)].

Scanning electron micrographs of zirconium(IV) arsenate [Fig. 7(a)] and polyaniline zirconium(IV) arsenate [Fig. 7(b)] indicate the binding of inorganic ion-exchange material with polyaniline. It has been revealed that zirconium(IV)arsenate shows semi-crystalline morphology. After the binding polyaniline with inorganic precipitate, the composite material shows amorphous nature. The typical X-ray diffraction pattern inorganic precipitate [Fig. 8(a)]
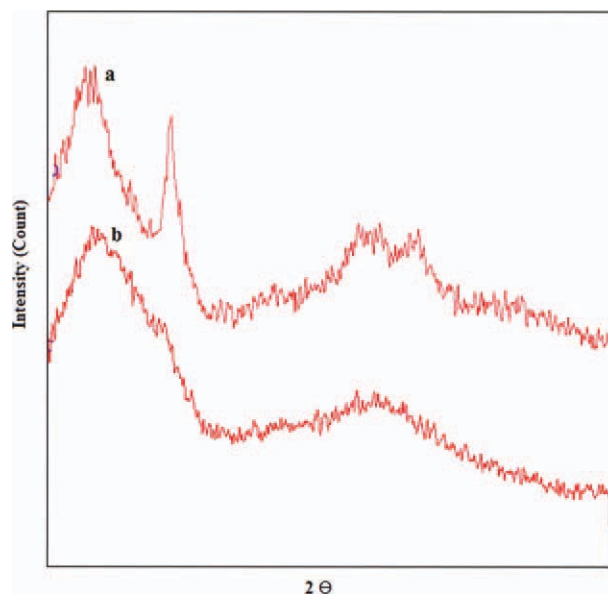
Figure 8 (a, b). X-ray diffraction pattern of zirconium(IV) arsenate (a) polyaniline zirconium(IV)arsenate (b).

TABLE IV
Distribution Coefficients (mL g^{-1}) K_d of Metal Ions on Polyanilinezirconium(IV) Arsenate in Different Solvent Systems

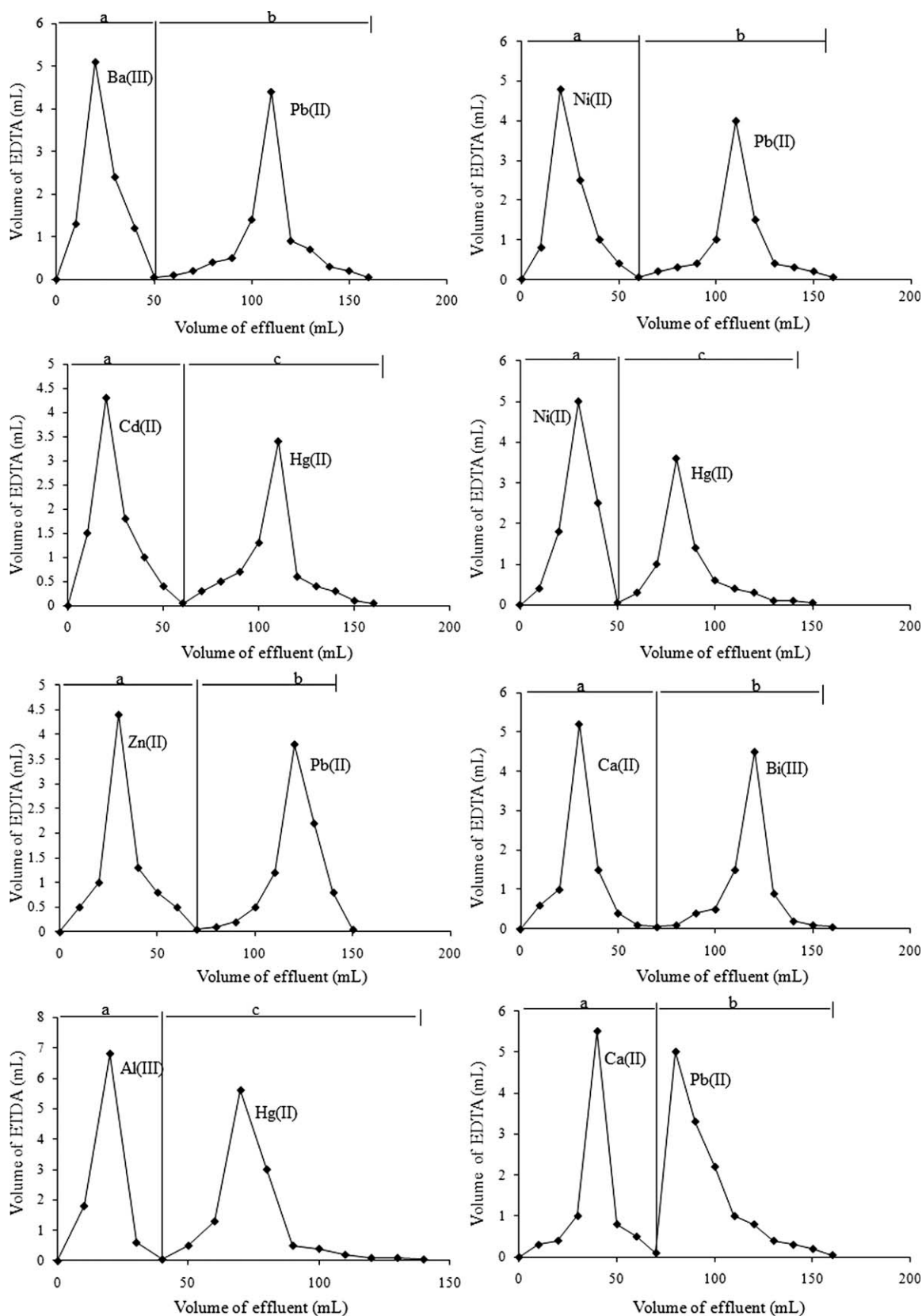| Metal ions	0.1M HClO$_4$	0.01M HClO$_4$	0.001M HClO$_4$	0.1M HNO$_3$	0.01M HNO$_3$	0.001M HNO$_3$	DMSO 0.001M	DMSO 0.1M	DMSO 0.01M
Mg^{2+}	196.29	254.16	370.58	166.66	193.93	375	512	300	333
Ba^{2+}	143.24	239.28	400	259.25	340	676.92	381	275	284
Zn^{2+}	181.25	266.66	400	220	327.62	500	380	250	372
Sr^{2+}	250	260	372.72	230	288.88	458.82	375	308	335
Ca^{2+}	75	200	294.73	270.37	280	343.47	326	233	320
Pb^{2+}	878.26	450	2020	160	684.615	1900	3200	1800	1900
Cd^{2+}	175.67	176.31	472.22	214.81	246.66	375	500	332	422
Cu^{2+}	212.5	333.33	472.22	155.55	203.03	421.73	518	212	382
Ni^{2+}	260	300	308.33	220	257.142	333.33	357	267	283
Hg^{2+}	247.82	950	1450	420	662.05	1340	8100	2667	3800
Al^{3+}	172.72	220	285.71	533	550	566.66	682	241	350
Fe^{3+}	390.90	444.44	450	200	433.33	458.82	533	371	477
Ce^{3+}	100	156.75	618.75	242.25	361.96	546.66	707	478	525
Bi^{3+}	360	613	2275	485.71	654.54	950	4550	467	717

shows weak and strong intensity peaks which confirm semi-crystalline nature while the composite material [Fig. 8(b)] exhibits weak intensity peaks which signify the amorphous nature of the material.

To explore the potentiality of composite cation exchanger in the separation of metal ions, distribution studies were performed in different solvents systems (Table IV). It was observed that K_d values increased with decrease in the concentration of the solvents (HClO$_4$, HNO$_3$, DMSO). Based on K_d values the composite material was found to be highly selective for Pb(II) and Hg(II) which are considered as the major pollutants in the environment. The separation capability of the exchanger has been demonstrated by achieving some important binary in mixture of metal ions viz. Ba^{2+}-Pb^{2+}, Pb^{2+}-Ni^{2+}, Cd^{2+}-Hg^{2+}, Ni^{2+}-Hg^{2+}, Zn^{2+}-Pb^{2+}, Ca^{2+}-Bi^{3+}, Al^{3+}-Hg^{2+}, and Ca^{2+}-Pb^{2+}. Table V

summarizes the salient features of these separations which have been separated based on the distribution coefficient values in different solvent systems. The separations are quite sharp and recovery was quantitative and reproducible. The order of elution and eluents used for some representative binary separations are also illustrated in Figure 9. The sequential elution of the ions through the column depends upon the separation factor. Greater the interaction of the metal ion with the eluent, the more easily it will be eluted from the column. Tables VI and VII shows the results of selective separation of Pb^{2+} and Hg^{2+} from synthetic mixtures of multicomponent metal ions. This establishes that polyanilinezirconium(IV)arsenate cation exchanger can be used for the removal and recovery of Pb^{2+} and Hg^{2+} from the industrial effluents containing other metal ions.

TABLE V
Quantitative Separation of Metal Ions from a Binary Mixture Using Polyanilinezirconium(IV)Arsenate Columns at Room Temperature

S. no.	Metal ions separation	Amount loaded (mg)	Amount found (mg)	% Recovery	Volume of eluent used (mL)	Eluent used
1	Ba^{2+}	13.73	13.11	95.5	50	0.001M DMSO
	Pb^{2+}	20.72	18.85	91.0	100	0.1M HNO$_3$
2	Ni^{2+}	5.86	5.72	97.5	60	0.001M DMSO
	Pb^{2+}	20.72	17.61	85	90	0.1M HNO$_3$
3	Cd^{2+}	11.24	10.17	90.5	60	0.001M DMSO
	Hg^{2+}	20.05	15.24	76	100	0.1M HClO$_4$
4	Ni^{2+}	5.86	5.72	97.5	50	0.001M DMSO
	Hg^{2+}	20.05	16.74	83.5	100	0.1M HClO$_4$
5	Zn^{2+}	6.53	5.58	85.5	70	0.001M DMSO
	Pb^{2+}	20.72	19.58	94.5	80	0.1M HNO$_3$
6	Ca^{2+}	4.01	3.54	88.5	70	0.001M DMSO
	Bi^{3+}	20.69	17.07	82.49	90	0.1M HClO$_4$
7	Al^{3+}	2.69	2.49	92.5	40	0.001M DMSO
	Hg^{2+}	20.05	18.15	90.5	100	0.1M HClO$_4$
8	Ca^{2+}	4.01	3.42	85.5	80	0.001M DMSO
	Pb^{2+}	20.72	17.09	82.5	90	0.1M HNO$_3$

Figure 9 Chromatograms of binary separations of metal ions on polyanilinezirconium(IV) arsenate columns. $a = 0.001M$ DMSO, $b = HNO_3$, $c = 0.1M$ $HClO_4$.

TABLE VI
Selective Separations of Pb²⁺ from a Synthetic Mixture of Ca²⁺, Sr²⁺, Ni²⁺, and Pb²⁺ Using Polyanilinezirconium(IV)Arsenate Columns

S. no.	Amount of Pb^{2+} loaded (mg)	Amount of Pb^{2+} found (mg)	% Recovery	Eluent used	Eluent volume (mL)
1	6.22	5.72	92	$0.1M$ HNO$_3$	80
2	12.44	11.20	90	$0.1M$ HNO$_3$	90
3	18.66	15.49	83	$0.1M$ HNO$_3$	110

TABLE VII
Selective Separations of Hg²⁺ from a Synthetic Mixture of Ca²⁺, Ni²⁺, Ba²⁺, Zn²⁺, and Hg²⁺ Using Polyanilinezirconium(IV)Arsenate Columns

S. no.	Amount of Hg^{2+} loaded (mg)	Amount of Hg^{2+} found (mg)	% Recovery	Eluent used	Eluent volume (mL)
1	4.01	3.53	88	$0.1M$ HClO$_4$	90
2	8.02	6.90	86	$0.1M$ HClO$_4$	100
3	12.03	10.11	84	$0.1M$ HClO$_4$	120

CONCLUSION

Semi-crystalline "polyanilinezirconium(IV)arsenate" cation exchanger shows selective sorption for Pb^{2+} and Hg^{2+}. It retains 75% of the initial ion-exchange capacity up to 300°C and can be used for the quantitative separation of metal ions of analytical importance, e.g., Ba^{2+}-Pb^{2+}, Pb^{2+}-Ni^{2+}, Cd^{2+}-Hg^{2+}, Ni^{2+}-Hg^{2+}, Zn^{2+}-Pb^{2+}, Ca^{2+}-Bi^{3+}, Al^{3+}-Hg^{2+}, and Ca^{2+}-Pb^{2+}. The material can further be explored for the removal and recovery of metal ions from industrial effluents. Polyanilinezirconium(IV)arsenate cation exchanger, thus, exhibits the characteristics of a promising ion-exchanger and its environmental applications.

References

1. Jain, A. K.; Sodhi, S. M.; Rajvanshi, S. Indian J Chem 2003, 41, 819.
2. Khan, A. A.; Alam, M. M.; Inamuddin. Mater Res Bull 2005, 40, 289.
3. Mojumdar, S. C.; Varshney, K. G.; Agrawal, A. Res J Chem Environ 2006, 10, 89.
4. Khan, A. A.; Alam, M. M.; Mohammad, F. Electrochim Acta 2003, 48, 2463.
5. Khan, A. A.; Alam, M. M. Anal Chim Acta 2004, 504, 253.
6. Varshney, K. G.; Tayal, N.; Khan, A. A.; Niwas, R. Colloids Surf A 2001, 181, 123.
7. Niwas, R.; Khan, A. A.; Varshney, K. G. Colloids Surf A 1999, 150, 7.
8. Varshney, K. G.; Tayal, N.; Gupta, U. Colloids Surf A 1998, 145, 71.
9. Niwas, R.; Khan, A. A.; Varshney, K. G. Indian J Chem 1998, 37, 469.
10. Nabi, S. A.; Bushra, R.; Naushad, M.; Khan, A. M. Chem Eng J 2010, 165, 529.
11. Nabi, S. A.; Naushad, M.; Bushra, R. Chem Eng J 2009, 152, 80.
12. Nabi, S. A.; Naushad, M.; Bushra, R. J Adsorpt Sci Technol 2009, 27, 423.
13. Nabi, S. A.; Shahadat, M.; Bushra, R.; Shalla, A. H.; Azam, A. Colloids Surf B 2011, 87, 122.
14. Khan, A. A.; Alam, M. M. React Funct Polym 2003, 55, 277.
15. Khan, A. A.; Innamuddin. Sens Actuators B 2006, 120, 10.
16. Rai, N.; Chattopadhyaya, M. C. J Indian Chem Soc 2002, 81, 174.
17. Agarwal, H.; Chandra, S. J Chem Soc 2006, 83, 369.
18. Arrad, O.; Sasson, Y. J Org Chem 1989, 54, 4993.
19. Vatutsina, O. M.; Soldatov, V. S.; Sokolova, V. I.; Johann, J.; Bissen, M.; Weissenbacher, A. React Funct Polym 2007, 67, 184.
20. Raman, N. K.; Anderson, M. T.; Brinker, C. J Chem Mater 1996, 8, 1682.
21. Schubert, U.; Husing, N.; Lorenz, A. Chem Mater 1995, 7, 2010.
22. Anil, K.; Chowdhury, D. K. J Chromatogr A 1974, 101, 63.
23. Guili, X.; Hung, M. R.; Liu, R. React Funct Polym 2005, 2, 285.
24. Topp, N. E.; Pepper, K. W. J Chem Soc 1949, 3299.
25. Snell, F. D.; Snell, C. T. Calorimetric Methods of Chemical Analysis; D. Van Nostrand: New Jersey, 1959; Vol. II. p 334.
26. Nabi, S. A.; Shahadat, M.; Bushra, R.; Shalla, A. H.; Ahmed, F. Chem Eng J 2010, 165, 405.
27. Nabi, S. A.; Khan, A. M. React Funct Polym 2006, 66, 495.
28. Nabi, S. A.; Naushad, M.; Inamuddin. J Hazard Mater 2007, 142, 404.
29. Daval, C. Inorganic Thermogravimetric Analysis; Elsevier: Amsterdam, 1963; p 315.
30. Rao, C. N. R. Chemical Application of Infrared Spectroscopy; Academic Press: New York, 1963; p 355.
31. Silverstein, R. M.; Bassler, G. C.; Morrill, T. C. Spectrometric Identification of Organic Compounds, 4th ed.; Wiley: New York, 1981, Chapter 3, p 111.
32. Nakamoto, K. Infrared Spectra of Inorganic and Coordination Compounds; Wiley: New Jersey, 1981; Vol. 8, p 121.