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Characterizations and Ion Exchange Behavior of A New Organic-inorganic Hybrid Cation-exchanger: Polyacrylamide Ferric Antimonate

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THE POSSIBILITY to reduce the levels of heavy metals from aqueous waste solutions by using polyacrylamide ferric antimonate ion-exchangers has been investigated in this study. These composite ion exchangers have high chemical stability and high loading capacity. The materials were characterized using X-ray diffraction analysis, infrared spectroscopy and thermogravemetric and differential thermal analysis. The distribution coefficients and separation factors of prepared materials were calculated for Fe (III), Pb (II), Cd (II), Cu (II), and Zn (II) ions. The samples prepared show high chemical stability in water, nitric and hydrochloric acids, as well as in potassium and sodium hydroxides. All these results support the suitability of the prepared material for removals of heavy metals from aqueous waste solutions.

Keywords: Characterization, Organic-inorganic hybrid and Distribution studies.

Composites ion exchangers consist of one or more ion exchangers combined with another materials, which can be inorganic or organic and it may be an ion exchanger. Thus, organic-inorganic hybrid materials are expected to provide many possibilities as new composite materials. The hybrid materials usually show intermediate properties between those of plastics and glasses (ceramics). Accordingly, hybridization can be used to modify organic polymer materials or to modify inorganic materials. In addition of these characteristics, the hybrid materials can be considered as new composite materials that exhibit very different properties from their original components (organic polymer and inorganic materials) [1-6]. Attempts are focused in this study to prepare composite ion exchangers for treatment of aqueous waste solutions. Heavy metals are non-degradable in the environment and can be harmful to a variety of living species. For this reason, the removal of these metals from aqueous waste solutions is important in terms of protection of public health and environment. The heavy metals levels in aqueous waste solutions, drinking water, and water used for agriculture should be reduced to the maximum permissible concentration[7]. Ion exchange method is one of the important procedures for removal of the heavy metals from aqueous waste solutions.

Material and Methods

All chemicals used in this work were of analytical grade and used without further purification. Distilled water was employed for all glassware and in preparing aqueous solutions.

Preparation and characterization of samples of polyacrylamide ferric antimonate

Polyacrylamide was prepared by mixing equal volumes of 20% acrylamide prepared in distilled water with 0.1 M potassium persulfate ($K_2S_2O_8$) prepared in 1 M HCl. A viscous solution was obtained by heating the mixture gently at 70 ± 5^0C with continuous stirring.

Sample (I) previously prepared by El-Naggar *et al*. (paper under publication)[8] it prepared by adding precipitate of ferric antimonate to polyacrylamide with stirrer by using magnetic stirrer to obtain homogenous precipitate. The precipitate was left to age in the mother liquor overnight, the precipitate washed by distilled water several times. The supernatant liquid was decanted and the gel was filtered using a centrifuge (about 10^4 rpm) and dried at 50 ± 1^oC. The product was crashed and sieved to obtain different mesh sizes of 0.12-1.00 mm.

Sample (II) was prepared by adding 0.5 M of ferric chloride dissolved in distilled water into polyacrylamide and then all content was added to 0.5 M antimony metal dissolved in aqua regia slowly with constant stirring using a magnetic stirrer at a temperature of 70 ± 5^0C. The resulting homogenous solution was precipitated by using ammonia solution drop by drop until PH $\approx$ 0.13 red brown precipitate will be formed. The precipitate was washed with distilled water until the fine particles were released. The supernatant liquid was decanted and gel was filtered using a centrifuge (about 10^4 rpm) and dried at 50 ± 1^oC. The product was crashed and sieved to obtain different mesh sizes of 0.12-1.00 mm.

Characterization of the prepared samples of polyacrylamide ferric antimonate

Some physical and chemical properties of polyacrylamide ferric antimonate samples were investigated using different techniques; these are:

Chemical stability of samples of polyacrylamide ferric antimonate as ion exchangers was studied in water, nitric and hydrochloric acid [1, 2, 3, 4, 5 and 6M], as well as in potassium and sodium hydroxides (0.1, 1M) by mixing 50 mg of ion exchanger and 50 ml of the desired solution with intermittent shaking for $\approx$ three weeks at 25 $\pm1^o$C. Chemical stability of samples is measured by gravimetric analysis.

Thermal analysis was undertaken using differential thermal analysis (DTA) and thermogravimetric analysis (TGA) with sample holder made of Al_2O_3 in N_2 atmosphere using a Shimadzu DTG-60/60H thermal analyzer obtained from Shimadzu Kyoto, "Japan". The heating rate was maintained at 20^oC/min with using alumina powder as a reference material.

The IR spectra of samples of polyacrylamide ferric antimonate were carried out by mixing of the ground solid with potassium bromide in ratio 1:5. A transparent disc was formed in a moisture free atmosphere. The FTIR spectra were recorded using BOMEM-FTIR spectrometer in the range 400-4000 cm^{-1}.

X-ray diffraction patterns of polyacrylamide ferric antimonite samples were carried out using a Shimadzu XD-D1 obtained from Shimadzu Kyoto, "Japan", X-ray diffractometer with Cu Kα radiation tube source and graphite monochromator operating at 30 kV and 30mA. The measurements were done in 2Ө ranges from 5 to 90 with scan speed 2°/min.

An inductive coupled plasma emission spectrometer model, ICPs-7500, Shimadzu Sequential Type, Kyoto "Japan" was used for measuring the trace concentrations of the different heavy metals in the application part of the work.

An atomic absorption spectrophotometer model, AA-6701 F- Shimadzu, Kyoto "Japan" was used for measuring the trace concentrations of the different heavy metals in the application part of the work.

Chemical composition of the prepared materials can be determined by X-ray fluorescence (XRF) of solid samples with Philips XRF detector, Holland.

Water content and apparent capacity measurements

The water content of polyacrylamide ferric antimonate samples was determined by annealing of 0.5 g of each samples dried at different drying temperatures (200 and 400°C), the results obtained from water content are given in Table 3.

Capacities were determined by repeated batch equilibration of 0.1M metal chloride solutions (pb^{2+}, Fe^{3+}, Cd^{2+}, Zn^{2+} and Cu^{2+}) with the solid material in V/m ratio 50 ml/g was carried out for the capacity evaluation. The mixture was shaked in a shaker thermostat at 25±1°C. After an overnight standing (sufficient to attain equilibrium), the supernatants solution are separated and determined using inductively coupled plasma spectrometer and atomic absorption spectrophotometer. The procedure was repeated until no further absorption of cations is occurred. The capacities were calculated from the following equation:

$$\text{Capacity in meq/g} = \frac{\% uptake}{100} \times C_O \times V/m \times Z$$

where:
C_o: is the initial concentration of solution, g/ml.
V: is the solution volume, ml.
m: is the weight of the exchanger, g.
Z: is the charge of the adsorbed metal ion.

Distribution and selectivity studies

The distribution coefficient (K_d) (define $k_d = C_s/C_l$, where C_s is the ion concentration in solid and C_l is ion concentration in liquid at equilibrium) of some heavy metals such as Fe (III), Pb (II), Cd (II), Cu (II), and Zn (II) ions on polyacrylamide ferric antimonate were determined by batch equilibration. Samples of the solid immersed in metals chloride solution of 10^{-3} M Fe (III), Pb (II), Cd (II), Cu (II), and Zn (II) ions with V/m ratio 100 ml/g as a function of different pH values. The mixture was placed in shaker thermostat adjusted at $25\pm1^{o}C$. After overnight standing (sufficient to attain equilibrium), the liquid phases were separated and measured by using inductive coupled plasma spectrometer (ICPS-7500) and atomic absorption flame emission spectrophotometer (AA- 6701F). The following relation was used to calculate the distribution coefficient (kd):

$$K_d = \frac{[A_o - A_f]}{A_f} \times V\!\!\Big/\!\!m \qquad ml/g \qquad\qquad (1)$$

where:

A_o: is the initial concentration of the considered element in solution.
A_f: is the final concentration of the considered element in solution.
V: is the volume of the solution (ml).
m: is the weight of the ion-exchanger (g).

Results and Discusion

In this study, samples of new hybrid cation-exchanger polyacrylamide ferric antimonate were prepared by mixing organic and inorganic parts. This material appears to be a promising hybrid material with good thermal, mechanical and chemical stability.

Tables 1 and 2 show that the polyacrylamide ferric antimonate samples are physically quite stable in water, HCl and HNO_3 solutions up to 6M; the samples are partially dissolved in potassium and sodium hydroxide solution (0.1 and 1.0M).

The capacity measurements of sample(II) for 0.1M metal concentration in the chloride forms were determined, the results obtained of capacity for pb^{2+}, Cd^{2+}, Fe^{3+}, Cu^{2+} and Zn^{2+} are presented in Table 3 the capacity of sample (I) for the studied metal ions were reported earlier by El-Naggar *et al*. (paper under publication)[8]. It is clear that the ion exchange capacities of different ions show that the order of selectivity for the cation.
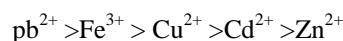
$$pb^{2+} > Fe^{3+} > Cu^{2+} > Cd^{2+} > Zn^{2+}$$

TABLE 1. Chemical stability of sample (I) of polyacrylamide ferric antimonate (mg/l) at 25°C relative errors about ±10%.

HCL	g/L	HNO$_3$	g/L	KOH	g/L
0.5	0.0034	0.5	0.0200	0.1	0.5220
1	0.0132	1	0.0200	1	5.2278
2	0.0132	2	0.0212	NaOH	
3	0.0150	3	0.0242		
4	0.0346	4	0.0270	0.1	0.4582
5	0.0420	5	0.0296	1	3.4224
6	0.0496	6	0.0304		

TABLE 2. Chemical stability of sample (II) of polyacrylamide ferric antimonate (mg/l) at 25°C relative errors about ±10%.

HCL	g/L	HNO$_3$	g/L	KOH	g/L
0.5	0.0036	0.5	0.0203	0.1	0.5430
1	0.0135	1	0.0205	1	5.3237
2	0.0136	2	0.0216	NaOH	
3	0.0151	3	0.0244		
4	0.0347	4	0.0274	0.1	0.4683
5	0.0423	5	0.0298	1	3.4624
6	0.0499	6	0.0308		

This capacities order agrees with the selectivity of the K_d values of these ions on the same materials as discussed later in this thesis. In addition, it may be due to the decreasing of the electronegativity of pb^{2+}, Cd^{2+}, Fe^{3+}, Zn^{2+} and Cu^{2+} (2, 1.94, 1.88, 1.81 and 1.65), respectively, the electronegativity of $pb^{2+} > Fe^{3+}$, Cu^{2+}, Cd^{2+} and Zn^{2+} and also may be due to the ionic radii, the studied ions with smaller ionic radii are easily exchanged and move faster than the ions with greater ionic radii, (0.065, 0.073, 0.085 and 0.097Å), respectively, according to the sequence $Fe^{3+} < Cu^{2+} < Cd^{2+} < Zn^{2+}$.

TABLE 3. Capacity measurements and water contents of polyacrylamide ferric antimonate samples towards pb^{2+}, Cd^{2+}, Fe^{3+}, Cu^{2+} and Zn^{2+} ions

z	H$^+$	Pb^{2+}	Fe^{3+}	Cu^{2+}	Cd^{2+}	Zn^{2+}
Capacity of sample (II),meq/g	-	5.22	4.11	3.05	2.28	2.27
Water contents at 200°C of sample (I) (%)	9.50	6.77	7.00	7.61	7.67	7.78
Water contents at 400°C of sample(I) (%)	30.00	16.10	17.80	18.25	18.56	18.85
Water contents at 200°C of sample(II) (%)	7.01	5.40	5.83	6.20	6.00	6.60
Water contents at 400°C of sample (II) (%)	25.80	14.53	15.21	15.46	16.50	16.98

IR spectra of the prepared ion exchangers were analyzed using infrared spectrophotometer at different drying temperatures 50 ± 1 ^{o}C as shown in Fig. 1. The main absorption band observed for polyacrylamide ferric antimonate at 3245 cm^{-1} can be attributed to the overlapping between the symmetric stretching vibration of NH$_2$ group and H$_2$O, the band at 1660 cm^{-1} from stretching vibration of C=O, the band at 1617 cm^{-1} from the bending vibration of NH$_2$ group, the band at 1457 cm^{-1} from the overlapping of stretching vibration of CH$_2$ group band and bending mode of M-O-H. The band at 1375 cm^{-1} from stretching vibration of C-N band, the band at 1221 and 1120 cm^{-1} from the a symmetric vibration of NH$_2$ and C-C band[9-12] and the band at 550-616cm^{-1} due to metal oxide band which was confirmed by thermal analysis. The IR spectra show that all metals ions exchanged as anhydrate ions as shown from Fig. 2. Figure 2 was taken for lead as an example for the sake of brevity, which was confirmed by TGA.
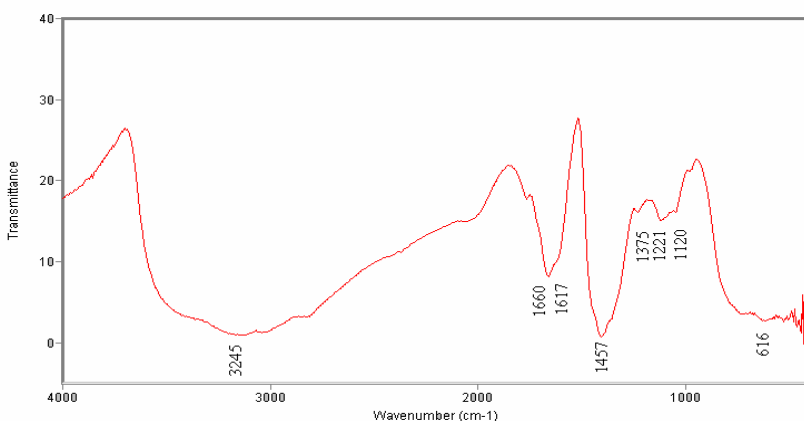
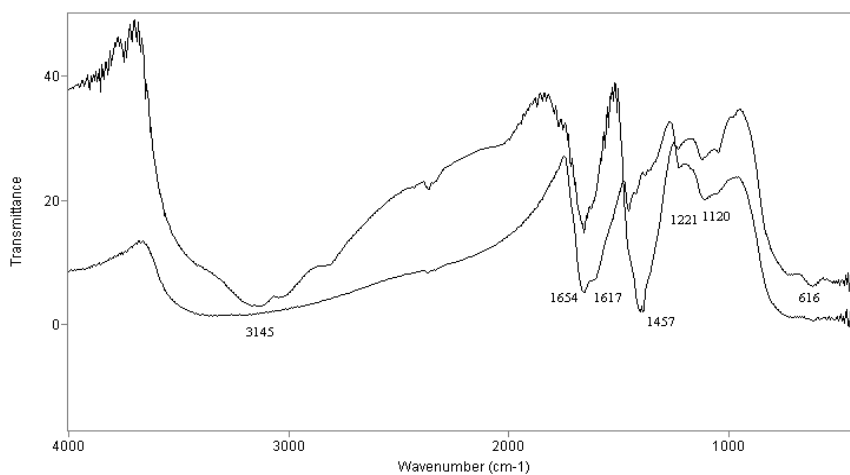**Fig. 1. Infrared spectra of sample (I) of polacrylamide ferric antimonate dried at 50 ±1°.**

Fig. 2. Infrared spectra of polacrylamide ferric antimonate before (b) and after (a) loaded by pb²⁺ dried at 50 °C .

Thermogravimetric (TGA) and differential thermal (DTA) thermograms obtained for the prepared samples of polyacrylamide ferric antimonate are given in Fig. 3 and 4. These thermograms indicated that the first characteristic endothermic peaks for the exchangers are due to the loss of free water observed at ~80-83 °C for sample (I) and ~92.5 °C and 280 °C which may due to the removal of the water of crystallization for sample (II). The second exothermic peaks at ~ 245 and 447.5 °C for sample (I) and ~ 469.5 -649.7 °C for sample (II) may be due to complete decomposition of the organic part of the materials [13-15] and the third endothermic peaks at ~ 663°C may be attributed the formation of metal oxide. It was found that, the weight losses are continued up to 650°C, and no weight loss occurred in the range ~ 650-1000°C. The trends are almost similar to those reported for zirconium phosphate[16]. This supported the fact that polyacrylamide ferric antimonate has a good thermal stability as compared with the other organic ion exchangers.

X-ray diffraction patterns obtained for the prepared ion exchangers are represented in Fig. 5 and 6 and manifested that the prepared samples dried at 50°C have amorphous structure. In addition, the crystalline character of the samples was increased with the heating temperature of the materials in the investigated temperature range, (50-600°C) [17].
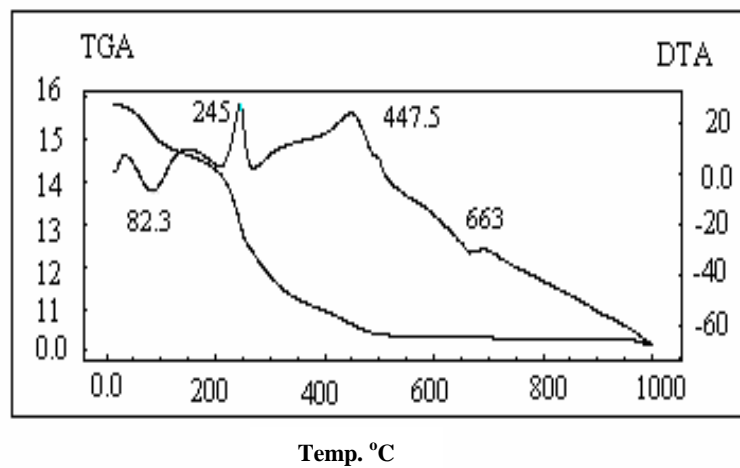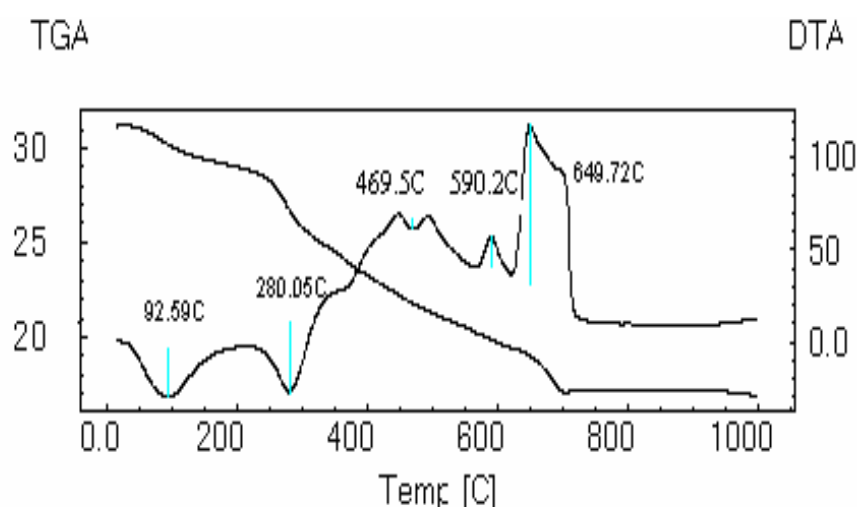
Temp. ᵒC

Fig. 3. DTA- TG thermograms of sample (I) of polyacrylamide ferric antimonate.

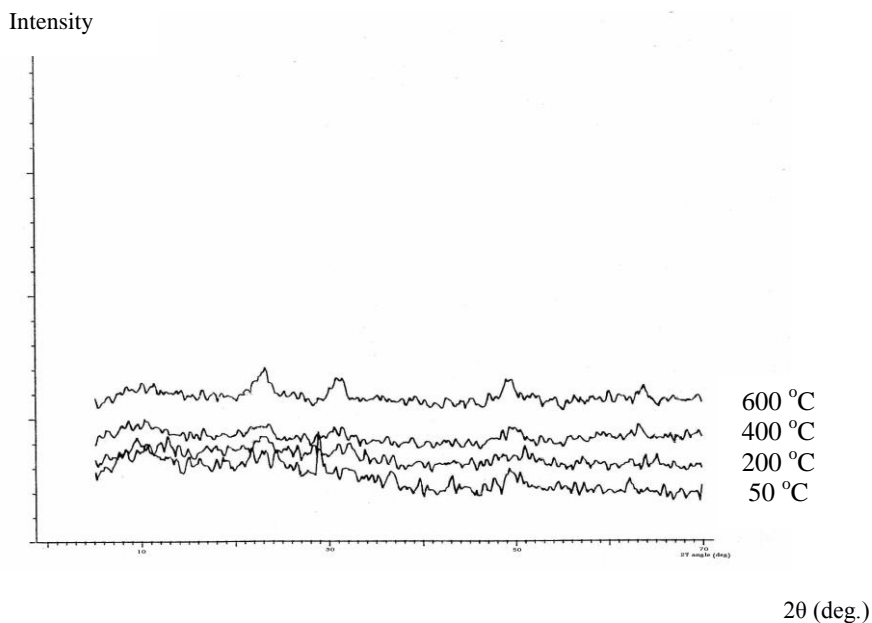**Fig. 4. DTA- TG thermograms of sample (II) of polyacrylamide ferric   antimonate.**

Intensity

2θ (deg.)

Fig. 5. X-ray diffraction patterns of sample (I) of polyacrylamide ferric antimonate at different drying temperatures.
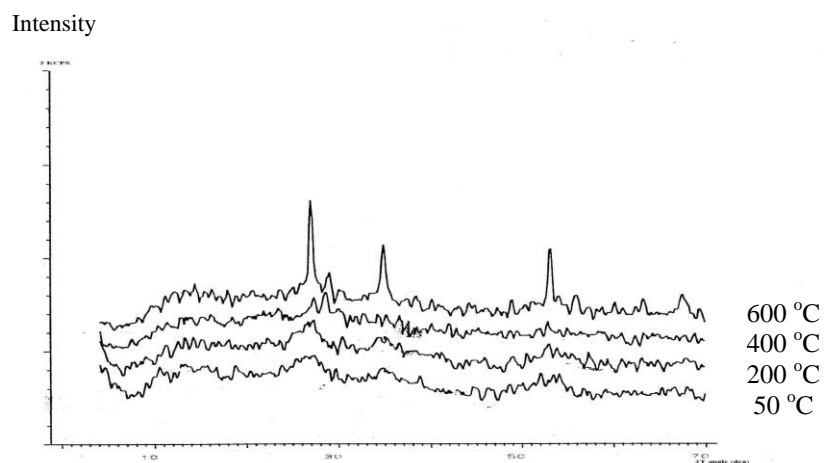
Intensity

Fig. 6. X-ray diffraction patterns of sample (II) of polyacrylamide ferric antimonate at different drying temperatures.

Distribution and selectivity studies

The distribution coefficient is an important parameter to explore the separation potentiality of polyacrylamide ferric antimonate for Fe (III), Pb (II), Cd (II), Cu (II), and Zn (II) ions. The distribution coefficients (K_d) and separation factors (α) for these ions were calculated and tabulated in Tables 4 and 5 for the exchanger samples (I) and (II), respectively.

TABLE 4. Values of distribution coefficients(K_d) and separation factors(α) of Fe^{3+}, Pb^{2+}, Cu^{2+}, Cd^{2+} and Zn^{2+} ions on sample (I) of polyacrylamide ferric antimonate(dried at $50^{\circ}C$ and reaction temperature $25\pm1^{\circ}C$) at different pH values.

| pH	Distribution coefficients(K_d mlg^{-1}) and separation factors(α)				
	pb^{2+}	Fe^{3+}	Cu^{2+}	Zn^{2+}	Cd^{2+}
0.55	111.8	31.4 (3.6)	61.3 (1.2) (0.5)	35.4 (3.2) (0.9) (1.7)	40.3 (2.8) (0.8) (1.5) (0.9)
1.55	320.3	87.7 (3.7)	138.9 (2.3) (0.6)	101.7 (3.1) (0.8) (1.4)	84 (3.8) (1.1) (1.6) (1.2)
2.50	1023	2450 (0.4)	353.2 (2.9) (6.9)	302.1 (3.4) (8.1) (1.2)	320 (3.2) (7.6) (1.1) (0.9)
3.70	19900	7092.8 (2.8)	1674.8 (11.9) (4.2)	1512.5 (13.2) (4.7) (1.1)	1420.8 (14) (5.0) (1.2) (1.1)

TABLE 5. Values of distribution coefficients(K_d) and separation factors(α) of Fe^{3+}, Pb^{2+}, Cu^{2+}, Cd^{2+} and Zn^{2+} ions on sample (II) of polyacrylamide ferric antimonate(dried at $50^{o}C$ and reaction temperature $25\pm1^{o}C$) at different pH values.

pH	Distribution coefficients($K_d\,mlg^{-1}$) and separation factors(α)				
	pb^{2+}	Cd^{2+}	Zn^{2+}	Cu^{2+}	Fe^{3+}
0.55	104.9	78.5 (1.3)	42.8 (2.4) (1.8)	126 (0.8) (0.6) (0.3)	74.6 (1.4) (1.1) (0.6) (1.7)
1.55	306.5	133.8 (2.3)	60 (5.1) (2.2)	177.4 (1.7) (0.8) (0.3)	148 (2.1) (0.9) (0.4) (1.2)
2.50	549.6	251.3 (2.2)	144 (3.8) (1.7)	220.2 (2.5) (1.1) (0.7)	162.7 (3.4) (1.5) (0.9) (1.4)
3.70	2603.2	1266 (2.1)	624.9 (4.2) (2.0)	552.8 (4.7) (2.3) (1.1)	207.2 (12.6) (6.1) (3.0) (2.7)

It was found that the selectivity order of the investigated cations on polyacrylamide ferric antimonite samples at the same condition has the following sequence;

$$pb^{2+} > Fe^{3+} > Cu^{2+} > Zn^{2+} > Cd^{2+}$$
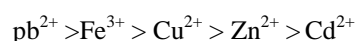
This sequence for sample (I) is shown in Table 4, which may be due to the ionic radii, the ions with smaller ionic radii are easily exchanged and move faster than that ion with greater ionic radii, (0.065, 0.073, 0.085 and 0.097Å), respectively, according to the sequence $Fe^{3+} < Cu^{2+} < Cd^{2+} < Zn^{2+}$. Also, it may be owing to the hydrated ionic radii according to the fact that on increasing atomic number decreasing the hydrated ionic radii.

$$pb^{2+} > Cd^{2+} > Zn^{2+} > Cu^{2+} > Fe^{3+}$$
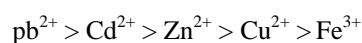
This order of sample (II) shown in Table 5, may be due to the stronger electrostatic interaction and reducing its mobility in the exchanger. Besides, it may be due to the electronegativity of pb^{2+}, Cd^{2+}, Fe^{3+}, Zn^{2+} and Cu^{2+} which are 2, 1.94, 1.88, 1.81 and 1.65, respectively, also may be due to the hydration energy, where the hydration energy of pb^{2+}, Cd^{2+}, Zn^{2+}, Cu^{2+} and Fe^{3+} are 1480, 1806, 2044, 2100 and 4376 kJ/mol, respectively.

It was found that the values of distribution coefficient increase with increasing the pH values, which is a characteristic behavior of cationic exchanger. The data given in Tables 4 and 5 indicate that the distribution coefficients have the affinity sequence $Pb^{2+} > Fe^{3+} > Cu^{2+} > Zn^{2+} > Cd^{2+}$ for sample (I) and $Pb^{2+} > Cd^{2+} > Zn^{2+} > Cu^{2+} > Fe^{3+}$ for sample (II). This sequence may be due to the selectivity of different samples for metals ions. The separation factors for the studied cations were calculated and indicated that, Pb^{2+} ion has a higher separation factor 2.8, 11.9, 13.2 and 14 for Fe^{3+}, Cu^{2+}, Zn^{2+} and Cd^{2+} ions, respectively, on sample (I) and 2.1, 4.2, 4.7 and 12.6 for Cd^{2+}, Zn^{2+}, Cu^{2+} and Fe^{3+} ions, respectively, on sample (II). These values indicate that Pb^{2+} ion can easily be separated from industrial waste solutions including the above cations [18].

Conclusion

The research carried out in this study clearly suggests that polyacrylamide ferric antimonate composite acts a good adsorbent for the removal of some heavy metals from industrial waste water. The following results have been obtained; the polyacrylamide ferric antimonate has a good ion exchange capacity, high stability and high selectivity for heavy metals. The selectivity of polyacrylamide ferric antimonate for Pb (II) and Fe (III) is higher than for other ions for sample (I) and the selectivity of polyacrylamide ferric antimonate for Pb (II) and Cd (II) is higher than for than other ions for sample (II).

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توصيف ودارسة سلوك التبادل الأيونى لمركب البولى أكريلاميد أنتيمونات الحديديك كمبادل كاتيونى

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مركز المعــامـل الحــــارة ـ هيئة الطــاقة الذرية ـ رقــم بريــدى 13759 القـاهرة و*قسم الكيمياء – كلية العلوم – جامعة الزقازيق – مصر .

يتضمن هذا البحث تحضير البولى أكريلاميد أنتيمونات الحديديك كمبادل كاتيونى . وقد تم توصيفه باستخدام الأشعة تحت الحمراء و الأشعة السينية و التحليل الحرارى . كما تم دراسة الثبات الكيميائى للمادة   المحضرة فى الأوساط المائية و الحامضية و القاعدية عند تركيزات مختلفة.

وقد تم تعين معامل الإنتشار لأيونات الرصاص و الحديد و الكادميوم و النحاس و الخارصين داخل مبادل البولى أكريلاميد أنتيمونات الحديديك وتم أيضا حساب معامل الفصل عند إس هيدروجينى مختلف. وقد أوضحت النتائج أن قيم معامل الإنتشار تزداد كلما ذادت قيمة الأس الهيدروجينى. كما وجد أن قيم معامل الإنتشار للأيونات المدروسة تأخذ الترتيب التالى بالنسبة للمادة المحضرة الأولى :

الرصاص > الحديد > النحاس > الخارصين > الكادميوم

كما تأخذ الترتيب التالى بالنسبة للمادة المحضرة الثانية :

الرصاص > الكادميوم > الخارصين > النحاس > الحديد

كما أوضحت النتائج انة يمكن فصل أيون الرصاص عن باقى العناصر الإخرى محل الدراسة.