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ORIGINAL ARTICLE

Adsorption-Desorption Behavior of Cadmium and Lead by modified Poly ethylene terephthalate fiber

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ABSTRACT
Cadmium and lead are known to be toxic for living organism even if it is present in low levels. This study deals with the use of the reactive poly (ethylene terephthalate) (PET) fibers for the removal of cadmium and lead from aqueous solutions. In this study, PET was grafted with 2-hydroxy propyl methacrylate (2-HPMA) as a monomer in the presence of benzoyl peroxide (Bz_2O_2) as an initiator. Reactive fibers were used as a new adsorbent for the removal of cadmium and lead ions from aqueous solutions. The effects of HNO_3 and NaOH on filter adsorption properties were studied. The results were shown that the used reactive fiber was resistance under basic and acidic conditions and ion adsorption was no significant change. Removal of adsorbed cadmium and lead from filter were examined by $Ca(NO_3)_2$, HNO_3 and NaOH. More than 93 percent of the adsorbed ions by the filter were removed by HNO_3. While $Ca(NO_3)_2$ was able to desorbed 40 percent of adsorbed ions.
Keyword: CD, Pd, PET

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INTRUDUCTION

On environmental topics; metals such as lead, mercury, cadmium, nickel and chromium are heavy metals and their effects and compounds are damaging to human health and the environment. These toxins are available in the air, water, building materials, kitchen appliances and even clothing [1, 2].

Cadmium increases naturally in the environment through gradual processes such as rock abrasion and soil erosion as well as from singular events such as volcanic eruptions and forest fires. It is also added to the environment by human activities such as the combustion of fossil fuels, the application of phosphate fertilizers, electroplating, metal production, and the manufacture of batteries, screens, and pigment [3,4].

Cadmium is, therefore, could naturally be present everywhere in soil, water, air, and foods. Greenockite, cadmium sulfide (77.6% Cd), is the most important cadmium mineral. Other minerals are cadmium carbonate (61.5% Cd), otavite, and pure cadmium oxide (87.5% Cd). Greenockite (CdS) is nearly always associated with sphalerite (ZnS) [4]. Cadmium has been classified as a human carcinogen and teratogen impacting the lungs, liver, kidneys, and reproductive organs [1, 5]. A well-known, major case of cadmium poisoning resulted in 'Itai-Itai' disease caused by cadmium contamination of the Jintsu River in Japan. The World Health Organization (WHO) has determined 0.003 mg L^{-1} to be the maximum acceptable concentration of Cd^{2+} in drinking water [6].

Lead is a heavy metal that is also present with other metals, and in agriculture has found many applications (used in pesticides). The source of Lead is mainly from the fuel of gasoline-powered vehicles. In addition, the lead content of chemical fertilizers and white iron pipe corrosion in water pipes, or the dissolution of glazed earthenware can enter in the food chain of animal and human. Transfer of lead in soil and plant tissues is very low. The Limit intake of lead in human solid food is about 600 micrograms per day [7-10].World Health Organization has been suggested the o.3 mg / kg as the limit of lead [WHO].[6]

Because of the low limit of cadmium and lead pollution acceptable in drinking water, there

are significant interests in the development of techniques to remove cadmium and lead from polluted water. Different methods, such as ion exchange, chemical precipitation, reverse osmosis, and ion adsorption cited for the removal of heavy metals from industrial wastewater have been used. The adsorption method, boasting high efficiency and ease of use, is one of the most widely used methods nominated Various adsorbents, including clays, zeolites, dried plant parts, agricultural waste biomass, biopolymers, metal oxides, microorganisms, sewage sludge, fly ash, and activated carbon have been used for cadmium removal [11-15].Investigation of the possibility synthetic fibers for filtration of heavy metals has been started from the 1986 [14]. Zhang et al (1994) used of Carboxyl group containing hydrazine-modified polyacrylonitrile (PAN) fiber as adsorbant [16]. Investigation of this modified ion exchange fiber (IEF) revealed that the maximum capacities of the modified PAN fiber for the heavy metal ions (Cu^{2+}, Cd^{2+}, Zn^{2+}, Co^{2+}, Pb^{2+}, Cr^{3+}, Ni^{2+} , Hg^{2+}) were determined 1.33, 1.30, 1.03, 1.02, 0.98, 0.96, 0.95 and 0.63 mmol/g, respectively. The product could be reused after regeneration with diluted nitric acid. Poly (ethylene terephthalate)-grafted-acrylic acid/acryl amide fiber was used by Ahmad Panahi et al for removal of lead from human plasma and environmental samples [17]. They reported that the sorption capacity of functionalized resin is 44.1 mg g^{-1} and recovery of 100% was obtained for the metal ion with 0.5*M* nitric acid as eluting agent. Hamidpour et al (2010) were used 0.01M Ca(NO3)2 for desorption cadmium and lead from zeolite and bentonite[18]. Arselan was reported 99 percent of chromium (VI) was desorbed by use of 1M KOH[19]. The modified PET for removal of Cd^2 ions and Cationic Brill Red X – 5 GN from aqueous solution were reported by azizinezhad et al [20,21]This research involves the use of poly (ethylene terephthalate) (PET) as an adsorbent. The objectives of this study were to investigate the adsorption-desorption behavior of (PET) grafted with 2-hydroxy propyl methacrylate for the removal Cd^{2+} and Pb^{2+}.It includes desorption properties with Ca^{2+} ion, nitric acid and NaOH and adsorption efficiency after treatment with acid and base.

MATERIALS AND METHODS

Preparation of Adsorbent

PET was used as the polymer base and was grafted with 2-hydroxy propyl methacrylate (2-HPMA) as a monomer using the method described by Azizinejhad, 2011, through the following steps.[22]

PET fibers (44 filaments, 167dTex) were produced by the Loom Department of Amir Kabir University of Technology (Tehran). They were fragmented to small hank size (0.1g) and then cleaned by Soxhlet for 6h with acetone. Finally they were dried at ambient temperature. To remove inhibitors existing in 2-HPMA, it was used after distillation under reduced pressure in inert atmosphere (23mmHg, T=99˚C). Bz_2O_2 (Aldrich) was recrystallized twice from the methanol-chloroform mixture as solvent, dried in a vacuum desiccator, and stored in a refrigerator.

The grafting procedure was achieved in a Pyrex polymerization tube containing 0.5 mol/L monomer, 0.1g PET fiber ,2×10^{-3} mol/L initiator, and 45mL doubly distilled water at 75˚C in a water bath (Lauda D 40 S, Germany). To provide various grafting yields, the operation was carried out in the range of 10-60 min. The removal of undesirable homopolymers was accomplished by N, N-dimethyl formamide (DMF) and (toluene-acetone) mixture for 6 and 8h, respectively

The percent of grafting was determined gravimetrically by the following formula (Eq.1) [22]:

$$\% \text{ Graft} = \frac{W_F - W_0}{W_0} \times 100 \qquad (1)$$

Where W_0 is the weight of the raw PET (mg) and W_F is the weight of the PET after grafting (mg).

Various percentages of grafted copolymer were provided on 3 levels (0-20, 20-40, and higher than 40%). The copolymer with the highest amount of monomer grafted (>40%) was used as adsorbent and its properties were measured.

Adsorbent properties

The specific surface area of the adsorbent was measured with BET (Branuer – Emmet - Teller) method using a Quantochrome Autosorb Instrument and found to be equal to 0.42 square meters per gram.

Cation exchange capacity (CEC) was obtained using the following method:

In the first step, the certain weight of grafted PET was saturated with sodium from sodium acetate. Then the sodium was extracted from the sorbent with an ammonium acetate solution. The sodium concentration in the extract was measured by flame photometer. CEC quantities were calculated to be 45.50 meq per 100 g sorbent.

According to Chaari et al., 2009, by the use of the graph obtained by plotting quantities of pH0.002 and ΔpH (ΔpH= $pH_{0.05}$-$pH_{0.002}$) [18]. The point of zero charge was determined 5.80.[23]

Adsorption properties Stability after treatment with Acid or basic solutions

All sorption experiments were conducted in batch experiments in 0.01M calcium chloride ($CaCl_2$) with the highest grafted copolymer. At adsorbent concentration of 0.25%, shaker speed of 150 rpm, volume solution of 20 mL, and temperature of 25±2°C. In addition to the above condition for cadmium removal used the 5 and 20 mg per liter Cd^{2+} at pH=6.7 and t=60 Min, and for lead removal used the 50 mg per liter Pb^{2+} at pH= 6.0 and t= 30 min.

The amount of cadmium and lead adsorbed were calculated using the following equation (2):

$q = (C_\circ - C) \times V / m$ (2)

where q is the amount of adsorbed cadmium on grafted fiber (mg/g), $C_\circ$ and C are the initial and equilibrium (final) cadmium ion concentrations in solution (mg/L), respectively, V is the volume of solution, and m is the mass of grafted fiber sample used (g).

After adsorption reaction the Cd^{2+} and Pb^{2+} ions were desorbed by 1 M NaOH or HNO_3 solutions.

Adsorption- desorption experiments were repeated for 5 times and after 5^{Th} steps adsorbed amount of Cd^{2+} and Pb^{2+} were determined.

After each step concentration of cadmium and lead were in filtered solution With the use of the AAS (GBC932 AA) instrument at a wavelength of 228.8 and 217 nm, Lead and Cadmium concentrations in the solutions, were measured, respectively.

Desorption procedure

Desorption experiments were did in three methods:

The first desorption experiment was performed in sequential technique immediately following the completion of sorption experiments by the method was used hamidpour et all [18]. Desorption of Cd^{2+} and Pb^{2+} were studied with the fiber samples initially treated with the metal loadings of 25, 50, 75 and 100% maximum sorption capacity during the sorption study. Immediately after removal of the Cd^{2+} and Pb^{2+} from solutions, the fibers washed and putted in 100 ml flask. Then 20 ml of 0.01 M $Ca(NO_3)_2$ solution were added to each flasks at shaker speed of 150 rpm, temperature of 25±2°C , t=60 Min for Cd^{2+} and t= 30 Min for Pb^{2+} desorption.

The second desorption experiment was based on the method which recommended by Arsalan[19] as below steps. Desorption of Cd^{2+} and Pb^{2+} were studied with the fiber samples initially treated with the metal maximum sorption capacity during the sorption study. Immediately after removal of the Cd^{2+} and Pb^{2+} from solutions, the fibers washed and putted in 100 ml flask. Then 20 ml of 1 M NaOH solution were added to each flasks at shaker speed of 150 rpm, temperature of 25±2°C , t=60 Min for Cd^{2+} and t= 30 Min for Pb^{2+} desorption. Then repeat these steps for the second time with the used fiber for desorption.

The third desorption experiment was based on the method which recommended by tabarzady[24] et all as below steps. Desorption of Cd^{2+} and Pb^{2+} were studied with the fiber samples initially treated with the metal maximum sorption capacity during the sorption study. Immediately after removal of the Cd^{2+} and Pb^{2+} from solutions, the fibers washed and putted in 100 ml flask. Then 20 ml of 1 M HNO_3 solution were added to each flasks at shaker speed of 150 rpm, temperature of 25±2°C , t=60 Min for Cd^{2+} and t= 30 Min for Pb^{2+} desorption. Then repeat these steps for the second time with the used fiber for desorption. After each steps solution were filtered then Cd2+ and Pb2+ concentration With the use of the AAS (GBC932 AA) instrument at a wavelength of 228.8 and 217 nm, Lead and Cadmium concentrations in the solutions, were measured, respectively.

RESULTS AND DISCUSSION

Adsorption properties Stability after treatment with Acid or basic solutions

Cadmium adsorption stability after treatment with NaOH

Effect of NaOH treatment on cadmium adsorption at 5 mg/l cadmium concentration is shown in Fig.1. Adsorption of Cd^{2+} per unit of adsorbent changed between from 1.24 to 1.28 mg/g and adsorbent removal 62 to 69 percentage of cadmium from solution. Removed cadmium proportional to the amount of cadmium adsorbed. Amount of desorbed cadmium were changed between 0.07 to 0.11 mg/g. This corresponds to approximately 5 to 8 percent of adsorbed cadmium. After 5 times treatment with NaOH the adsorption effect of fiber was not changed and its equal 1.35 mg/g. its showed the resistance of filter under alkaline condition. Due to the high amount of adsorbed cadmium desorption values did not seem real and the low desorption rate due to deposition of released cadmium in alkaline condition.

Effect of NaOH treatment on cadmium adsorption at 20 mg/l cadmium

concentration is shown in Fig.2. Adsorption of Cd^{2+} per unit of adsorbent changed between from 4.22 to 4.38 mg/g and adsorbent removal 53 to 55 percentage of cadmium from solution. Removed cadmium proportional to the amount of cadmium adsorbed. Amount of desorbed cadmium were changed between 0. 7 to 0.27 mg/g. This corresponds to approximately 2 to 6 percent of adsorbed cadmium. After 5 times treatment with NaOH the adsorption effect of fiber was not changed and its equal 4.29 mg/g. its showed the resistance of filter under alkaline condition. Due to the high amount of adsorbed cadmium desorption values did not seem real and the low desorption rate due to deposition of released cadmium in alkaline condition.

Cadmium adsorption stability after treatment with HNO3

Results of filter treatment with HNO_3 on cadmium adsorption from 5 mg/l cadmium concentration are shown in Fig.3. Adsorption of Cd^{2+} per unit of adsorbent changed between from 1.31 to 1.33 mg/g and adsorbent removal 66 percentage of cadmium from solution. Removed cadmium proportional to the amount of cadmium adsorbed. Amount of desorbed cadmium were changed between 1.27 to 1.29 mg/g. This corresponds to approximately 97 to 98 percent of adsorbed cadmium. After 5 times treatment with HNO_3 the adsorption effect of fiber was not changed and its equal 1.32 mg/g. it's showed the resistance of filter under acidic condition.

Results of filter treatment with HNO_3 on cadmium adsorption from 20 mg/l cadmium concentration are shown in Fig.4. Adsorption of Cd^{2+} per unit of adsorbent changed between from 4.28 to 4.30 mg/g and adsorbent removal 54 percentage of cadmium from solution. Removed cadmium proportional to the amount of cadmium adsorbed. Amount of desorbed cadmium were changed between 4.11 to 4.14 mg/g. This corresponds to approximately 95 to 96 percent of adsorbed cadmium. After 5 times treatment with HNO_3 the adsorption effect of fiber was not changed and its equal 4.29 mg/g. it's showed the resistance of filter under acidic condition

Lead adsorption stability after treatment with NaOH

lead adsorption stability after treatment with NaOH

Effect of NaOH treatment on lead adsorption at 50 mg/l lead concentration is shown in Fig.5. Adsorption of Pb^{2+} per unit of adsorbent changed between from 10.14 to 11.94 mg/g and adsorbent removal 51 to 60 percentage of lead from solution. Removed lead proportional to the amount of lead adsorbed. Amount of desorbed lea were changed between 0.72 to 0.84 mg/g. This corresponds to approximately 7 to 8 percent of adsorbed lead. After 5 times treatment with NaOH the adsorption effect of fiber was not changed and its equal 10.14 mg/g. its showed the resistance of filter under alkaline condition. Due to the high amount of adsorbed cadmium desorption values did not seem real and the low desorption rate due to deposition of released cadmium in alkaline condition.

Lead adsorption stability after treatment with HNO3

Results of filter treatment with HNO_3 on lead adsorption from 50 mg/l lead concentration are shown in Fig.6. Adsorption of Pb^{2+} per unit of adsorbent changed between from 11.92 to 12.04 mg/g and adsorbent removal 60 percentage of lead from solution. Removed lead proportional to the amount of lead adsorbed. Amount of desorbed lead were changed between 11.42 to 11.44 mg/g. This corresponds to approximately 95 to 96 percent of adsorbed lead. After 5 times treatment with HNO_3 the adsorption effect of fiber was not changed and its equal 11.93 mg/g. it's showed the resistance of filter under acidic condition.

Desorption procedure

Cadmium desorption by use of Ca(NO3)

Effect of Cd^{2+}concentration on adsorption is shown in Fig.7. Adsorption of Cd^{2+} per unit of adsorbent increased almost linearly from zero at zero concentration of adsorbate to a maximum of adsorption (removal) of Cd2+ 23.4 mg/g for 120 mg/L of cadmium concentration. This is apparently due to the increase in diffusion forces of Cd^{2+} ions to reach surface of the fiber. Amount of desorbed cadmium were changed between 2.4 to 10.9 mg/g in first step and 1.3 to 7.6 mg/g in second step. This corresponds to approximately 40 percent of adsorbed cadmium. The results showed that Ca^{2+} could desorbed cadmium ions from filter and recovered it.

Cadmium desorption by use of NaOH

Amount of desorbed cadmium in two steps were showed in table 1. It's changed between 1.89 to 0.60 mg/g in first and second step respectively. This corresponds to approximately 8 to 3 percent of adsorbed cadmium. The results showed that NaOH could not desorbed cadmium ions from filter but based on the previous results it can recovered the filter. The high pH cause to precipitation desorbed cadmium.

Cadmium desorption by use of HNO3

Amount of desorbed cadmium in two steps were showed in table 2. It's changed between 22.24 to 1.08 mg/g in first and second step respectively. This corresponds to approximately 95 to 93 percent of adsorbed cadmium. The results showed that HNO_3 desorbed nearly all cadmium ions from filter and

recovered it.

Lead desorption by use of $Ca(NO_3)_2$

Effect of Pb^{2+}concentration on adsorption is shown in Fig.8. Adsorptions of Pb2+ per unit of adsorbent increased almost linearly from zero at zero concentration of adsorbate to 7 mg/g of Pb2+ for 60 mg/L of lead concentration and then decrease the slope with the increase of lead concentration. Maximum adsorption was 8.4 at 120 mg/L of lead concentration. This is apparently due to the increase in diffusion forces of Pb^{2+} ions to reach surface of the fiber. Amount of desorbed lead were changed between 1.9 to 3.6 mg/g in first step and 1 to 1.9 mg/g in second step. This corresponds to approximately 40 percent of adsorbed lead. The results showed that Ca^{2+} could desorbed lead ions from filter and recovered it.

Lead desorption by use of NaOH

Amount of desorbed lead in two steps were showed in table 3. It's changed between 0.65 to 0.21 mg/g in first and second step respectively. This corresponds to approximately 8 to 3 percent of adsorbed lead. The results showed that NaOH could not desorbed lead ions from filter but based on the previous results it can recovered the filter. The high pH cause to precipitation desorbed lead.

Lead desorption by use of HNO3

Amount of desorbed lead in two steps were showed in table 4. It's changed between 7.78 to 0.52 mg/g in first and second step respectively. This corresponds to approximately 93 percent of adsorbed lead. The results showed that HNO_3 desorbed nearly all lead ions from filter and recovered it.

CONCLUSION

The results of the present study showed that PET–g–2-HPMA is a recoverable adsorbent for the removal of Cd^{2+} and Pb^{2+} ions from aqueous solutions. The reactive fiber used was resistance in acidic and basic conditions. HNO_3, NaOH and $Ca(NO_3)_2$ were the most effective for desorption of Cd^{2+} and Pb^{2+} ions respectively. Although use of NaOH caused to precipitation of the desorbed ions in alkaline condition and decrease its concentration.

Fig1. Effect of NaOH treatment on cadmium adsorption in 5 mg/l of Cd^{2+} solution

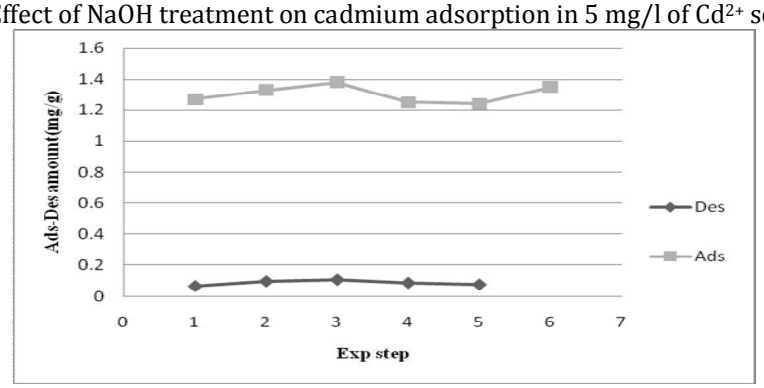

Fig2. Effect of NaOH treatment on cadmium adsorption in 20 mg/l of Cd^{2+} solution

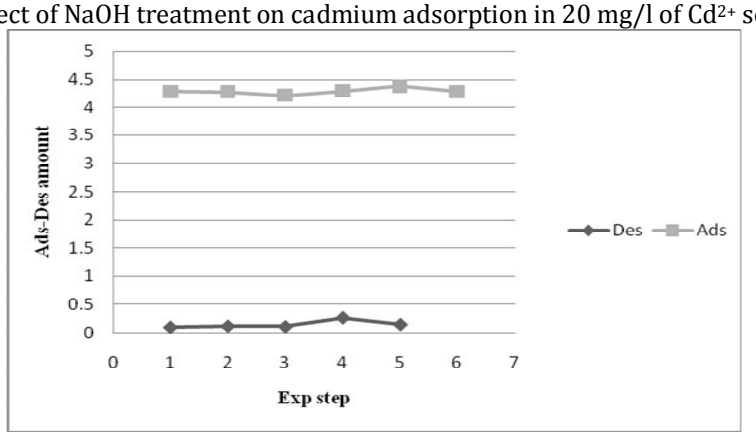

Fig.3. Effect of HNO_3 treatment on cadmium adsorption in 5 mg/l of Cd^{2+} solution

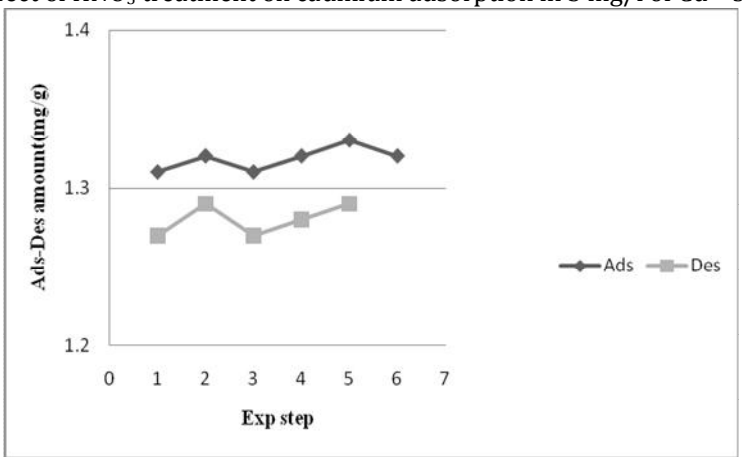

Fig.4. Effect of HNO_3 treatment on cadmium adsorption in 20 mg/l of Cd^{2+} solution

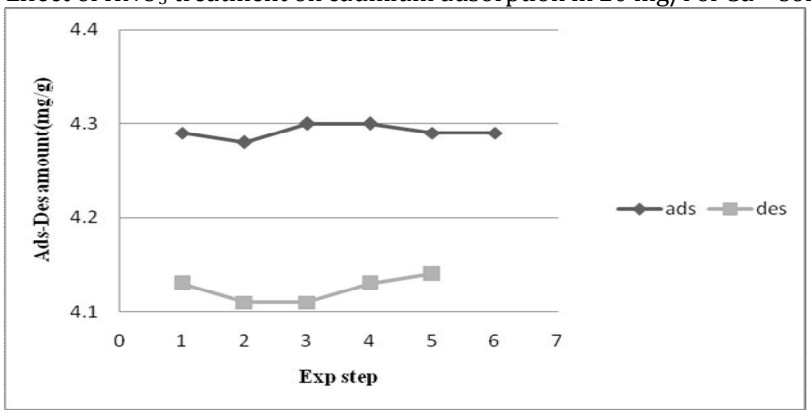

Fig.5. Effect of HNO_3 treatment on lead adsorption in 50 mg/l of Pb^{2+} solution

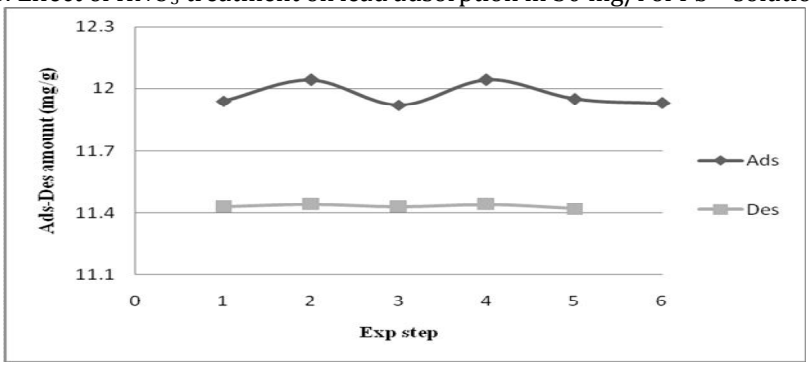

Fig.6. Effect of NaOH treatment on lead adsorption in 50 mg/l of Pb^{2+} solution

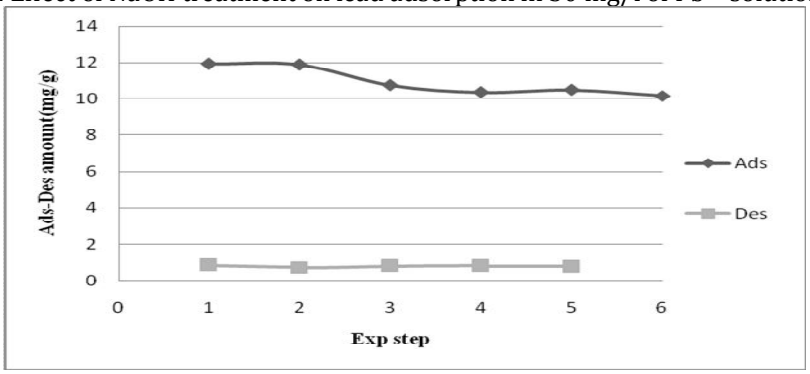

Fig7. Effect of initial Cd^{2+} concentration on adsorption and desorption of Cd^{2+} by Ca^{2+} ion.

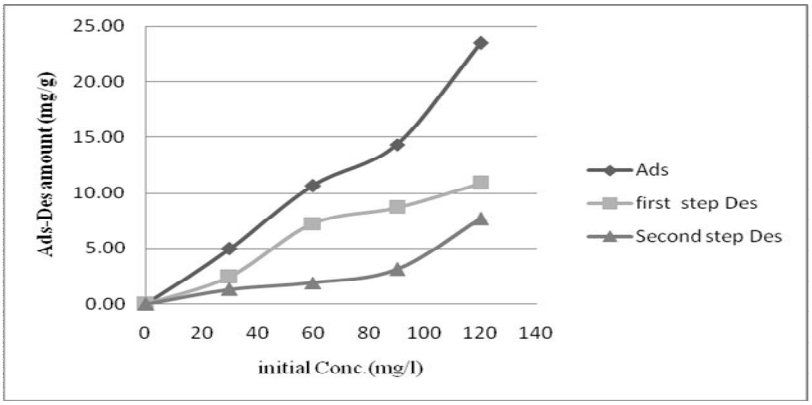

Fig.8. Effect of initial Pb^{2+} concentration on adsorption and desorption of Pb^{2+} by Ca^{2+} ion.

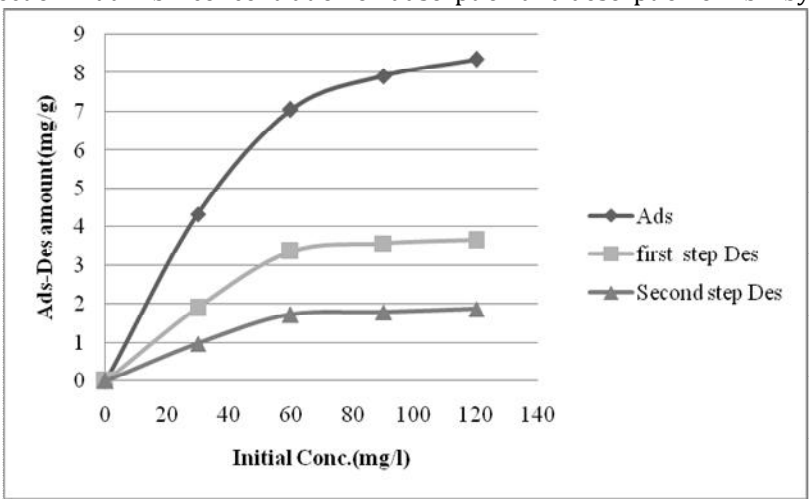

Table.1. Amount of cadmium desorption by use of NaOH

Desorption (mg/g)	adsorption (mg/g)	Secondary Conc. (mg/l)	Initial Conc. (mg/l)	time Min	Step
	23.68	60.8	120	60	Adsorption
1.89	21.79			60	First step desorption
0.60	19.9			60	Second step desorption

Table.2. Amount of cadmium desorption by use of HNO_3

Desorption (mg/g)	adsorption (mg/g)	Secondary Conc. (mg/l)	Initial Conc. (mg/l)	time Min	Step
	23.4	61.51	120	60	Adsorption
22.24	23.4			60	First step desorption
1.08	1.16``			60	Second step desorption

Table.3. Amount of lead desorption by use of NaOH

Desorption (mg/g)	adsorption (mg/g)	Secondary Conc. (mg/l)	Initial Conc. (mg/l)	time Min	Step
	8.44	98.9	120	60	Adsorption
0.65	8.44			60	First step desorption
0.21	7.79			60	Second step desorption

Table.4. Amount of cadmium desorption by use of HNO_3

Desorption (mg/g)	adsorption (mg/g)	Secondary Conc. (mg/l)	Initial Conc. (mg/l)	time Min	Adsorption
	8.34	99.15	120	60	First step desorption
7.78	8.34			60	Second step desorption
0.52	0.56			60	adsorption

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