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SEPARATION AND PRECONCENTRATION OF Cu(II),Ni(II),Cd(II),Pb(II),Mn(II),Zn(II) AND Fe(III) FROM INDUSTRIAL WASTE WATER USING REAGENT- BONDED POLYURETHANE FOAM

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ABSTRACT

The modification of flexible polyether type polyurethane foam (PUF) and incorporation of aminophenol and 2- naphthol or acetyl acetone were utilized for the preparation of three new reagent-bonded foams 2-aminophenol bonded-PUF, hydroxyphenylazoacetylacetone bonded–PUF and hydroxyphenylazonaphthol bonded-PUF. The sorption behaviors of Cu(II),Ni(II),Cd(II),Pb(II),Mn(II), Zn(II) and Fe(III) ions on the prepared foams were studied and the optimum sorption conditions for the tested metal ions were determined. The optimum pH value for the sorption of Cu(II),Ni(II),Cd(II),Pb(II),Mn(II),Zn(II) and Fe(III) ions on the prepared reagents-bonded foams was ranged between 3-8. The maximum sorption capacities of the tested metal ions on the proposed reagent – bonded foams were ranged between $1.2\times10^{-05}$and 8.9×10^{-04} mol g^{-1}. Langmuir and Freundich isotherm constants and correlation coefficients for the present systems were calculated and compared. Columns packed with the proposed reagent foams have been successfully employed for the separation and preconcentration of the above mentioned metal ions from industrial waste waters.

Key wards: New chelating foams, reagent- bonded- foams, trace metal ions, heavy metal, and preconcentration.

1. INTRODUCTION

Separation and pre-concentration techniques, such as liquid-liquid extraction **[Nishihama et al., (2001)]**, solid phase extraction **[Camel et al., (2003); Pereira et al., (2003); Mester et al., (2005); Davis et al., (2006) and Sharaf et al., (2007)]** or co-precipitation **[Sharaf et al., (2007)]** has been widely used in analytical chemistry. Among these techniques, solid-phase extraction has been used frequently as it enables the simultaneous preconcentration of trace elements and removal of interferences. and reduces the usage of organic solvents that are often toxic and may cause contamination. Solid-phase extraction offers ease separation and recovery using continuous flow systems that make possible a high degree of automation **[Pereira et al., (2003)]**.

Flexible polyether type polyurethane foam (PUF) is considered as a good sorbent material in solid-phase extraction **[Cerutti et al., (2005)]**. PUF has been used unloaded **[Braun et al., (1985) and Bowen (1970)]**. loaded with complexing agents **[Nabivanets et al., (1999)]**, immobilizing finely divided solid reagents **[Lemos et al., (2001)]** modified with some chelating agents **[Farag et al., (2007) and Moawad et al., (2003)]**, and bonded with selected organic reagents **[EL.Shahat et al., (2003)]**. The unloaded system has been used mainly for absorption of anionic complexes or organic compounds **[Farag et al., (2005)]**. The loaded and other modified foams depend directly on the immobilizing or bonded reagents.

On–line preconcentration systems utilizing PUF have been recently described **[Lemos et al., (2007) and deJesus et al., (1998)]**. These systems have been coupled with analytical techniques such as molecular absorption spectrophotometry **[Ferreira et al., (1999)]**, flame atomic absorption spectrometry **[Casella et al., (1999); Tarley et al., (2004) and Mohamed et al., (1999)]**, and inductively coupled plasma optical emission spectrometry.

In the present work. the sorption of Cu(II),Ni(II),Cd(II),Pb(II), Mn(II),Zn(II) and Fe (III) ions on these three new chelating foams,2-aminophenol- bonded PUF. hydroxyphenylazoacetylacetone bonded – PUF and hydroxyphenylazonaphthol bonded-PUF. has been investigated. The analytical characteristics of the chelating foams such as pH, equilibrium time and their sorption capacities towards the examined metal ions. were established and the optimum sorption conditions was determined.

2. EXPERIMENTAL

All the reagents used in this work were of analar grade. Atomic absorption. solar 969AA spectrometer was used for all spectro-photometric analysis. pH Meter thermal Orion model 920 A. The conductivity measurements were carried out using a conductivity apparatus. MettlerTolido Mc 226. Flask shaker with variable speed, URN-480. Gemmy. The determination of fluorine in different solutions.Willard and winter distillation apparatus. Flame photometer, Genway for determination of sodium and potassium.

Standard solutions for the heavy metals Cu(II),Zn(II),Cd(II)Pb(II), Ni(II),Mn(II) and Fe(III) were provided from Merck and BDH Chemical-IND CO. Standard solutions of H_2SO_4, NaOH and standard solution of $AgNO_3$ were used for determination of alkalinity as well as chloride content in different solutions

2.1. Preparation of reagent bonded polyurethane foam (BPUF) [Burhan et al., (2008)]:

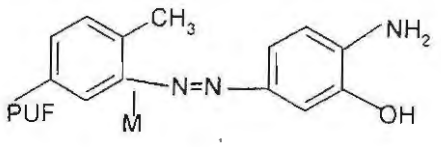
20 gm untreated PUF cubes soaked in HCl 1:1 solution for 1 hour to liberate the maximum number of the free amino groups by hydrolysis of the free isocyanate groups in the foam. The foam was suspended in an ice-water mixture, 350 ml. and treated with one mole $NaNO_2$(added in small portions, 1ml) until the reaction mixture showed a permanent dark blue color with starch-iodine paper. The foam was then attained a yellow color due to the formation of diazonium chloride, then it was left for 1 hour at temperature ranged between 0 C^o and 3 C^o. The foam was then reacted with ortho-aminophenol (10.9 g in 100 ml of 10% NaOH solution). The addition of the last solution was carried gradually with a continuous stirring at 0-3 C^o for 2 hours. The resultant solution was kept below 0 C^o for 25 hours. The resulting brown colored foam was washed with doubly distilled water, acetone and dried in air. The washed product is named amino-phenol bonded PUF (AP-BPUF).

** Preparation of Hydroxyphenylazoacetyle acetone BPUF and Hdroxphenyl azonaphthol BPUF:

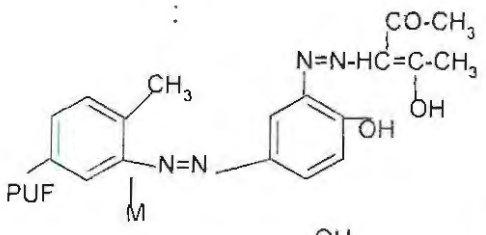
5 g from the produced brown foam was diazotized again by the method mentioned before in order to give Hydroxyphenylazoacetyle acetone BPUF and Hdroxyphenylazonaphthol BPUF. Thus it is coupled with 2- Naphthol (3.6 g in 100 ml 1 mol L^{-1} NaOH), and another amount

of the brown foam was also treated with acetyl acetone (2.5 g AA in 100 ml ethanol and 20 g sodium acetate).

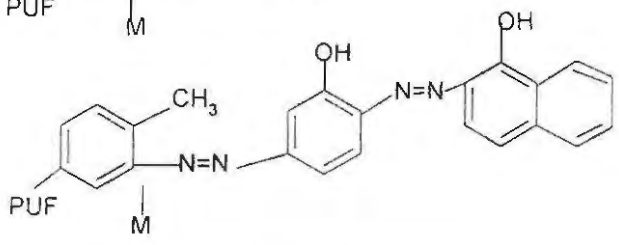
Chemical Structures of the Prepared Modified BPUF :



2 -Aminophenol BPUF



Hydroxyphenylazoacetylacetone BPUF



Hydroxyphenylazonaphthol BPUF

2.2. Foam column preparation:

The glass column, having a stopcock. was 10 cm long, and 1.0 cm in diameter. A small amount of glass beads placed on the disc to prevent loss of the sorbent (foam) during sample loading. Then the column was packed with 1.0 gm of the foam. The foam was packed by gentl pressure with glass rod on the foam cubes onto the column filled with little water to avoid air bubbles. This process was followed by adding glass beads on the top of foam bed to prevent foam flotation upward. The bed height of the foam column was about 60 mm.

2.3. General procedures:

2.3.1. Batch method

The optimum pH of metal ion uptake was determined by batch equilibrium technique. Metal ion solutions 20 ml, 1μg ml^{-1} at different pH values was shaken with 100 mg BPUF for 1 hour, the pH of the metal

ion solution was adjusted before equilibrium over a range of pH ranged between1 and 9 with HCl or NaOH. After equilibrium, the remained metal ions were determined by flame atomic absorption and the recovery percentage was calculated.

2.3.2. Column method

In this method the foam was packed in the glass columns were treated with 20 ml 0.1 mol L^{-1} HNO_3 solution and washed with double deionized water until free from acid. A suitable aliquot of the solutions contain the ions Cu(II) ,Ni(II) ,Cd(II) , Pb(II), Mn(II), Zn(II) and Fe (III) with a concentration amounts to 25 μg L^{-1} was passed through the column after adjusting its pH to the optimum value at flow rate 2 ml min^{-1}. Stripping of the metal ions from the foam was carried out by 0.1 mol L^{-1} HNO_3, 25 ml The eluate was collected in a 25 ml volumetric flasks, the volume was made up to the mark with deionized water and then the solution was aspirated into the flame for AAS determination.

2.3.3. Determination of the sorbent capacity

The total capacity of the modified bonded foam was determined by shaking 20 ml of the metal ion solution of various concentrations (10-120 μg ml^{-1}) with 100 mg of modified foam for 1.0 hour at optimum sorption pH. The reagent foam was filtered off and the concentration of the remaining metal ion was determined by flame AAS. The uptake was calculated from the difference between the original and remaining quantities of different ions as we can see later.

3. RESULTS AND DISCUSSION

3.1. Sorption of metal ions as a function of pH:

The effect of pH on the metal ions on the sorption effeciency using the modified bonded polyurethane foam (BPUF) was examined using the batch method. Aliquots having varying pH values containing each metal ion separately. Were stirred with 100 mg BPUF for 1.0 hour. The percentage sorption of metal ion was calculated from the relation:

Sorption % = $[C_o\text{-}C/ C_o] \times 100$

Where: C_o and C are the initial and remaining concentrations expressed as μg ml^{-1} of the metal ion respectively. It was found that the uptake of different metal ions has a limiting value laid between pH 3 and pH 8, and followed by a decrease at higher PH values. The decrease in percentage

of sorption higher than pH 8 may be due to the precipitation of metals as hydroxides. The results showed the higher affinity of the hydroxyphenyl-azoacetylacetone bonded –PUF (HPAA-BPUF) and hydroxyphenylazon-phthol bonded-PUF (HPA- Naph-BPUF) than
2- aminophenol- bonded PUF (AP-BPUF), this may be due to the presence of a well-known chelating centers used as metal ion chelating sites.

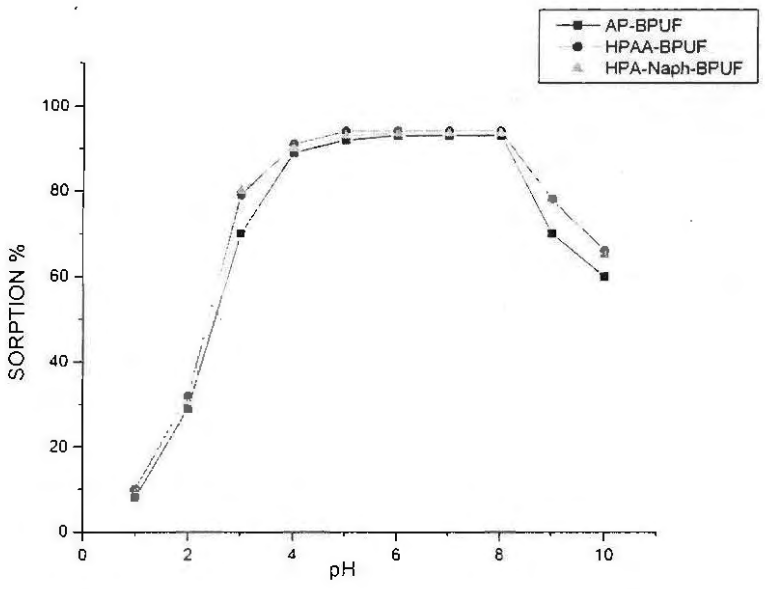


Fig.1.Effect of pH on the sorption of Fe(III) with different modified BPUF

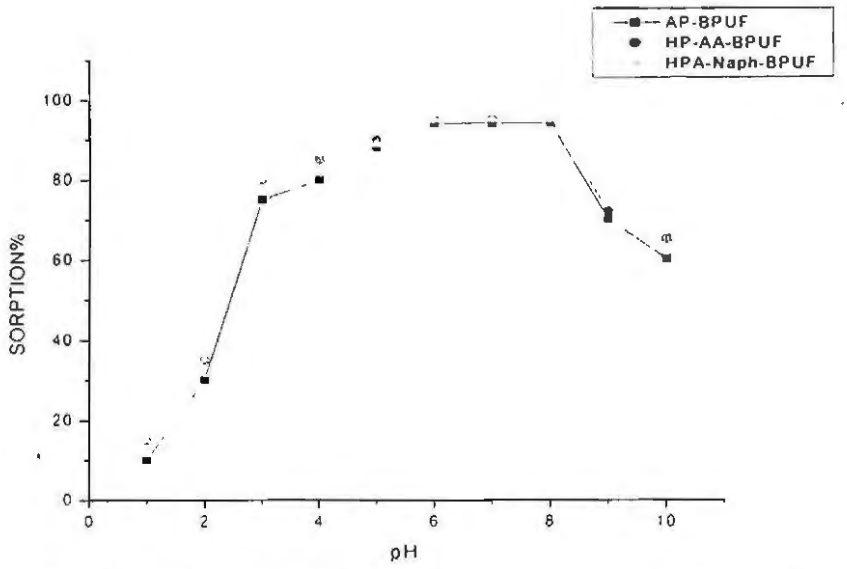


Fig.2.Effect of pH on the sorption of Pb(II) with different modified BPUF

3.2. Sorption capacity of BPUF:

Extraction isotherm for each metal ion with the BPUF was studied. Aliquots of 25 ml solutions containing varying concentrations of metal ion were adjusted to the optimum pH and shaken for 1.0 hour with 100 mg BPUF sorbent at 25 C^0. The amount of remaining metal ion was determined by the recommended method. The adsorption capacity of BPUF at optimum conditions was calculated according to the following equation:

$$Q = [(C_o - C) \times V]/ m$$ at the maximum sorption

where: V= the sample volume in liter, m = the weight of BPUF in gram, C_o = the initial concentration µg / ml, C = the adsorbed concentration.The results of adsorption capacities were presented in Table 1 and illustrated in Figs 3-5. The results showed that, for all metal ions BPUF coupled with two reagents have higher capacities than foams coupled only with one reagent.

Table (1): The calculated capacities for each metal ion at saturation of the foam

BPUF	Sorption capacity mol g^{-1} foam						
	Pb	Fe	Cu	Zn	Cd	Ni	Mn
AP-BPUF	$5.55x10^{-5}$	$2.0x10^{-4}$	$1.96x10^{-4}$	$2.83x10^{-4}$	$1.16x10^{-4}$	$2.81x10^{-4}$	$3.4x10^{-4}$
HPAA-BPUF	$5.80x10^{-5}$	$2.4x10^{-4}$	$2.0x10^{-4}$	$2.92x10^{-4}$	$8.9x10^{-4}$	$2.90x10^{-4}$	$3.6x10^{-4}$
HPA-Naph-BPUF	$5.7x10^{-5}$	$2.23x10^{-4}$	$4.72x10^{-4}$	$3.0x10^{-4}$	$5.78x10^{-4}$	$3.1x10^{-4}$	$3.66x10^{-4}$

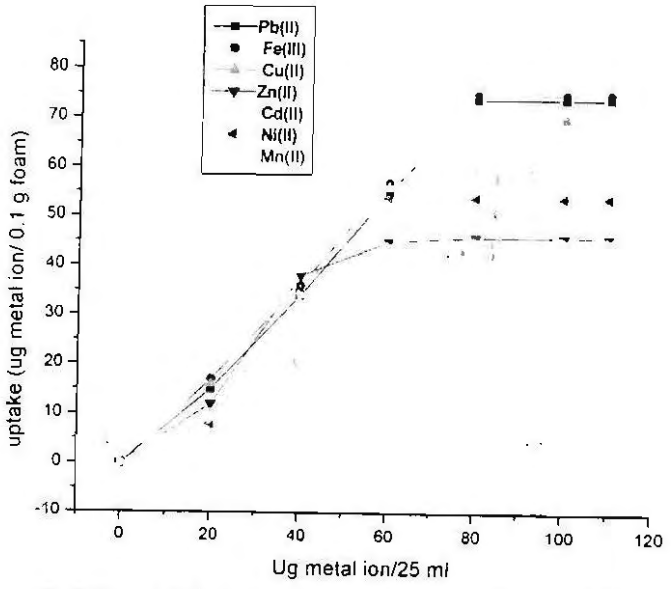


Fig.3 Extraction isotherm of Pb,Fe.Cu,Zn,Cd.Ni,Mn with AP-BPUF.

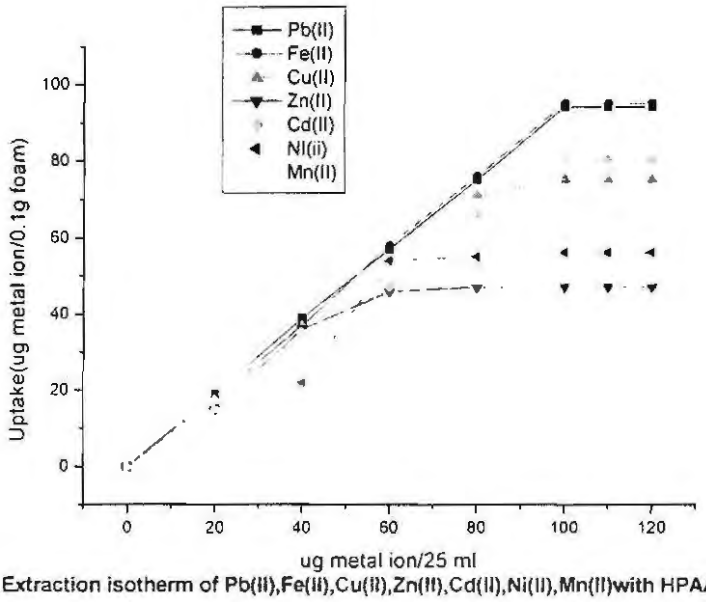


Fig.4.Extraction isotherm of Pb(II),Fe(II),Cu(II),Zn(II),Cd(II),Ni(II),Mn(II)with HPAA-BPUF

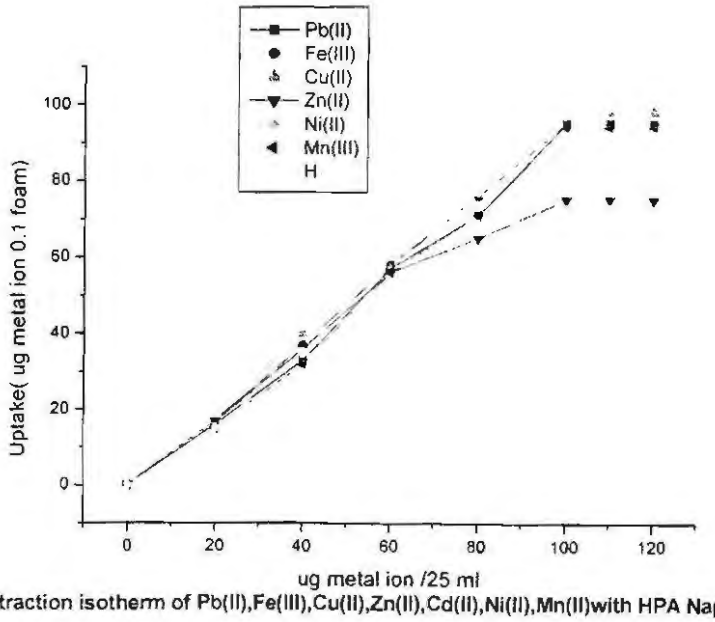


Fig.5.Extraction isotherm of Pb(II),Fe(III),Cu(II),Zn(II),Cd(II),Ni(II),Mn(II)with HPA Naph-BPUF

3.3. Separation of the metal ions using the modified BPUF:

BPUFs filled columns were applied to preconcentrat ion of Pb (II), Fe (III), Cu (II), Zn (II), Cd (II) Ni (II) and Mn (II) from synthetic solutions. The adsorbed metal ions were eluted by 25 ml HNO_3 0.1 M solution .500 ml sample solution of concentration 25 μg L^{-1} from each metal ion were prepared and adjusted to the optimum pH. The results (Table 2) showed that the tested metal ions can be concentrated effectively from large volumes of dilute aqueous solutions up to 1000 ml.The recovery of these metal ions was ranging between 86.0 to 100.0 % which is quite reasonable in separation and in analytical work.

Table (2): Results of the preconcentration of the examined metal ions.

Metal ion	AP-BPUF		HPAA-BPUF		HPA-Naph-BPUF	
	Eluted metal ion ug	% Recovery	Eluted metal ion ug	% Recovery	Eluted metal ion ug	% Recovery
Pb	11.25	90.0	12.5	100	11.25	90.0
Fe	12.0	96.0	12.38	99.0	12.5	100
Cu	10.75	86.0	11.88	95.0	10.75	86.0
Zn	12.0	96.0	12.38	99.0	12.0	96.0
Cd	12.38	95.0	12.5	100	12.5	100
Ni	11.5	92.0	12.5	100	11.5	92.0
Mn	11.38	91.0	12.5	100	11.75	94.0

The efficiency for separation of metals from different aqueous media of these modified foams was tested on the water of the cooling towers of the sulfuric acid unit in AZFC (Abu Zaable for Fertilizers and Chemicals Company-Egypt). The analysis of water is shown in table (3). The results obtained for the separation and preconcentration of the examined metal ions from this industrial water was presented in Table 4where it was found that the recovery of these metals ions was ranging between 89.3 to 106.66% .

Table (3): Analysis of water of the cooling towers of the sulfuric acid unit in AZFC.

Component	Analysis	Item	Analysis
Fe^{+++}	32.0ppm	pH	8.83
Ni^{++}	0.75ppm	Ca^{++}	48.33ppm
Mn^{++}	0.38ppm	Mg^{++}	13.37ppm
Zn^{++}	1.0ppm	P^{+++}	6.55ppm
Cd^{++}	0.19ppm	Conductivity	527µs/cm
Cu^{++}	0.44ppm	P- Alkalinity	15.0ppm
Pb^{++}	0.35ppm	M- Alkalinity	185ppm

Table (4): Percentage recovery of different ions from water of cooling tower by the BPUF.

Metal ion	AP-BPUF		HPAA-BPUF		HPA-Naph-BPUF	
	Eluted metal ion ppm	% Recovery	Eluted metal ion ppm	% Recovery	Eluted metal ion ppm	% Recovery
Pb	0.31	89.3	0.35	100	0.31	89.3
Fe	29.44	92.0	30.08	94.0	29.76	93.0
Cu	0.38	85.71	0.48	108.57	0.44	100
Zn	0.90	96.0	0.98	101.96	0.89	95.33
Cd	0.18	96.66	0.2	106.66	0.19	100
Ni	0.68	90.0	0.75	100	0.69	91.66
Mn	0.34	90.0	0.38	100	0.35	93.33

3.4. Adsorption isotherms:

In order to investigate the thermodynamic parameters in terms of adsorption isotherms, a series of experimental points for the adsorption of metal ions (Pb^{++} and Fe^{+++}) on the BPUFs were taken into consideration two commonly used mathematical expressions to describe the adsorption equilibrium; namely are Freundlich and Langmuir isotherm models were tested with the experimental data. The Freundlich isotherm **[Mohamed M.S.et al., 1999]**, most widely used for mathematical description of adsorption. This isotherm gives an empirical expression encompassing the surface heterogeneity and the exponential distribution of active sites and their energies. The Freundlich model is expressed as: $q_e = K_f C_e^{1/n}$ **or** $\text{Log } q_e = \text{Log } K_f + 1/n \text{ Log } C_e$ (the linearised form)

Where: q_e amount of the metal ion adsorbed (mol g^{-1})

C_e amount of the metal ion in the solution mol L^{-1}

K_f and **1/n** are characteristic constants.

From the plots of Log q_e vs. Log C_e illustrated in Figs(6,7)for Fe^{+++} and Pb^{++}, the Freundlich constants 1/n and K_f were calculated where the first constant equals the slope whereas the second one equals the intercept. The numerical value of adsorption capacity (K_f), and **(1/n)** indicating the free energy and capacity of adsorption process. The numerical value of 1/n< 1 indicates that sorption capacity is only slightly reduced at lower equilibrium concentrations. The results were tabulated in table (5):

Table (5): Frenudlich isotherm parameters for the sorption of Fe^{+++} and Pb^{++} with modified BPUF

Frenudlich isotherm parameters	Metal ion Fe^{+++}			Metal ion Pb^{++}		
	BPUF			BPUF		
	AP-BPUF	HPAA-BPUF	HPA-Naph-BPUF	AP-BPUF	HPAA-BPUF	HPA-Naph-BPUF
R	0.99115	0.9924	0.96743	0.98838	0.9460	0.97399
1/n	0.66	0.44	0.47	0.21614	0.31998	0.52
K_f mol g^{-1}	3.2×10^{-2}	1.54×10^{-3}	2.12×10^{-3}	2.06×10^{-5}	9.46×10^{-05}	1.49×10^{-04}

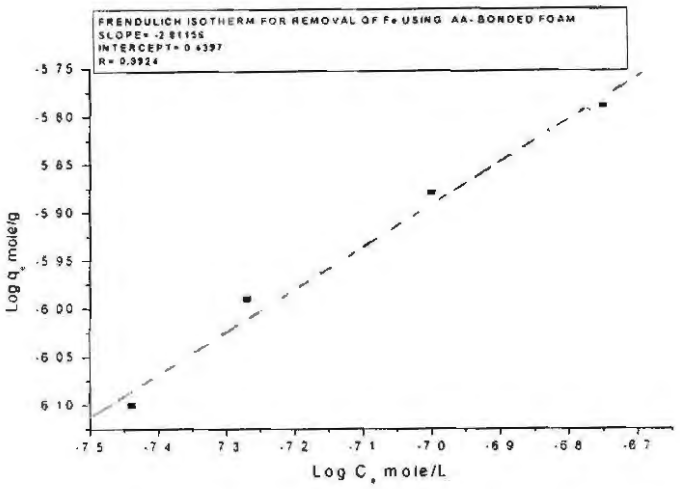


Fig.6. Freundlich isotherm for the sorption of Fe (III) with HPAA-BPUF

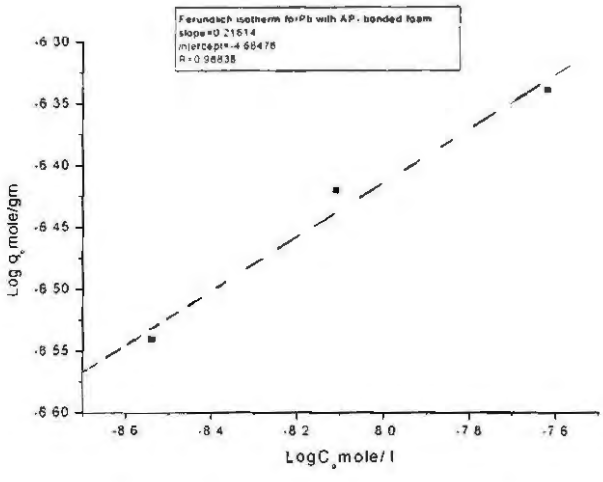


Fig.7. Freundlich isotherm for the sorption of Pb (II) with AP-BPUF

The Langmuir model was originally developed to represent chemisorption on asset of well defined localized adsorption sites having the same sorption energy. independent of surface coverage and no interaction between adsorbed molecules. The familiar form of Langmuir isotherm based on the kinetic consideration is expressed as: $C_e / q_e = 1/Q_o\, b + C_e / Q_o$

Where: C_e = equilibrium concentration of the metal ion in the solution. mol L^{-1}

q_e = amount of the metal ion adsorbed on the BPUF. mol g^{-1}

Q_o = constant related to maximum amount of metal ion adsorbed

b = constant related to the binding energy of the metal ion

For testing the curve fit of the Langmuir model to experimental data, different plots of C_e / q_e against C_e as shown in Figures (8, 9).
The model parameters Q_o and **b** were obtained from the slope and intercept of the plot. The value of Q_o corresponds to the monolayer coverage and independent of temperature, while the sorption coefficient b is related to the enthalpy of adsorption.

Table (6):tabulate the obtained results for Fe^{+++} and Pb^{++} with the BPUFs

Langmuir isotherm parameters	Metal ion Fe^{+++}			Metal ion Pb^{++}		
	BPUF			BPUF		
	AP-BPUF	HPAA-BPUF	HPA-Naph-BPUF	AP-BPUF	HPAA-BPUF	HPA-Naph-BPUF
R	0.93885	0.9995	0.9971	0.99987	0.97202	0.94783
Qo mol g^{-1}	$33.23x10^{-08}$	$20.84x10^{-7}$	$24.07x10^{-7}$	$5.027x10^{-7}$	$6.21x10^{-7}$	$7.57x10^{-7}$
B dm^3 mol^{-1}	36.14	5.39	8.594	$4.27x10^{8}$	$1.93x10^{9}$	$9.81x10^{7}$

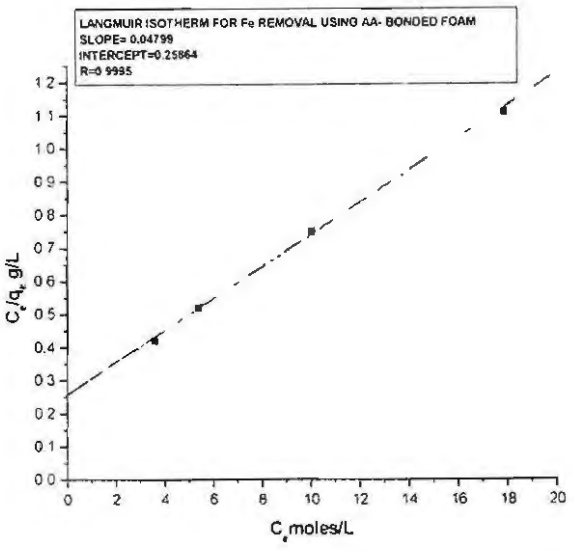


Fig.8.Langumir isotherm for the sorption of Fe (III) with HP AA-BPUF

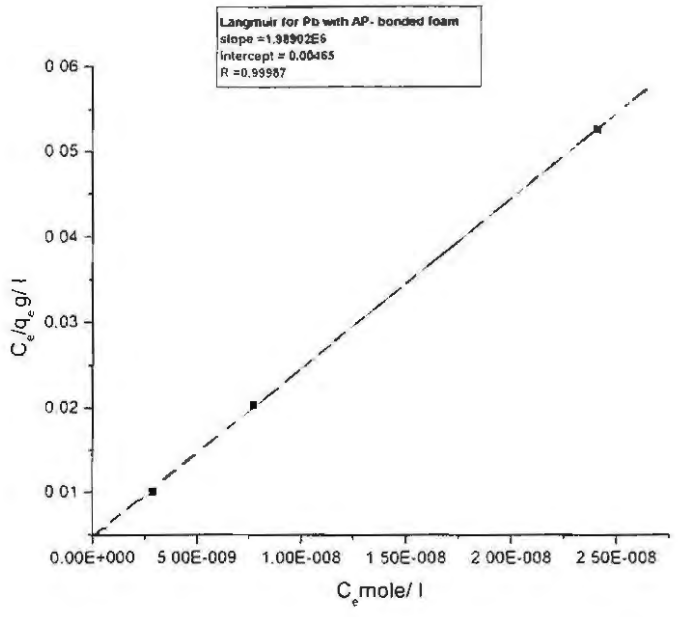


Fig.9.Langumir isotherm for the sorption of Pb (II) with AP-BPUF

CONCLUSION

This work deals with the separation and preconcentration of metal ions of Pb(II),Fe(III),Cu(II),Zn(II),Cd(II) Ni(II) and Mn(II) from industrial water using new polymeric extractors. These modified BPUF were prepared based on covalent coupling of 2- aminophenol with PUF through an azo group. This bonded foam was coupled with acetyl acetone as well as with β- Naphthol to produce two new modified BPUF (hydroxyphenylazoacetylaceton BPUF and hydroxyphenylazonaphthol BPUF.The optimum pH for adsorption and separation of different ions using these three modified BPUF was found to be ranged between 3-8.
The maximum sorption capacities for these BPUF were ranged between $1.2\times10^{-5}$and 8.9×10^{-4} mol g^{-1}.

The thermodynamic consideration in terms of adsorption isotherms was studied, where the adsorption of the metal ions with these modified BPUF is follow Langmuir and Freundlich adsorption isotherms. These BPUF have good application on the separation and preconcentration of heavy metals from industrial water as cooling water of the sulfuric acid unit.
The second coupled foam was found to be more effective in separation process than 2- aminophenol BPUF.

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فصل وتركيز أيونات CuII,NiII.CdII,PbII.MnII,ZnII,FeIII, من مياه صرف صناعية باستخدام رغوة البولى يوريثان المطعمة ببعض الكواشف

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تطوير رغوة البولي يوريثان وذلك بإدخال عليها بعض الكواشف مثل أمينو فينول و٢- بيتا نافثول وأستيل اسيتون ، هذا للحصول على ثلاثة أنواع جديدة من الرغوة المرتبطة بالكاشف الكيميائي وهم : ٢- أمينو فينول المرتبط مع الرغوة ، هيدروكسي فينيل آزو أسيتيل أسيتون المرتبطة بالرغوة هيدروكسي فينيل آزو نافثول المرتبط بالرغوة.

عملية الادمصاص لايونات CuII.NiII.CdII.PbII.MnII.ZnII.FeIII بواسطة هذه الرغوة المحضرة درست وحددت أنسب ظروف الأدمصاص ولقد وجد أن أنسب قيم الاس الهيدروجيني التي يتم عندها الأدمصاص لايونات هذه العناصر بواسطة الرغوة المحضرة تتراوح ما بين ٣-٨

أقصى قيم السعة لهذه المستخلصات المحضرة بواسطة الرغوة المرتبطة مع الكواشف المذكورة يتراوح ما بين ١,٢ x ١٠$^{-٥}$ و ٨,٩ x ١٠$^{-٤}$ مول/جم.

وقد تم دراسة عملية الأدمصاص هذه وتطبيقها على منحنيات لانجمير وفريندلش تم حسابها ومقارنتها.

أيونات العناصر المذكورة قد فصلت وتركزت باستخدام أعمدة من الرغوة المحضرة والمطعمة بالكواشف المذكورة وقد تم تطبيق ذلك على مياه صرف صناعية.