PREPARATION, IDENTIFICATION AL (III) AND SELECTIVE ADSORPTION BY IMPRINTING POLYMER IN WATER IN BAGHDAD

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ABSTRACT

This research aimed to form a molecularly imprinted polymer by adding aluminum ion at the first step were to the allyl bromide monomer resulted was formation using bulk polymerization. To acquire the highest adsorption capacity, molar ratios of template, monomer, and cross-linking agent, as well as solvents and multiple monomers, were investigated. Scanning electron microscopy (SEM) was used to analyze the produced aluminum. the maximum adsorption capacity of Al-IIP were 0.1531 µmol/g and 0.2362 µmol/g respectively. aluminum adsorption followed a Langmuir isotherm model. Solid-phase extraction (SPE) syringe packed with ionic imprinted polymers (IIP) were used to selective separation and preconcentration for aluminum (III) ion from aqueous solutions to determine the aluminum by flame atomic absorption spectroscopy (FAAS).

Keywords: isotherm, monomer, langmuir, allyl bromide

تحضير وتحديد بالامتزاز الانتقائي للايون الالمنيوم بالطبعة البوليمرية في المياه في بغداد

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يحيى كمال البياتي — استاذ
قسم الكيمياء . كلية العلوم . جامعة بغداد . بغداد . العراق

المستخلص

يهدف البحث إلى تكوين بوليمر مطبوع جزيئيًا عن طريق إضافة أيون الألومنيوم في الخطوة الأولى إلى مونومر بروميد الاليل الناتج عن تكوين البلمرة السائبة,للحصول على اعلى سعة امتصاص .تم فحص النسب المولية للقالب والمونمر وعامل الربط المتبادل , وكذلك المذيبات والمونمرات المتعددة. تم استخدام الفحص المجهري الالكتروني .لتحليل الالمنيوم الناتج .كانت اعلى سعات امتزاز للالمنيوم 0.087 (مول/جم) و0.089 (مول /جم) على التوالي . يتبع امتزاز الالمنيوم من نوع لانكماير .تم استخدام حقنة للاستخلاص بالطور الصلب (SPE) المعبأة بالبوليمرات الأيونية المطبوعة (IIPs) للفصل الانتقائي والتركيز المسبق لأيون الالمنيوم(III) من المحاليل المائية لتحديد الالمنيوم عن طريق مطيافية الامتصاص الذري باللهب (FAAS) .

الكلمات المفتاحية:ايزوثرم, مونمر ,لانكماير , بروميد الاليل

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INTODUCTION

The sole oxidation state in which aluminum is found is AL^{3+}, making it the third most significant element on Earth. Aluminum is a metal that humans are exposed to through food, drink, and medications (10). Nausea,mounth and ulcers ,skin and diarrhea are all of signs of almuinum exposure . Those who consume aluminum's will experienceeffect on their neurological systems.This leads to memory loss, balance problems, and a lack of coordination (9). Aluminum (8.1%) is the most common metal found in the Eearth's crust, though it is never found naturally by itself .pure aluminum is a lightweight , non magnetic, silvery –white with numerous beneficial qualities . It is widely utilized in thousands of industria applications , including those requiring robust, lightweight materials for construction as well as for cooking utensils and exterior buildingdecoratio (3). Ionic Imprinted polymer (IIP) technique is ionic Polymer that has binding sites and imprinting holes which are highly similar toa specific type of Molecule (The templates) (1, 2, 6, 11). In other words, imprinted cavities can bind to the templates in size, Shape, and Functional divisions. The templates are combined with the Functional polymer during bulk polymerization (7, 8, 11, 15). The ionic imprinting Procedure is simple and involves Copolymerizing a functional monomer or a Series of functional monomers, a cross-linker, And an initiator in the presence of a catalyst (18, 25, 28). The Functional monomer has unique functional Groups to connect with in template molecules (24, 26). An imprinted polymer with a permanent Memory for the imprinted species is created After Polymerization and extraction from the Template molecule (14, 19, 27). This allows the polymer to Preferentially rebind the imprinted molecule from a mixture of nearly comparable molecules (21, 22). The high degree of cross-linking Enables the micro cavities to maintain them Shape after the template is removed, resulting in A more effective process. The Three-Dimensional Cavities that are complementary to Those of the template in terms of shape and Chemical functionality arrangement should be left in the polymer matrix (12,13). Ionic Imprinting Has evolved into a potent technology for Creating durable Materials with high selective adsorption of target chemical species (5, 23) Imprinted ion, which includes the specificity of the ligand-Metal interaction, is what gives ion-Imprinted Polymer (IIP) their high selectivity, their metal Ion and their size coordination Geometry, Coordination number their metal ions and their Charge, and their charge density. By utilizing the specialized IIP technology, IIP technology can be in the adsorption of heavy metal (16). Ion imprinting is a simple and effective separation technique to produce specific adsorbents for different metal ions (17). The primary benefit of using IIP is the low cost of preparation when compared to other materials with comparable selectivity over biological counterparts. It is also relatively inert in acids, bases, and organic solvents, and has excellent stability over a wide range of temperatures and pressures. Its structure must be stiff enough to maintain the cavity structure after the mold has been removed, while also allowing for Mold release and Reception. Environmental contaminants, medication delivery, food Analysis, chemical Sensors, proteins, and receptor systems (17). are all examples of Modern IIP applications. Because of them numerous advantages, including cost effectiveness High stability affinity to target-Molecule and easy integration into standard fabrication methods, IIP have garnered attention globally. Consequently, numerous applications have been developed incorporating MIPs, such as SFE, Affinity separation, chemically sensors, immunity Testing and drug delivery monitoring (5). Because of their numerous advantages, including low Cost, high stable, affinity to target-molecule and easily integrate into standard fabrication methods, MIPs have attracted attention in the world. Therefore, a number of applications have been Developed incorporating MIPs, such as SFE, affinity separating, chemically sensors, immune Testing, or drug delivery monitoring. Both the silicone and the mold have been cleaned and contain clear areas that have been identified as identification sites. These sites and model particles Complement each other in shape, size and chemical function. IIP shows the ability to selectively Select the template and its derivatives. This study was aimed to synthesis new molecular imprinted polymers from different monomers which useful for determination of aluminum ion in different water samples.

MATERIAIS AND METHODS

Aluminum chloride dihydrat (99.9%) Sigma-Aldrich, Allyl bromide (99.9%) Sigma-Aldrich, Ethylene glycol methacrylate (EGDMA) (99.9%) Sigma-Aldrich benzoyl peroxide were taken from Sigma Aldrich (St. Louis, MO, USA, www.sigma-aldrich.com), methanol, nitrogen gas (99.99%) and acetic acid were purchased from Merck (Darmstadt, Germany).

Mip procedure

For preparation aluminum ionic imprinted polymer (aluminum-IIP) was take 1mmol (0.2414g) of $AlCl_3.6H_2O$was (aluminum-IIP) was take 1 mmol (0.2414 g) of $ALCL_3$.6H2O dissolved in (2 ml of methanol) was mixed with 4 m mol (0.4g) of allyl bromide as the monomer in 2 ml of methanol was added and held for several second at room temperature. Adding 20 mmol (3.9 g) of ethylene glycol methacrylate (EGDMA) in 2 ml of methanol to the solution as the cross linker and (0.3 g) of benzoyl peroxide as the initiator, the mixture was shaken for five minutes. N_2 is passed through the mixture for 30 minutes to extract oxygen from the solution. Then the solution was placed in a water bath at 60°C overnight. When the reaction is complete, the ionic imprinted polymer becomes hardened. After the polymerization process, the polymer is dried and crashed to obtain a polymer particle. To successfully remove the template from IIP, Soxhlet solid liquid phase extraction was performed using Porogenic solvents (v/v) (methanol, acetic acid) 60:10. The polymer was dried at room temperature after being removed by repeated washing for 16–18 hours. Polymers were crushed in a mortar and sieved to a particle size of 125 microns. Each vacuum plastic syringe (column) was packed with Al-IIP (0.15 g) with used 3ml solution for solid phase extraction.

Sampling procedure

Stock solution was prepared at concentration 100 ppm by dissolving 0.111 g of $AlCl_3.6H_2O$ in distilled water in 100 ml volumetric flask and supplementing to the mark. From stock solution 100 ppm prepare 0.1, 0.2, 0.5, 1, 1, 5, 2, 2.5and 3 ppm in distilled water in 25ml volumetric flask and supplementing to the mark. The aluminum ion is detected by the following steps:

A- Dissolve 3.4 g of sodium acetate in distilled water in a 250ml volumetric flask and supplement to the mark

B- Dissolve 0.15 g Eriochrome cyanine R dye in distilled water in 100ml volumetric flask and complete to the mark. This preparation expires for one a year. 10 ml from the prepared dye is taken and supplemented with distilled water in volumetric flask of 100 ml. This preparation expires for six months

C- Dissolve 0.1 g of ascorbic acid in distilled water. and completed to the mark in

D- 1N was prepared from acetic acid

E- 0.02 N was prepared from H_2SO_4

Next, standard solutions of 0.1, 0.2, 0.5, 1, 1.5, 2, 2.5, and 3 ppm that were previously prepared and transferred to a 50 mL volumetric flask and followed by these steps:

a- 1 ml of H_2SO_4 was added to each of the blank and standard solutions

b- 1 ml of ascorbic acid was added to each of the blank and standard solutions

c- 10ml of sodium acetate was added to each of the blank and standard solutions

d - 5ml of Eriochrome cyanine R dye was added to each of the blank and standard solutions and industrial water. The volume is completed in distilled water to 50 ml volumetric flask and left for 15 minutes, before the measurement (20). The prepared standard solutions were measured in a UV-VIS instrument.

RESULTS AND DISCUSSION

Scanning electron microscope (SEM)

A scanning electron microscope creates a high-resolution image by scanning the surface of a material with a concentrated beam of electrons. SEM creates images that reveal information about the surface composition. The figure depicts the morphology of IIP for aluminum before and after washing. The fig reveals obvious aluminum holes in sizes eliminated by soxhlet extraction.as Shown in Fig.1. The holes formed as a result of removing aluminum ions gave very small spherically shaped with small sizes around

(0.421-0.758) Mm which have relationship with capacitance calculation

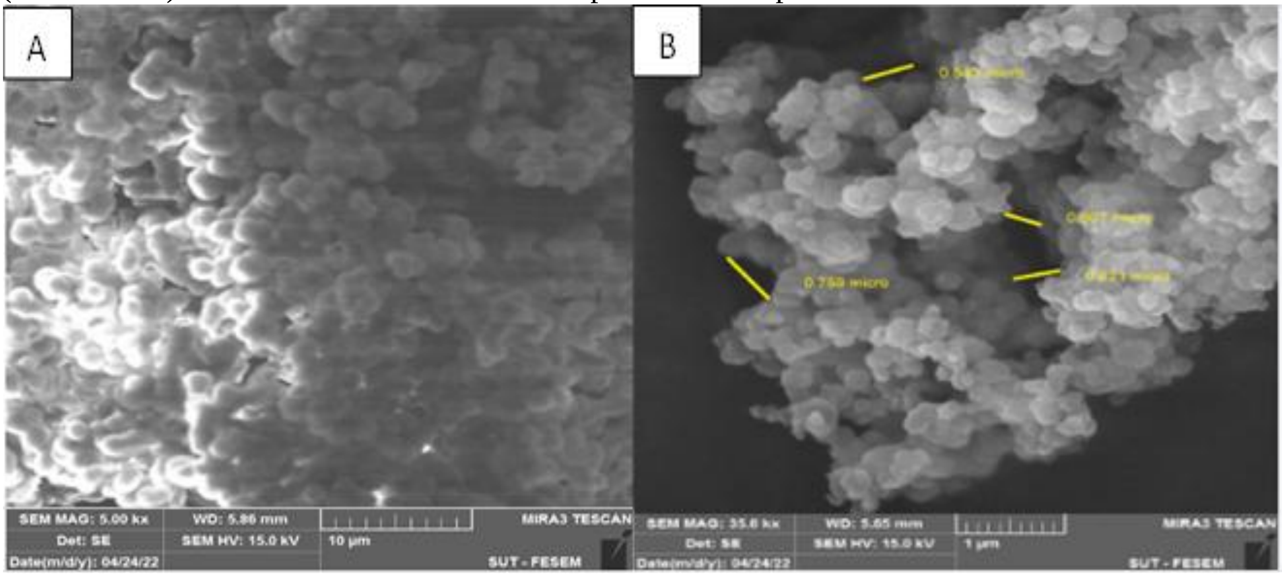


Figure 1. SEM photograph of the surface of Al-IIP (ally bromide), (A) before Aluminum removal, (B) after aluminum removal

Table 1. Contrast ratios between templet, monomer, crooslinker the preparation of AL –IIP

		Salt $AlCl_3.6H_2O$	Monomer (allyl bromide)	Cross linker EGDMA	Initiator Benzoyl peroxide	Solvent	Result
IIP	%	6.949	13.899	79.150	0.3	6ml	White
	mmole	1.8	3.6	20.5	0.32	CH_3OH	
IIP	%	4.961	14.885	80.152	0.3	6ml	White
	mmole	1.3	3.9	21	0.32	CH_3OH	
IIP	%	4.05	16.177	79.772	0.3	6ml	White
	mmole	0.999	3.99	19.675	0.32	CH_3OH	
NIP	%	------	16.177	79.772	0.3	6ml	White
	mmole	------	3.99	19.675	0.32	CH_3OH	

Isotherms of Adsorption

Several elements influence the relationship between whole capacity and cavity in IIP. The permanent holes that occur during the polymerization process after drying generate holes of varying sizes depending on the solvent quality. Particles with holes of various sizes and shapes are generated and distributed in IIP, which frequently sieved to obtain a narrower range. Because print molecules are embedded in the polymer, extracting the template and allowing rebinding is challenging unless the particles are tiny**.** The following equation was used to compute Q value.

$$Q = [(Ci – Cf) Vs] / Wmip \quad ...1$$

Ci = initial concentration of templet (µmole / mL)

Cf = final concentration of templet (µmole / mL)

Vs = volume of solution tested (mL)

W mip=is the mass of adsorbent in the mixture

Concentration of Al^{3+}ion	Absorption
0.1	0.0076
0.2	0.0246
0.5	0.0665
1	0.12
1.5	0.1694
2	0.2288
2.5	0.2918
3	0.3384

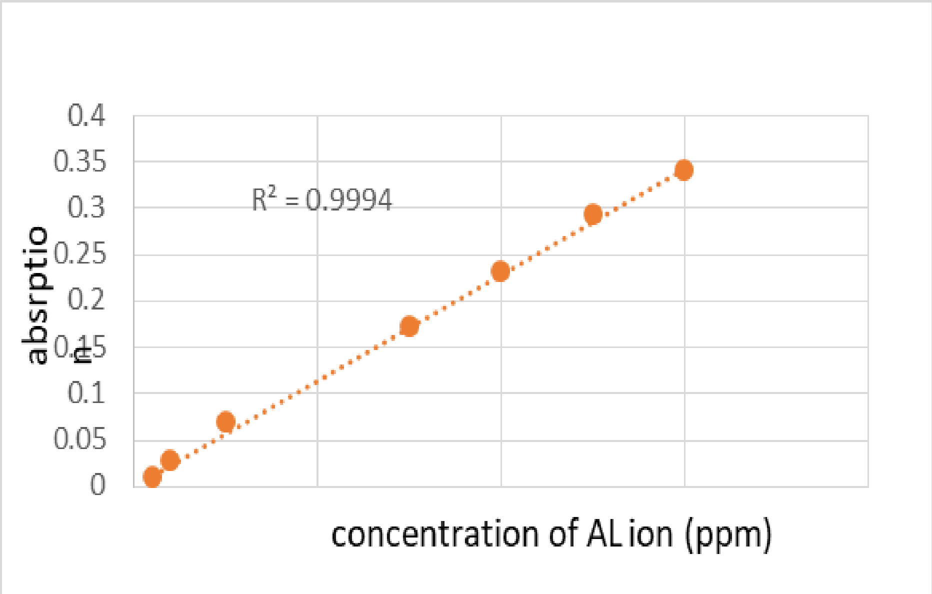


Figure 2. Calibration curve between concentration of aluminum ion standard and absorptions in UV- VIS spectrophotometer

The amount of solutions produced of $AlCl_2.6H2O$ by the isothermal process was measured using an UV-Visible technique and compared to atomic absorption technique. Adsorption values realization of Al-IIPs and the effect of initial aluminum ion concentration A range of (0.00041) to (0.01242) mol/ml was studied on the adsorption capacity. As shown the adsorption capacities of Al-IIP in the table.2 in UV-Visible spectrophotometer and table.3 in atomic absorption spectrophotometer.

Table 2. The optimal synthesis conditions for the ionic imprinted polymer for Aluminum developed in this study used UV- VIS spectrophotometer

Al-IIP (allyl bromide)					
Mass of IIP mg	**Ci ppm**	**C_i µM**	**C_{free} µM**	**Q µMole/g**	**Q_{free} mL/g**
0.2	0.1	0.00041	0.00028	0.004	13.780
	0.2	0.00082	0.00015	0.020	134.437
	0.5	0.00207	0.00039	0.050	129.230
	1	0.00414	0.00171	0.036	21.240
	1.5	0.00621	0.00194	0.064	32.899
	2	0.00828	0.00297	0.079	26.704
	2.5	0.01035	0.00492	0.081	16.554
	3	0.01242	0.00715	0.079	11.061

Table 3. The optimal synthesis conditions for the ionic imprinted polymer for aluminum developed in this study used A.A.S

Al- IIP(allyl bromide)					
Mass of IIP mg	**Ci ppm**	**C_i µM**	**C_{free} µM**	**Q µMole/g**	**Q_{free} mL/g**
0.2	0.1ppm	0.00041	0.00005	0.011	222.448
	0.2ppm	0.00082	0.00015	0.020	135.333
	0.5ppm	0.00207	0.00025	0.054	212.010
	1ppm	0.00414	0.00210	0.061	29.035
	1.5ppm	0.00621	0.00245	0.056	22.904
	2ppm	0.00828	0.00206	0.093	45.045
	2.5ppm	0.01035	0.00394	0.096	24.347
	3ppm	0.01242	0.00589	0.098	16.640

Langmuir isotherm models were used to calculate aluminum-IIPs maximal adsorption capacity in UV-Visible technique and atomic absorption technique.the adsorption capacity increases sharply initially and gradually increasing with the increase in the concentration of aluminum ion. as shown in Fig.3 and Fig.4.

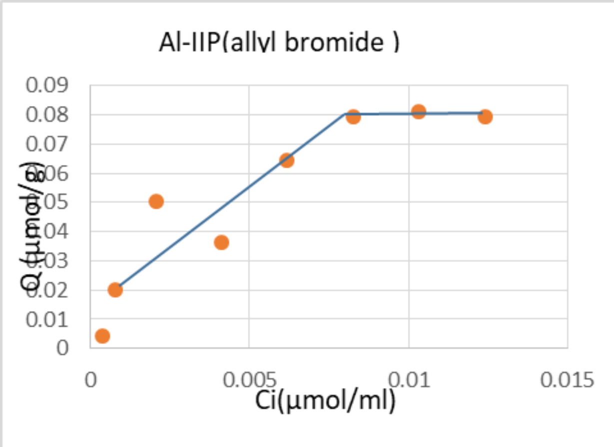


Figure 3. Langmuir isotherm model in UV-Visible spectrophotometer

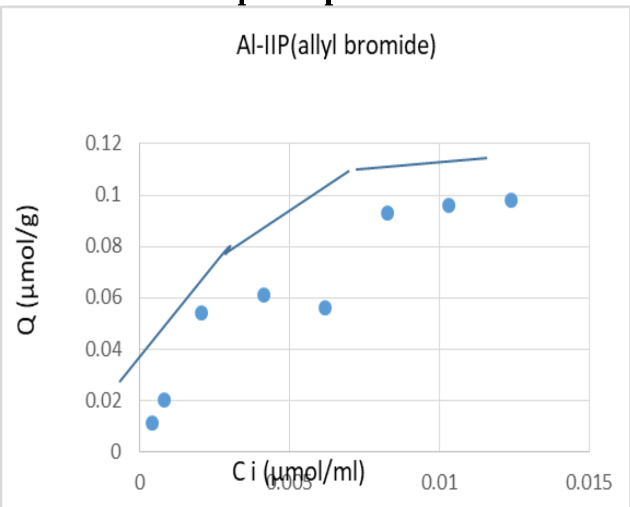
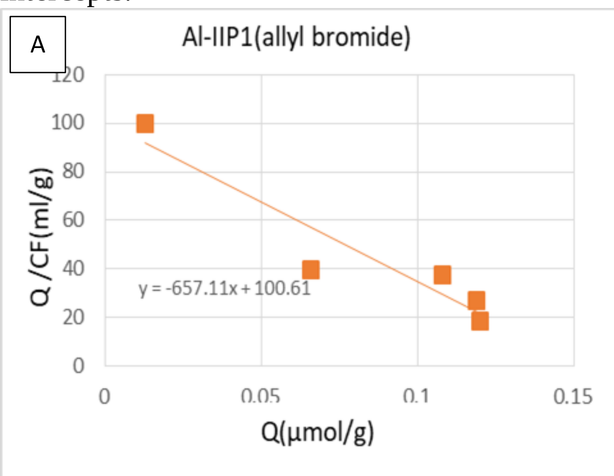


Figure 4. Langmuir isotherm model in atomic absorption spectrophotometer

As Shown in Fig.5 in UV-Visible spectrophotometer technique and atomic absorption spectrophotometer tsechnique. linear plot of Q/ C free. Q, the equilibrium dissociation constant was estimated from the slopes, and the apparent maximum number of binding sites was derived from the y-intercepts.



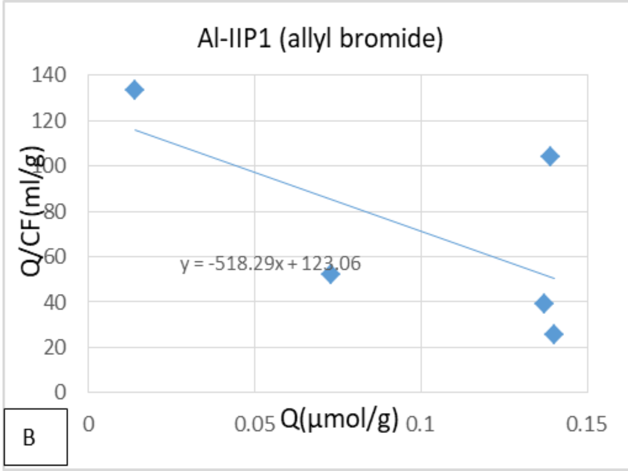


Figure 5. The relation between capacity Q (µmol/g) and Q/Cf (ml/g) in UV-Visible spectrophotometer Technique (A) and atomic absorption spectrophotometer Technique (B)

Slop=-1/kd ……2

-657.11=-1/ kd =0.00152

Intercept=Q max/kd ……3

100.61=Q max/0.00152

Q max=0.1531µmol/g

Atomic Absorption spectrophotometer (AAS): Standard solutions containing 0.1, 0.2, 0.5, 0.1, 1.5, 2, 2.5 and 3 ppm were prepared and measured in an atomic absorption at wave length 309.3nm

Slop Slop=-1/kd ……2

-518.29=-1/ kd =0.00192

Intercept=Q max/kd ……3

123.06=Q max/0.00192

Q max=0.2362 µmol/g

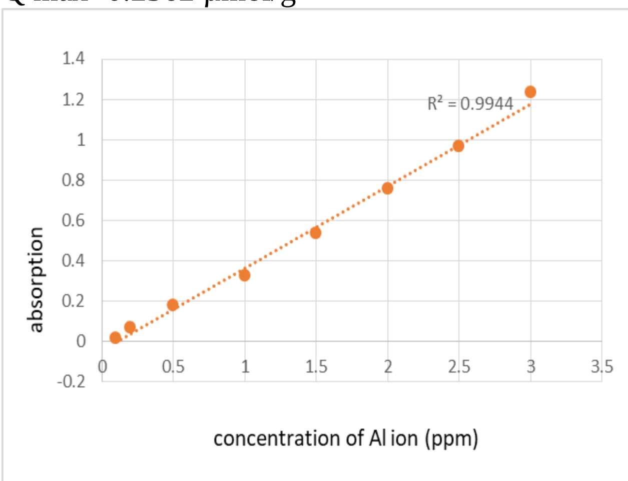


Figure 6. Calibration curve between concentration of aluminum ion standard and absorptions in atomic absorption spectrophotometer Technique

Industrial water was taken and filtered. Then these solutions were introduced into the Al-

IIP-SPE packed column system in the same manner as described previously. The obtained results as well as the recovery tests are shown in Table.4 and Table.5.

Table 4. Application measurement results for water sample using UV-VIS spectrophotometer

Water source	Absorption Mean	RSD% =(δn-1/Mean) *100 Precision	Rec. % =(practical value/True value)*100 Accuracy	RE% = 100-Rec
West water of al-Dura oil refinery	0.0253	0.395	102.84	-2.84
Power station fuel water	0.213	0.469	93.42	6.58
Turbine water from power station	0.063	0.158	94.88	5.12

Table 5. Application measurement results for water sample using AAS spectrophotometer

Water source	Absorption Mean	RSD% =(δn-1/Mean) *100 Precision	Rec. % =(practical value/True value)*100 Accuracy	RE% = 100-Rec
West water of al-Dura oil refinery	0.725	0.137	96.28	3.72
Power station fuel water	0.551	0.181	103.76	-3.76
Turbine water from power station	0.312	0.320	97.19	2.81

Table 6. compare the capacity between two methods analytical technique by using atomic Absorption and the proposed method IIP determination of Al ion in water source

Water source	Capacity Q µmol/g for atomic technique	Capacity Q µmol/g for uv-visible
West water of al-Dura oil refinery	0.080	0.007
Power station fuel water	0.112	0.096
Turbine water from power station	0.023	0.014

* According to the above table, the comparison between two methods analytical techniques using atomic absorption and the comparison method IIP by UV for Al^{3+}ion , there was no significant difference between two methods.Evidence of the method's efficiency and reliability in the analysis and estimation of the elements.

CONCUSION

Novel aluminum –IIP were prepared by bulk polymerization with allyl bromide selected as a functional monomer and EGDMA as a cross linker. Additionally, Benzyl peroxide was used as an initiator in the presence of chloroform solvent. The optimal molar ratios of Al (III) ion to monomer and cross linker dosage were studied. SEM made apparent the irregular shapes and three-dimensional network structure of polymers. The results of FT-IR proved the successful elution of Al (III) ion by CH_3OH/CH_3COOH (60:10 v/v) solution. Adsorption followed Langmuir isotherm models. The elution process has almost no influence on cavity structure and chemical property of the polymer, indicating that aluminum -IIP have excellent stability and regeneration capabilities.

CONFLICT OF INTEREST

The authors have declared no conflict of interest.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

DECLARATION OF FUND

The authors declare that they have not received a fund.

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