# Synthesis, Characterization of polyacrylamide as chelating agent for some metal ions

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## *Abstract*

A polyacrylamide was prepared by applying the solution technique used for synthesis of organic polymer. It was characterized by different techniques such as elemental analysis , Infrared spectroscopy and thermal analysis .The extracting efficiency of the resin was studied toward several metal ions from their diluted solutions (50ppm) by atomic absorption spectrometry .The effect of pH and treatment time on the chelating efficiency of the resin was examined and the optimum conditions were found, the chelating efficiency of the resin decrease with pH less than 1.5 and from 1.5 to 7.5 above that the resin became soluble. The loading capacities of the resin for $Zn^{2+}$,$Cd^{2+}$,$Cu^{2+}$and $Pb^{2+}$ ions were found to be 1.40,1.0,0.62 and 0.42 mg ion/gm resin respectively. The recovery efficiency of these ions was also studied as a function of treating time and the pH of the acidic eluent, and it was found to be in the sequence of $Zn^{2+}>Cd^{2+}>Cu^{2+}>Pb^{2+}$.

**KEYWORDS***:* Synthesis, characterization, ion exchange, polyacrylamide

## *Introduction*

Acrylamide is a chemical intermediate used in the production and synthesis of polyacrylamides[1].These high molecular weight polymers can be modified to develop nonionic ,anionic ,or cationic properties for specific uses, the largest use for polyacrylamide is in treating municipal drinking water [2].The polymer is also used to remove suspended solids from industrial waste water before discharge,reuse,or disposal.Polyacrylamide used for potable water should not contain more than 0.05% residual monomer [3].The polymer bind with particles and form heavy aggregates that rapidly settle out of solution leaving a clear supernatant [2]. Polyacrylamide is a soil stabilizer and also finds use in foundry operations to facilitate free sand flow into molds[4].Minor uses of polyacrylamide are as latex thickeners, emulsion stabilizers for printing inks, gelling agents for explosives, binders in adhesives and adhesive tape; in the production of diazo compounds ; and for gel chromatography and electrophoresis [5,6] , it is also used in gelatin capsules[7] .In this work, the polyacrylamide resin has been investigated for the analytical purpose. The polymer properties as a cationic exchanger resin and the chelating capacity of the resin towards several metal ions such as $Zn^{2+}$,$Cd^{2+}$,$Cu^{2+}$and $Pb^{2+}$ were studied .

## *Experimental*

### *Polymerization:*

Acrylamide monomer was obtained from Aldrich chemical company and was washed with (5%v/v)$H_2SO_4$ , (5%w/v)$Na_2CO_3$ and distilled water respectively.

Emulsion polymerization was used for preparation of the polymer by using 0.0382 gm ($2x10^{-4}$ Mole ) of potassium thiosulphate ,0.0378 gm($1.4x10^{-4}$ Mole) of potassium persulphate as an initiator , 0.01 gm ($1.4x10^{-4}$ Mole)of acrylamide, 60 gm of water and 0.5 gm ($1.6x10^{-3}$ Mole) Cetyltrimethyl ammoniumbromide(CTAMB) as emulsifier.The resultant polymer was washed several times with methanol and water, and then dried to constant weight at 25$^{o}$C under vacuum.

## *Instruments:*

A Shimadzo model 630-12 atomic absorption spectrophotometer (AAS) equipped with air-acetylene burner was used for concentration measurements, according to standard analytical procedure [8].IR spectrum of a polymer as a Nujol mulls between KBr plates was recorded from 4000 to 650 $cm^{-1}$ using a Pye Unicam SP-300 IR spectrophotometer. Thermogravimetric Analyser (TGA) Du-Pont Thermo Balance model 951was used .The instrument was calibrated with calcium oxalate standard ,all the measurements were carried out under nitrogen gas atmosphere as effluent. Carlo-Erba EA-1108 Elemental analyzer was used for the CHN analysis .The number average molecular weight (Mn) was determined by a viscosity method, it has been found that the polymer has Mn of 9503**.**

## *Chelating Capacity of the Resin:*

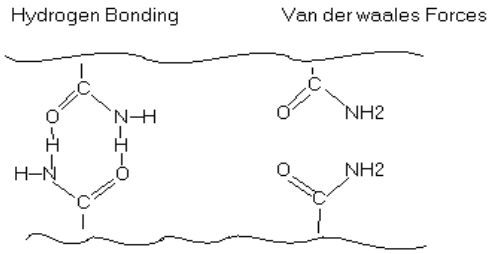
0.02 gm of the resin was shaking for (2,4,6,8 and 24 hr.) with15 ml solution of the metal ion of a known concentration (50 ppm of $Zn^{2+}$,$Cd^{2+}$,$Cu^{2+}$and $Pb^{2+}$) which was diluted from the stock solutions(1000)ppm which supplied by Merck as metal nitrate. All these diluted samples were run at different pH values 1.5, 2.5,3.5,4.5,5.5,6.5 and 7.5( by using 1M NaOH and 1M HCl). After being shaken for different times, the filtrate was determined by (AAS) using standard calibration graph for each examined metal ion.

## *Regeneration efficiency*

The regeneration efficiency of the loaded resin with different metal ions was examined by treating the loaded resin with 5M *HCl* as an eluent for different period of time ,then the concentration of the metal ion in the eluent was determined after treatment by atomic absorption , the resin shows a high recovery for metal ions[9,10,11].

## *Results and discussion*

The chains of polymer are usually crosslinked either chemically or physically .In the chemically crosslinked ; the polymer chains are connected by covalent bonds ,thus it is difficult to change the shape of such polymer ,while polymer chains which are physically entangled are connected through non covalent bonds i.e.




### *Identification ofthe polymer:*

The IR spectrum of the prepared polymer displays bands characteristic of the $-NH_2$ band for its monomer. Some other assignment is the band $-CH_2$ stretching bands at 2900 $cm^{-1}$,$-CH_2$ bending at 1400$cm^{-1}$.The strong band at 1650$cm^{-1}$ due to C=O stretching band (Fig.1,Table1).

**Table(1):The functional groups of infrared spectrum of the resin.**

| Band frequency($cm^{-1}$) | Band intensity | Mode of vibration | Functional groups |
|---|---|---|---|
| 3400-3200 | Strong | Stretch | $-NH_2$(amide) |
| 2900 | Strong | Stretch | $-CH_2$(aliphatic) |
| 1650 | Strong | Stretch | -C=O(carbonyl) |
| 1400 | Medium | Bend | $-CH_2$(aliphatic) |
| 1320 | Medium | Bend | -C-N |

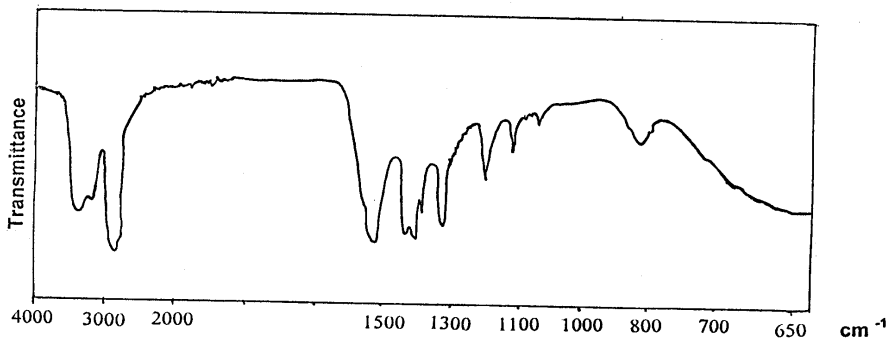



Fig.1:The I.R. spectrum of the resin

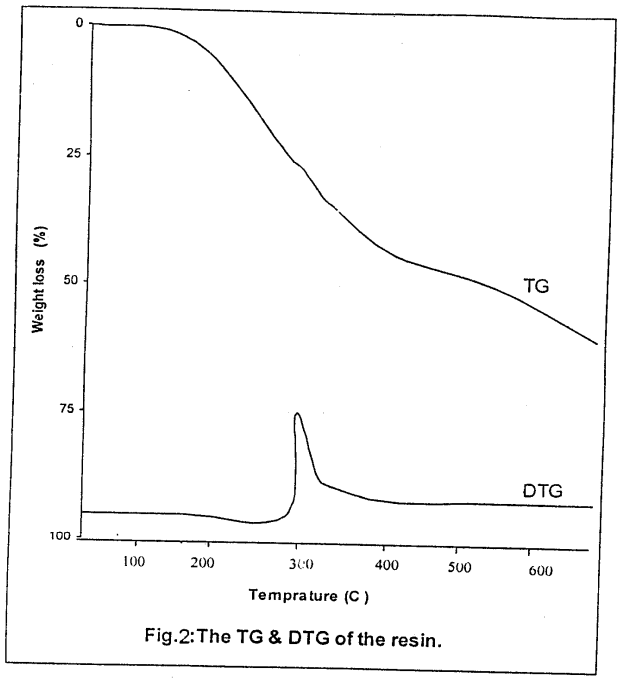



Fig.2:The TG & DTG of the resin.

## ***Elementary analysis:***

The elemental analysis of the polyacrylamide(Table2) shows that its contents consist of C=50.50%, N=10.71% and H=6.89% which coincides with the theoretical calculated values for C=50.56%,N=10.79% and H=6.99% respectively.

**Table (2): The elemental analysis of the resin.**

| | **Calculated (%)** | **Found (%)** |
|---|---|---|
| **C** | 50.56 | 50.50 |
| **H** | 6.99 | 6.89 |
| **N** | 10.79 | 10.71 |

## ***Thermogravimetric analysis*:**

Thermogravimetric analysis (TGA) and differential thermogravimetric analysis(DTGA) techniques were used in this study for the evaluation of the thermal stability of the resin .

Fig.(2) shows the TG and DTG curves for a known amount of the resin which indicates clearly that the resin was decomposed in one step at a temperature about 300$^{o}$C. This may be due to the loss of $-NH_2$ group which also indicates the thermal stability of the resin. The repeating units of polyacrylamide contain hetroatoms like oxygen and nitrogen in the chains posses high thermal stability due to great rigidity and intermolecular forces .

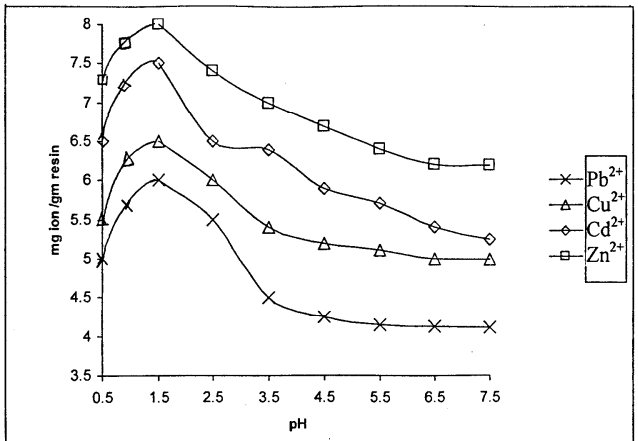



**Fig.(3):Effect of pH on loading capacity of the resin at treatment time (24 hr.)**

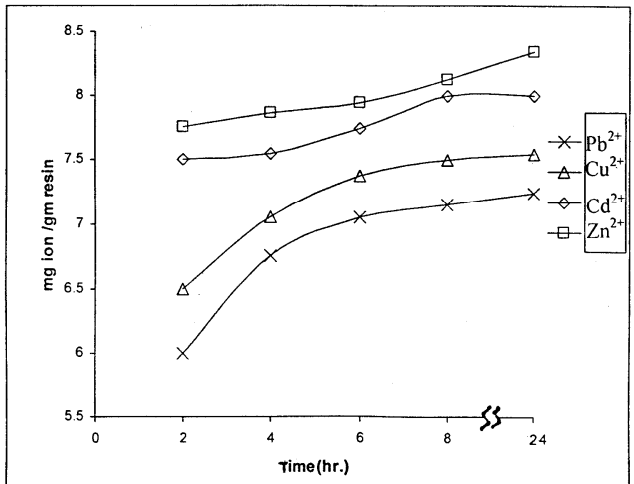



**Fig.(4): Variation of loading capacity of the resin as a function of time at pH 1.5 for the four ions.**

## ***Effect of pH and treatment time on the loading capacity:***

The polyacrylamide resin expected to show a cationic exchanger toward some metal ions .On the other hand it is necessary to take into consideration sorption by the formation of complexes with metal ions by the batch process [12-14].The optimum conditions regarding pH and treatment time for the extraction of these metal ions were obtained, since the effect of pH on the total loading capacity of the resin at treatment time (24 hrs.)are shown in Fig. 3 .

The variation of the loading capacity of the resin with the treatment time at pH 1.5 for the metal ions was determined(Fig.4) which shows that the resin reached its maximum affinity for four tested metal ions at 6hrs.The results obtained in this work showed that the resin has a remarkable capability of chelating toward the metal ions studied, and the chelating efficiency was in the following order $Zn^{2+}>Cd^{2+}>Cu^{2+}>Pb^{2+}$as shown in Table3.

**Table (3): The chelating efficiency data of the resin at pH 1.5 .**

| Metal ion | Loading capacity (mg ion/gm resin) | Efficiency (%) | Regeneration (%) |
|---|---|---|---|
| $Pb^{2+}$ | 0.42 | 28 | 30 |
| $Cu^{2+}$ | 0.62 | 40 | 70 |
| $Cd^{2+}$ | 1.0 | 58 | 80 |
| $Zn^{2+}$ | 1.4 | 80 | 100 |

## *Conclusion*

Polyacrylamide resin shows a high capacity of zinc, cadmium, copper and lead from hydrochloric acid solution and the sorption was found to be dependent on the time and pH. The recovery efficiency of these ions was found to be in sequence of $Zn^{2+}>Cd^{2+}>Cu^{2+}>Pb^{2+}$.

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***المستخلص***

في الدراسة الحالية تم تحضير راتنج البولي اكريل امايد بطريقة تحضير البوليمرات العضوية ،تم تشخيص هذا الراتنج باستخدام تقنيات التحليل الحراري،التحليل العنصري ومطيافية الأشعة تحت الحمراء . تم دراسة كفاءة الراتنج في استبدال عدد من الايونات المعدنية من محاليلها المخففة (50 جزء بالمليون )بإتباع تقنية الامتصاص الذري و دراسة تأثير كل من الحامضية و زمن المعاملة على كفاءة الراتنج التبادلية حيث وجد أن الكفاءة الاستبدالية للراتنج تجاه الايونات المدروسة كالأتي:الزنك> الكادميوم> النحاس>الرصاص إذ تم حساب سعة تحمل الراتنج لكل ايون فكانت 1.4،1.0،0.62،0.42 ملغم ايون /غرام من الراتنج على التوالي و بذلك فان الراتنج أبدى كفاءة عالية في الاستبدال تجاه تلك الايونات، حيث إن هذه الكفاءة تعتبر دالة مع الزمن و الدالة الحامضية من ناحية أخرى درست كفاءة استعادة الايونات من الراتنج بمعاملتها مع محلول حامض الهيدروكلوريك المخفف وقد وجد أن استعادة هذه الايونات حسب الترتيب الآتي: الزنك> الكادميوم> النحاس>الرصاص .