

## **SEPARATION OF YTTRIUM Y(III) FROM RUBIDIUM RB(I) USING POLY HYDROXAMIC ACID**

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**Abstract:** The polyhydroxamic acid PHA performed better when used as a simulation mode for the single-step separation of <sup>86</sup>Y from Rb. While Polyacrylamide (PAAm) was modified with hydroxylamine to create (PHA) resin. Acrylamide monomers were polymerized using a  $\gamma$ - ray method to create polyacrylamide. PHA was utilized for the way to simulate obtaining <sup>86</sup>, <sup>87</sup>, and <sup>88</sup>Y from Rb. In relationship to pH, the yttrium and rubidium adsorption behaviors on the produced PHA under various environments, including, acetate and citrate buffer solutions as well as HCl, have been investigated. In the present investigation, we found that Y(III) and Rb(I) were separated using PAH resin at pH 5.5, where Rb passed through the column while Y was retained. Y was totally eluted using 2 M HCl.

**Keywords:** PHA Resin, Polyacrylamide, Gamma Rays, Separation, Y, Rb.

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## فصل اليتريوم Y(III) من الروبيديوم Rb(I) باستخدام حمض البولي هيدروكساميك

الملخص: أظهر حمض البولي هيدروكساميك PHA أداءً أفضل عند استخدامه كنمط محاكاة للفصل نظير Y86 من Rb. بينما تم تعديل بولي أكريلاميد (PAAm) باستخدام هيدروكسيل أمين لإنشاء راتينج (PHA). تم بلمرة مونومرات الأكريلاميد باستخدام طريقة أشعة جاما لإنشاء بولي أكريلاميد. تم استخدام PHA لطريقة محاكاة لإنتاج نظائر Y 88,87,86 من Rb. تم دراسة سلوكيات امتصاص اليتريوم والروبيديوم على PHA المنتج في بيئات مختلفة حسب قيمة pH، بما في ذلك المحاليل المنظمة كالأسيئات والسترات بالإضافة إلى حمض الهيدروكلوريك. وجد أن Y (III) و Rb (I) تم فصلهما باستخدام راتينج PAH عند درجة حموضة 5.5، حيث مر Rb عبر العمود بينما تم الاحتفاظ بـ Y. تم غسل Y تمامًا باستخدام 2 مولار حمض الهيدروكلوريك.

الكلمات المفتاحية: راتنج PHA، بولي أكريلاميد، أشعة جاما، فصل، Y، Rb.

## 1. Introduction

The labeled molecules with the indexes of  $^{86g,87g,88g}Y$  isotopes have been used in medical diagnostics procedure, tumor therapy, biological tracers or detector calibration. The extraction of radioactive substances from their surrounding substrate constitutes one of the fundamental activities performed in radiochemical labs.  $\beta^+$  emitters radionuclides with medicinal applications were created (Qaim, 2011).  $\beta^+$  emitters,  $^{86}Y$  ( $T_{1/2} = 14.7$  h), have been made in one of two ways: either by irradiating strontium with proton or by irradiating rubidium (Vértes et al., 2003) with  $^3He$  (Abdollah Khorshidi, 2023). For the radiochemical separation of yttrium from rubidium in earlier study, solvent extraction and ion-exchange chromatography were utilized. (Agarwal et al., 2003; Garmestani et al., 2002; Mitra, A. et al., 2021; S. Kandil et al., 2009; S. A. Kandil et al., 2007; Ketterm et al., 2002; Pal et al., 2006; Abdollah Khorshidi, 2023). For the purpose of this work, PAAm was modified to generate (PHA) resin, which was then employed as an emulator approach to separate  $^{86, 87, \text{ and } 88} Y$  from its parent Rb.

Since more than 40 years ago, hydroxamic acid (HA) groups were discovered in chelating ion-exchange resins, and numerous analytical uses for these polymers have been researched. Numerous heavy metal ions derivatives and the HA groups are known to produce stable chelates. (Cao et al., 2020; Hosseini, 2011; Johann et al., 2019; Li & Yamamoto, 2013; Mzinyane et al., 2021; Singha & Pal, 2020). HA, with the general formula R-CO-NHOH, is regarded as the compounds that result from the hydrogen that is bonded to nitrogen in the HA molecules.

By polymerizing poly(ethylacrylate-divinylbenzene) and then reacting with  $NH_2OH$ , (PHA) resins were created (Lee & Hong, 1994). Using copoly(acrylamide-divinylbenzene) and  $NH_2OH$  hydrochloride under the influence of KOH, as well as acrylhydroxamic acid and divinylbenzene and free radical solution polymerization, (PHA) chelating ion-exchange resins were created. For the sorption of the ions  $Fe^{3+}$ ,  $Cu^{2+}$ ,  $Co^{2+}$ , and  $Pb^{2+}$ , the generated resins were applied ( Khodadadi, R. et al., 1995). By reacting ethylacrylate/acrilonitrile/divinylbenzene copolymer with  $NH_2OH$  under the influence of sodium exthoxylate, Using amidoxime and hydroxamic groups, a novel ion-exchanger was developed (Lee et al., 2001).

Ethylacrylate and divinylbenzene have been utilized to create (PHA) resin  $Cu^{2+}$  and  $Fe^{3+}$  metal ions were complexed with the ready resins. A coordination complex was created by the ligand between a metal ion and an oxygen atom from a carbonyl group (Neagu et al., 2003). Through modification of (PAAm), which was originally produced by employing  $\gamma$  rays as an initiator to polymerize acrylamide monomers, The PHA has been created.  $NH_2OH$  is used to treat (PAAm) and change the amide group into a hydroxamic group.  $Y^{3+}$  and  $Sr^{2+}$  were isolated from  $Zr^{6+}$  using (PHA) resin (Hassan et al., 2011). Sago starch that had been modified with poly(methylacrylate) was utilized to create a new polymer with HA functional group using hydroxylamine in an alkaline solution (Lutfor et al., 2001)

## **2. Experimental**

### **2.1. Reagents and chemicals.**

Acrylamide was purchased from Merck for this investigation (Darmstadt, Germany). The crosslinking agent employed was N,N-methylene-bis-acrylamide (NMBA), which was purchased from Sigma-Aldrich (St. Louis, MO, USA). To change the pH of the medium, sodium hydroxide and hydrochloric acid were purchased from Merck, hydroxylamine hydrochloride from Fluka (Buchs, Switzerland).  $Y_2O_3$  99.97% from Cambridge, England's Koch-Light Laboratories Ltd; RbCl 98% (from Aldrich).

### **2.2. Instrumentation.**

With the aid of the ULTIMA2 ICP instrument from Jobin Yvon S. A., France, the composition of the separated material was examined using inductively coupled plasma optical emission spectrometry (ICP-OES).

### **2.3. Gamma Cell**

The irradiation source was a  $^{60}Co$  gamma cell of the Russian type MC-20, with a dosage rate of 2 kGy  $h^{-1}$ , it contains two 5-liter chambers.

### **2.4. FT-IR Analysis.**

The FT-IR spectrometer (Bomen, Hartman & Borunz spectrometer, Model MB 157) was used to collect the infrared spectra of polymer samples that contained KBr pellets.

### **2.5. SEM**

The morphology of the resin was determined by scanning electron microscopy (SEM) (Mira3, Tescan, Brno, Czech Republic).

### **2.6. Preparation of (PHA).**

When acrylamide monomers in an aqueous solution are polymerized by radiation, NMBA is used as an agent to crosslink the monomers, resulting in crosslinked polyacrylamide (PAAm) (Park et al., 2004). A solution of 10 g acrylamide and 1 g NMBA was prepared using 100 cc of distilled water. After putting the mixture into glass ampoules, air was removed from the ampoules using nitrogen gas. Before being exposed to  $^{60}Co$  radiation at a dosage rate of 2 kGy  $h^{-1}$ , the ampoules were sealed. The fragmented irradiation crosslinked polymers were then cleaned with acetone and water to remove any lingering monomers, dried, and stored. The conversion percentage, which was calculated by gravimetric analysis and was found to be roughly 90%, was determined.

PHA in hydrogen form was created by adding a determined amount of sodium hydroxide and hydroxylamine hydrochloride solutions to a slurry of dry crosslinked PAAm (Siyam, 2001). After that, the mixture was agitated at room temperature for five minutes. After six hours at 70 °C, the response was maintained. H<sup>+</sup> was released as a byproduct of the process. Following a thorough wet, the generated (PHA) was filtered out of the combination. Next, for a minimum of five minutes, the resin was immersed in a 3 M HCl solution. The resin underwent filtration and many water washes to remove any remaining chlorine, and it was then dried at 50 °C until its weight remained consistent.

### 2.7. Experiment in batches.

The distribution coefficient must be determined to distinguish between Y and Rb. A stock solution of 5000 mg/L Y and Rb was prepared. The mixture was heated up until almost dry, and the remaining material was dissolved in 100 cc of double-distilled water.

50 mg of sorbent and 100 µl of stock solution were combined. Then, 4.9 ml of various HCl concentrations or an alternate medium, like 0.1 M citrate buffer or 0.1 M acetate buffer at various pH were supplied in 100mm x 15 mm ampoule. The ingredients were stirred to reach equilibrium for two hours. Using the following equation, the distribution coefficient (K<sub>d</sub>) was determined (Saraydin et al., 2001) .

$$K_d = \frac{C_{ads}}{C_{unads}} \times \frac{v}{m}$$

where v is the volume of the aqueous phase in milliliters, m is the amount of the resin in grams, C<sub>ads</sub> is the concentration that has been sorbed on the resin, and C<sub>unads</sub> is the concentration that is still in solution.

To measure the quantities of yttrium and rubidium in each batch experiment, ICP-OES will be used.

### 2.8. Cation-Exchange Column Chromatography.

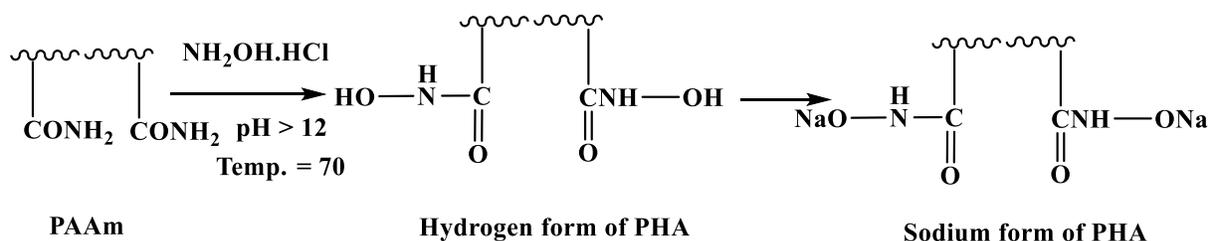
This work involved a thorough investigation of the separation of Y from Rb utilizing PHA resin and 2 M HCl. A 100 ml solution containing 100 ppm of Y and Rb dissolved in acetate buffer pH 5.5 was loaded onto a 22 cm long x 1.5 cm diameter column packed with PAH at a depth of 2 cm. Quartz wool was positioned at the top exchanger to prevent the adsorbent particles from being disturbed during solution addition and to regulate the flow of solutions through the column. A 1 ml stock sample was retained as a control. During this phase, load samples were collected to look for breakthrough. The load samples' ICP analysis confirmed that there was no Y column breakout. A further 50 cc of acetate buffer pH 5.5 was used to wash the column.

The resin was completely adsorbed by yttrium whereas rubidium passed through the column. To elute Y, 135 ml of 2 M HCl were used. Nine samples, totaling approximately 15 milliliters, were gathered and put through an ICP analysis. Below are the specifics of the separating process.

### 3. Results and Discussion

#### 3.1. The Resin's preparation.

PHA resin is created when sodium hydroxide and hydroxylamine hydrochloride combine with comparable crosslinked PAAm (Siyam 2001). The sodium form of PHA and hydrogen are produced in a hypothetical reaction between PAAm, sodium hydroxide, and hydroxylamine hydrochloride, as schematically shown in Scheme 1.

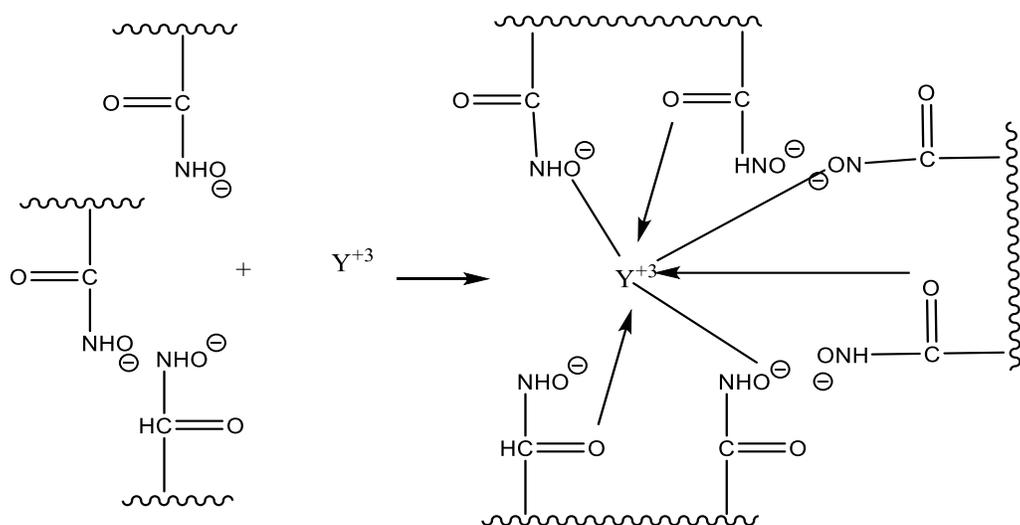


Sch. (1): Preparation of PHA from PAAm.

#### 3.2. Analysis using FT-IR.

The polyacrylamide (PAAm), polyhydroxamic acid (PHA), and metal-loaded PHA samples' infrared spectra are displayed in Figure 1. The crosslinked PAAm's infrared spectra exhibits absorption bands for carbonyl groups, amide (N-H), and CH<sub>2</sub> stretching at 3401, 2998, and 1649 cm<sup>-1</sup>, respectively. PHA resin exhibited discrete absorption bands at 3200, 3444, 1670, and 930 cm<sup>-1</sup>, corresponding to hydroxamic (O-H), amide (N-H), carbonyl, and (N-O) groups. The absorption band at 2862 cm<sup>-1</sup> is connected to the C=N group in hydroxamic acid's enol form. By comparing the spectra of the PHA sample and the metal ion loaded PHA sample, it is possible to see that the N-O band shifts in the metal ion loaded sample from 930 to 775 cm<sup>-1</sup> as a result of the interaction of homologous groups with metal ions.

The N-O group of the sorbent exhibits lower stretching frequency due to the stronger M-O bond, which is indicative of a significant interaction between metal ions and the N-O group. The O-M-O stretching vibrations' additional peaks, which are visible at a wavelength of about 700 cm<sup>-1</sup>, can also be identified. In Scheme 2, it is depicted how metal ions might interact with PHA polymers (Tompkins & Mayer, 1947)



Sch. (2): Yttrium ion interactions with PHA resin.

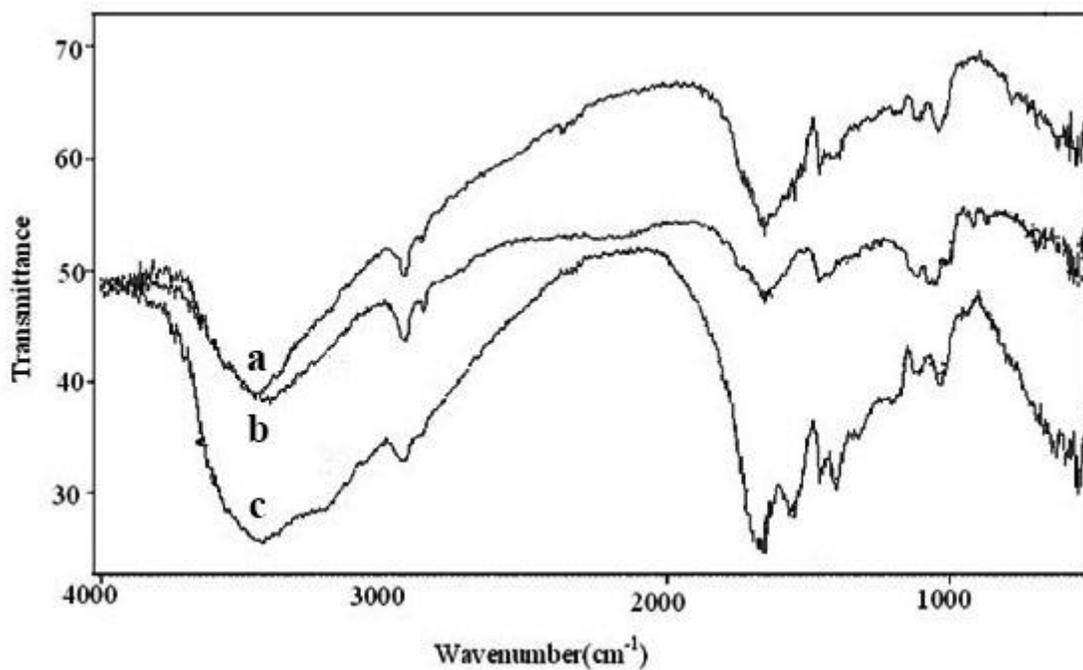
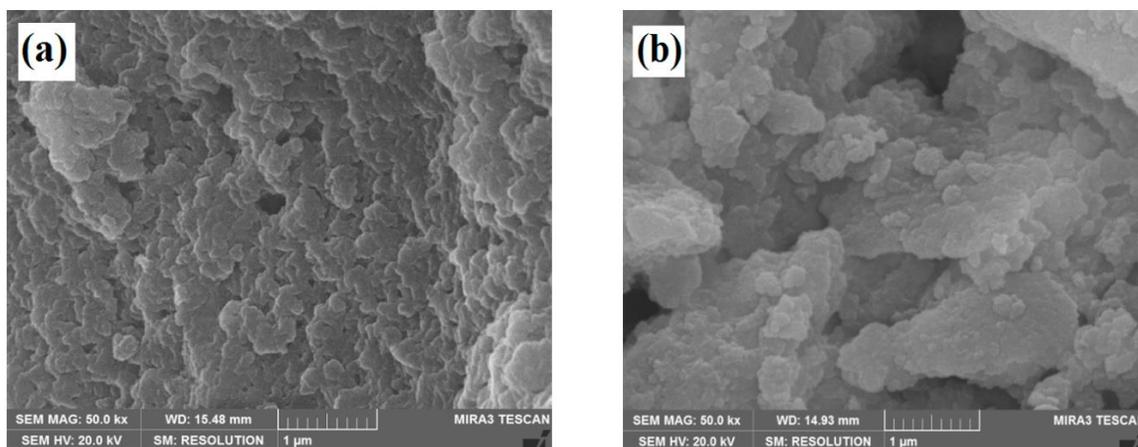


Fig. (1) : FTIR spectra of a) The polyacrylamide (PAAm), b) polyhydroxamic acid (PHA), and c) metal-loaded PHA

### 3.3. SEM

SEM analysis is a productive way to view morphology. Figure 2 shows the morphologies of the poly (hydroxamic acid) both before and after yttrium Y(III) ions were adsorbed. The presence of coarse pore structures and surface on the resin could have had a role in the adsorption of metal ions. The morphological surfaces changed after adsorption. The resin's porosity and structure altered, and its surface developed a metallic sheen. We reach the conclusion that yttrium Y(III) ions adsorbed on the resin's surface. The outcomes showed that the resin's porous structure is primarily mesoporous.



**Fig. (2):** SEM images of the resin before (a) and after (b) adsorption.

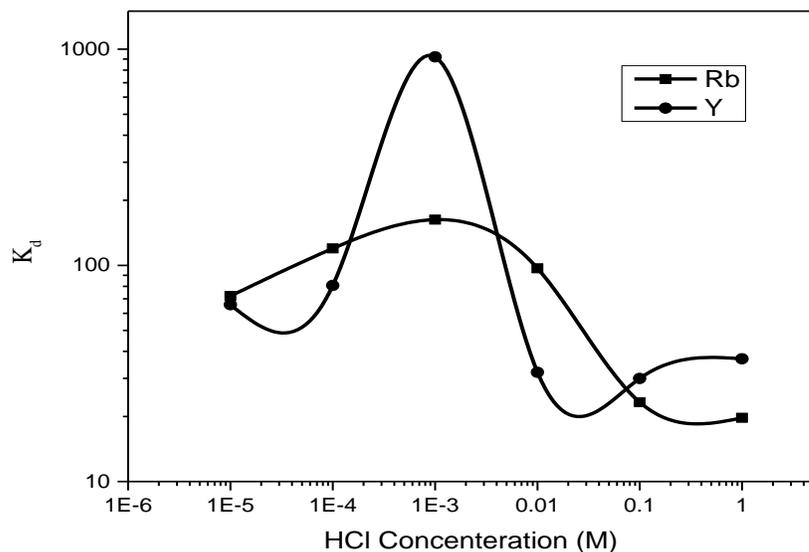
### **3.4 Behaviors of Y and Rb Adsorption on Cation Exchangers.**

Specifically HCl, acetate buffer, and citrate buffer were used to study the yttrium and rubidium adsorption behaviours on the synthetic PHA resin. However, batch mode research has examined the effects of equilibration time based on Y and Rb uptake, finding that equilibrium was attained after two hours of shake.

#### **3.4.1. Hydrochloric Acid.**

The effect of pH solution is a significant factor that affects the adsorbent's ability to bind metal ions (Yang, K.Y, et al.2020]. Figure 3 illustrates how pH affects resin adsorption for Y(III). Since yttrium tends to hydrolyze above pH 6, higher pH ranges were avoided for study.

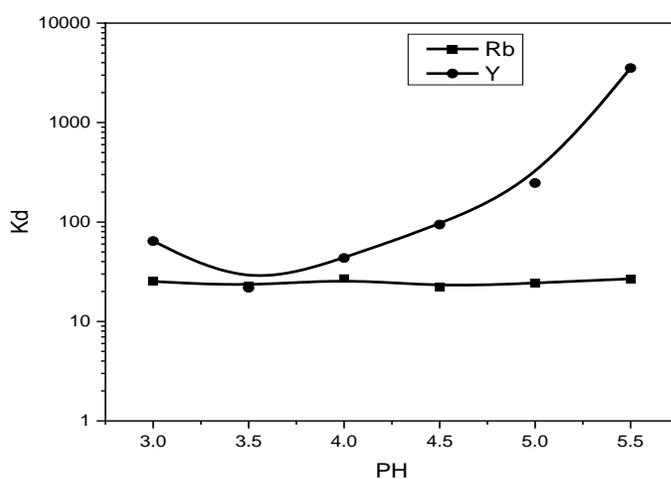
The  $K_d$ -values of Rb and Y increased from  $10^{-5}$  to  $10^{-3}$  mol/L, and then they decrease as acidity increased to 0.1 mol/L as a result of a change in PHA resin charge. This can be explained as follows: as pH increases, amide, carboxylic, and amidoxime groups dissociate more readily, creating more binding sites for the adsorption of metal ions. Low levels of metal ion adsorption were observed under more acidic concentration, or low pH values of roughly 2.0–3.0. This is explained by the competition between metal ions and  $H^+$  ions for the active sites. The surface layer of the adsorbent surface may shrink as a result of hydrogen bonds forming between its active sites. This surface layer acts as a barrier slowing down the adsorption of metal ions.



**Fig. (3) :** Y(III) and Rb(I) distribution coefficients on PHA resin as a function of HCl concentration, with a 2-hour shaking period.

### 3.4.2. 0.1M Acetate Buffer.

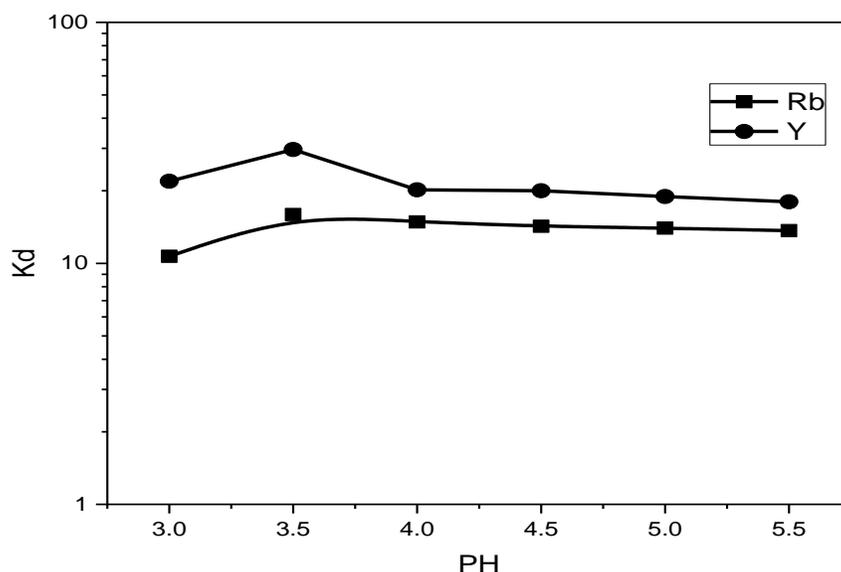
Figure (4) depicts the  $K_d$ -values of Rb and Y in 0.1 mol/L acetate buffer over the pH range of 3 to 5.5. As the pH value rises, the  $K_d$ -values of Y gradually rise as well. The  $K_d$ -values for Rb remain unchanged. Additionally, it has been found that Y is more adsorbable than Rb.



**Fig. (4):** Y(III) and Rb(I) distribution coefficients on PHA resin with a 2-hour shaking period as a function of pH in 0.1 M acetate buffers.

### 3.4.3. 0.1 M Citrate Buffer

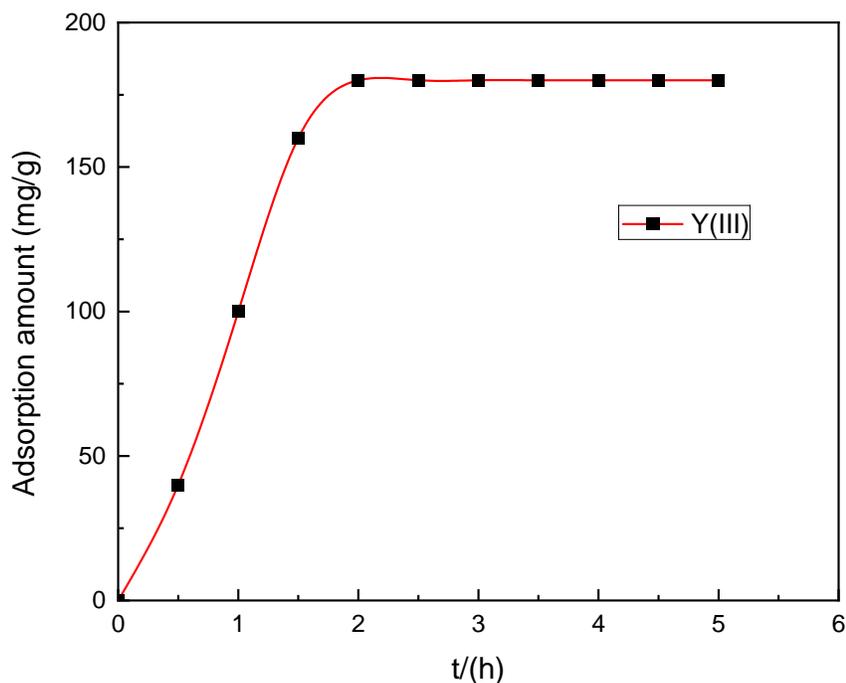
On the PHA increase, Y and Rb behaviors in 0.1 mol/L citrate buffer at pH values ranging from 3 to 5.5 were examined (Fig. 5). Their modest and largely stable adsorbabilities over the pH range of 3 to 5.5 were demonstrated by the findings.



**Fig. (5)** : Rb(I) and Y(III), distribution coefficients on PHA resin as a function of pH of 0.1 M citrate buffers, shaking period of two hours.

### 3.5. Effect of Contact Time

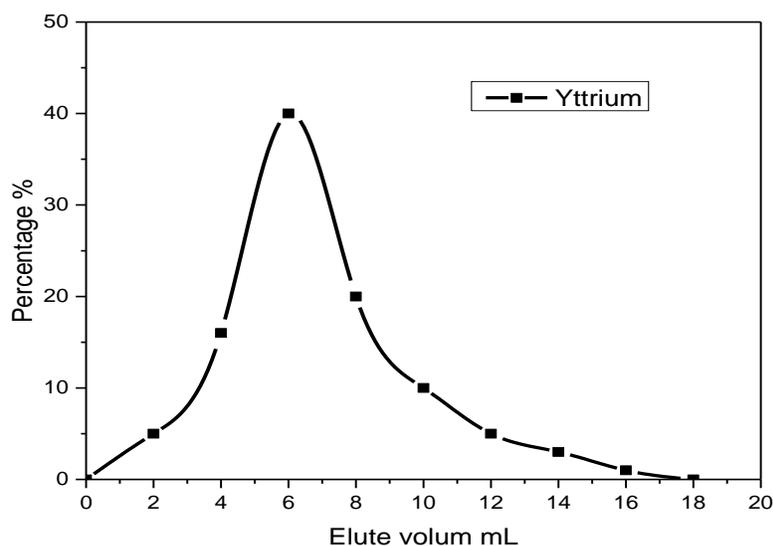
The effects of contact time on the adsorption of the (PHA) for Y (III) was investigated and the results are shown in Figure 6. It is clear that the adsorption capacity of Y (III) increased with increases in contact time, reached equilibrium after around two h, and remained steady the rest of the time. Remarkably, the amount of adsorption was obviously faster at the initial stage, potentially because, initially, the adsorbent site was vacant and the solute concentration gradient was high. Thus, it appeared that the adsorption capacity of Y (III) on the resin was a rapid process, with equilibrium reached in two hours (Li, C.X, et al.2015). Therefore, the contact time of three hours was considered appropriate in the following experiments.



**Fig. (6):** Effect of contact time on the adsorption (adsorption rate) of Y(III) metal ions onto (PHA) resin.

### 3.6. Rb(I) and Y(III) separation by PHA-Column Chromatography

The ideal conditions for separating Y from Rb were discovered in batch mode and applied in column mode. Practically, from all of the data on PHA in batch mode that were provided, these components showed a considerable variation that might help separate Y from Rb. Additionally, in column mode, the effective parameter is the flow rate correlated with time. However, 100 ml were supplied at a flow rate of 1.5 ml/min during the loading process. All of the yttrium during loading was adsorbed on the resin, while rubidium went through the column. This indicates that Rb has separated and that Y elution has begun. The elution profile for separating Y from the column was detailed in Fig (7). As previously indicated, a 100 mL stock solution containing 100 ppm of each Rb and Y diluted in 0.1 M acetate buffer pH 5.5 was put into a glass column that measured 22 cm long by 1.5 cm in diameter and 2 cm deep in PHA. Rb displayed low  $K_d$  in the 0.1 M acetate buffer at this pH. (3). Rb thus traversed the column with a percentage of 100% of the starting value. No Rb passed through the column after additional washing with 0.1 M acetate buffer pH 5.5, but yttrium was still present (Fig. 4; its  $K_d$ -values are fairly high). Y was completely eluted with 2mol/L HCl, and the full amount of yttrium was collected in 135 mL.



**Fig. (7):** Y(III) elution profile from a PHA resin-packed column. 15 milliliters is the fraction volume.

#### 4. Conclusion

In this paper, we reported that Poly (hydroxamic acid) PHA resin was prepared by modification of polyacrylamide (PAAm) using hydroxylamine. Polyacrylamide was prepared by polymerization of acrylamide monomers by gamma rays technique. PHA was used for the separation of Y(III) from Rb(I) as a simulation mode for the separation of  $^{86,87,88}\text{Y}$  from its parent (Rb). Based on our systematic characterizations and batch adsorption experimental results, the obtained polymer shows that Y(III) and Rb(I) were separated using PAH resin at pH 5.5, where Rb passed through the column while Y was retained. Y was totally eluted using 2 M HCl.

#### 5. Conflicts of interest

“There are no conflicts to declare”.

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