

# Removal of Pb (II) by using SA-G- Poly (Acryl Acid-Acrylic Amide)/TiO<sub>2</sub> Characterization and Kinetic Study

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## Abstract

We used free radical polymerizing (TiO<sub>2</sub> NPS) and sodium alginate (SA) with acrylic acid and acrylamide to make hydrogel nanocomposites. The initiator and crosslinking agents were KPS and MBA, respectively. TEMED was also used. SA-(g)-P(AAC-AAM)/TiO<sub>2</sub> nanocomposite is a super absorbent for pollutants. The mixture was used to extract Pb (II) from water. The nanocomposites' structure and morphology were studied using FTIR and field emission scanning electron microscopy (FESEM). These rates were used to investigate the kinetics of Pb (II) adsorption. The adsorption kinetics are pseudo-second-order. The hydrogel nanocomposite successfully adsorbs and removes Pb (II).

**Keyword:** TiO<sub>2</sub> nanoparticles, hydrogel Nano composites, adsorption, sodium alginate, Pb (II).

## 1. Introduction

Water is one of the most important necessities of life that all living organisms need, including [1]. Clean drinking water is important for health, but industrial development and the increase in human consumption of natural materials, as well as significant mining operations in recent times, caused the entry of undesirable substances into the water and thus the occurrence of water pollution [2-4]. The presence of heavy metals in wastewater such as (Pb, Cd, Ni, Cr) and others has a significant impact on human life due to its high toxicity [5]. For example, lead is associated with the components of a living cell and thus will interfere with its work [6]. The symptoms of lead poisoning are dizziness, severe fatigue, depression and others. Therefore, removing these pollutants from water was considered a challenge [7]. Many techniques have been used, such as ion exchange, electrodeposition, membrane filtration, and others to get rid of water pollution with heavy metals. Despite this, some defects were found in these techniques [8-12]. Adsorption is an ideal process to remove heavy metal ions from polluted water due to its rapid mobility and ability to remove metal ions as well as its potential for metal ion selection [13-16].

## Experimental

### Chemicals and materials

Sodium alginate (SA), Titanium Dioxide (TiO<sub>2</sub>), Acrylic acid (AAC), Acryl Amide (AAM), N, N'-Methylene-bis-acrylamide (MBA), NaOH, HCl, potassium persulfate (KPS), Solution of Pb (II). All compounds that were used of the highest analytical purity. Deionized water was used to prepare all of the solutions.

Synthesis of SA-g-P (AAC- MA)/TiO<sub>2</sub> Hydrogel Nano composite

SA-g-P(AAC-MA)/TiO<sub>2</sub> nanocomposite hydrogel was prepared using free radical copolymerization in the aqueous solution, which included dissolving the 0.1g of

TiO<sub>2</sub> NPs in 20mL of deionized water with continuous stirring for 4h, after that it was transferred to the ultra-sonication for 4h. the reaction mixture was transferring to a three-necked round bottom flask container condenser, funnel separated and nitrogen gas. Then 0.5 g of SA was slowly added with stirring to the reaction mixture then added 4g AAC for 15 min, Then 2g/2ml H<sub>2</sub>O AAM solution was prepared and added slowly with stirring for 15min, The solution of 0.05g/2ml H<sub>2</sub>O of MBA was add gradually in form of drops with continuous stirring for 15min After this 4 drops of TEMED solution was added to the reaction mixture with continuous stirring for 5min. The solution of KPS (was prepared by dissolved 0.03g in 2mL deionized water) was added into the reaction mixture for 15 min with stirring at room temperature. The temperature was raised to 70 °C for 2h to complete the polymerization reaction. To remove the unreacted parts from SA-g-P(AAC-AAM)/TiO<sub>2</sub> hydrogel Nano composite, it was immersed in deionized water. Finally, it was putting in an oven with 50°C for drying and to getting a constant weight.



Fig 1: Photo of (A)preparing and cutting(B)washing (C)drying and grinding of hydrogel Nano composite.

Characterization of Hydrogel and Nanocomposite Hydrogel

Fourier Transform Infrared Spectroscopy

To categorize functional groups of SA-g-P (AAC-AAM) hydrogel and SA-g-P(AAC-AAM)/TiO<sub>2</sub> Nano composite hydrogel, FTIR spectroscopy (Shimadzu8400S, Japan) was

utilized. Using potassium bromide K Br in the wavelength range (4000-400  $\text{cm}^{-1}$ ), FTIR spectroscopy data for the generated surface were acquired.

Field Emission Scanning Electron Microscopy FESEM  
FESEM was used using field-emission scanning electron microscopy (FESEM) identifying the characteristics of the external structure morphology in terms of size and shape of the Nano-hydrogel composite with different magnification forces before and after loading titanium dioxide on it.

#### Calibration Curves of Pb (II) Ions

To determine the calibration curve of the lead ion Pb(II) a set of standard solutions of lead ion with concentrations ranging (1-60mg/L) were prepared, and using atomic absorption spectrometry the absorbance was measured and these results showed that it obeys the Beer-Lambert law [15].

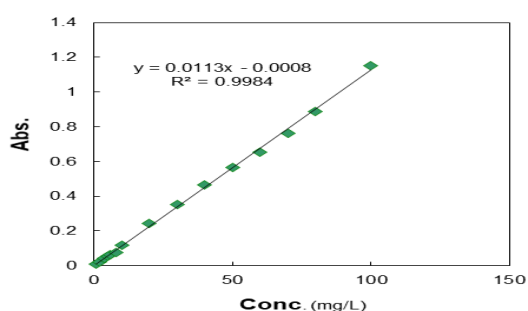


Fig (2): Calibration Curves of Pb (II) IONS

#### Kinetic Studies

The equilibrium period was calculated in this investigation by dissolving 0.05 g of adsorbent in 10 ml of a standard lead ion solution using a thermostatic shaker while maintaining the solution temperature at 25°. The centrifuge apparatus was used to separate the fluids, and the absorbance was measured with an atomic absorption device to calculate the equilibrium time.

#### Results and Discussion

##### FTIR Analysis

The FT-IR spectrum after Pb(II) adsorption process on the surface of the indicates the association of Pb(II) ions with amino groups, which affects their vibration, as the ion bonding leads to a decrease in the permeability at the adsorption peaks. Also, the adsorption of Pb affects the expansion and bending of the OH and C-H groups, which indicates a change in the positions of the hydroxyl, amine and carbonyl groups during the adsorption of these ions. The bands appears in FT-IR spectrum is 1490  $\text{cm}^{-1}$  belong to C=O, Amide, 2800  $\text{cm}^{-1}$  that refers to  $\text{CH}_3$  stretching, 1735  $\text{cm}^{-1}$  belong C=O of COOH, 1396 refers to C=C [16-18].

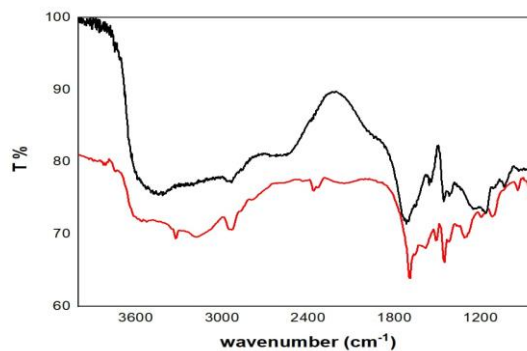
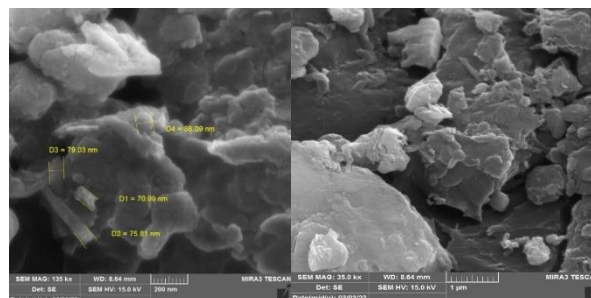


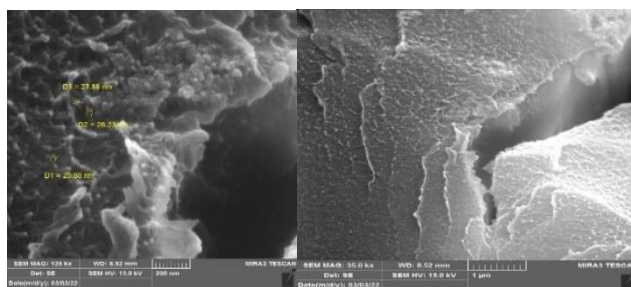
Fig (3) The FT-IR Spectrum after Pb (II) Adsorption Process

#### Field Emission -Scanning Electron Microscopes FESEM

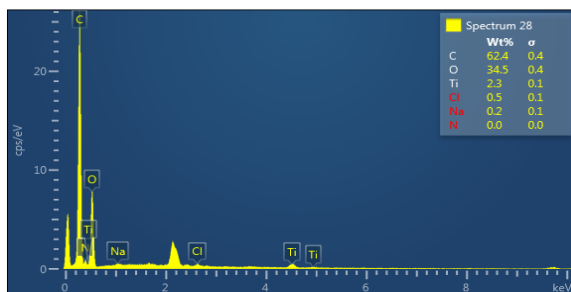
The scanning electron microscopy technique FE-SEM was used to determine the surface characteristics (particle shape, size, homogeneity between the components of the compound and their distribution on the surface, how the polymer chains are interconnected, the nature of the surface being porous or smooth) [18]. The hydrogel is a nano-composite with a sponge-like structure and a network of tight layers that is rough and porous. It also had randomly collected wrinkles, as shown in figure (4), but following Pb adsorption on the overlay's surface, the FESEM picture revealed that the surface became smoother and more cohesive as the pores on the surface were filled with Pb ions [18, 19] This verifies the presence of adsorption, as seen in Figure (5). As well as using EDX the adsorption process' effectiveness was also demonstrated by comparing the oxygen and carbon ratios of the hydrogel complex before and after adsorption, as shown in Figure (6).



(Figure 4): FESEM Images of the Overlapping Surface of SA-g-poly (AAC-AAM)/TiO<sub>2</sub> before Adsorption at Magnification Powers Vary.



(Figure5): FESEM Images of the Overlapping surface of SA-g-poly (AAC-AAM)/TiO<sub>2</sub> after Adsorption at Magnification powers vary.

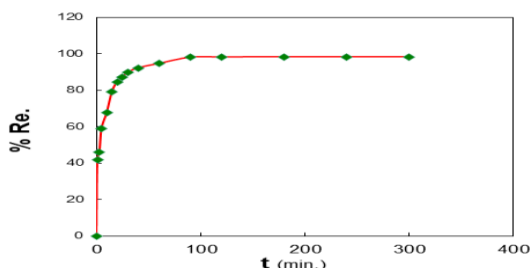


(Figure 6): The Ratio of Element that Form Hydrogel Composite after

## Adsorption Pb (II).

### Adsorption kinetics

An SA-g-P(AAC-AAM)/TiO<sub>2</sub> nanocomposite hydrogel adsorbs Pb (II). Figure (6) shows the results in relation to contact time. The rate of ion adsorption on SA-g-P (AAC-AAM)/TiO<sub>2</sub> nanocomposite is higher at first, then gradually drops to virtually constant after 120 minutes. Possibly, there were many accessible adsorption sites at the start of the process, which later became saturated [19].



(Fig 7): Effect of Contact Time Pb (II) Adsorption at 25°C.

Figure 7 and table 1 demonstrate that the correlation coefficient (R<sup>2</sup>) for the pseudo-second-order model is greater than the pseudo-first-order model for Pb(II) ions adsorption process on SA-g-P(AAC-AAM)/TiO<sub>2</sub>. So the adsorption mechanism is pseudo-second-order.

**Table (1) Kinetic Adsorption Coefficients of Pb on SA-g-P(AAC-AAM)/TiO<sub>2</sub> at 25 °C.**

Pseudo- first order Pb (II) on SA-g-P(AAC-AAM)/TiO2					
R2	Qe (mg/ g)	K1 (min-1)	Slope		
0.8109	12.5535	0.022	-0.022		
Pseudo - second order Pb (II) on SA-g-P(AAC-AAM)/TiO2					
R2	qe (mg/ g)	K2 (g. mg-1/: min)	Intercept	Slope	H (mg. g-1/min)
0.9999	59.8802	0.0527	0.0527	0.0166	18.9753

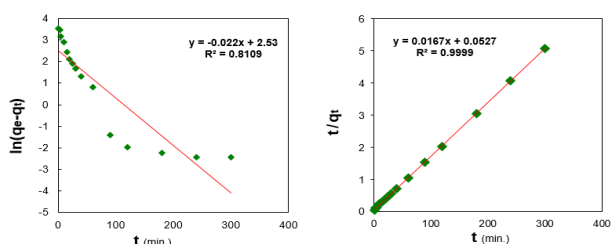


Fig8: (a) Pseudo-First-order Model desorption Pb (II) on SA-g-P(AAC-AAM)/TiO<sub>2</sub> and (b) Pseudo-second-order model adsorption Pb (II) on SA-g-P(AAC-AAM)/TiO<sub>2</sub> at 25 °C.

## 2. Conclusion

The study concluded the following:

- 1-The adsorbent compound (SA-g-p(AAC-AAM)) effectively adsorbs heavy element Pb (II) ions from aqueous solutions.
- 2-The equilibrium time for lead ion adsorption is 120 minutes.
- 3-In the Pb (II) adsorption procedure, the adsorbent composite SA-g-p(AAC-AAM)/TiO<sub>2</sub> should be 0.05g.
- 4- A pseudo-second-order model is used to explain Pb (II) adsorption on the surface of the SA/g-poly (AAC-AAM) composite.

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