# **RESEARCH PAPER**

# Removal of Pb (II) Ions from Water by Adsorption on Sodium Alginate-g-poly (Acrylic acid-co-Itaconic acid)/Titanium Dioxide [SA-g-p(AA-IA)/ TiO<sub>2</sub>] Hydrogel Nanocomposite

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# ABSTRACT

The increasing contamination of water bodies by heavy metals, particularly lead (Pb (II)), poses a significant threat to environmental and public health. This research investigates the synthesis, characterization, and Pb (II) adsorption capabilities of a novel sodium alginate-g-poly (acrylic acid-co-Itaconic acid)/ titanium dioxide [SA-g-p(AA-IA)/ TiO<sub>2</sub>] hydrogel nanocomposite. The nanocomposite was synthesized via free-radical polymerization and characterized using FTIR, XRD, and FESEM, which confirmed successful grafting, the incorporation of TiO<sub>2</sub> nanoparticles, and a porous structure. The influence of various operational parameters, including time, pH, temperature, and Pb (II) dose, on adsorption was systematically evaluated. The nanocomposite demonstrated a substantial Pb (II) adsorption capacity, reaching approximately 998.62 mg/g at pH 10, with equilibrium attained within 90 minutes. Kinetic analysis indicated that adsorption followed a pseudo-second model, suggesting chemisorption as primary mechanism. Adsorption isotherm data fitted to Freundlich and Temkin models, showing multilayer adsorption. Thermodynamic analysis revealed the process to be spontaneous ( $\Delta G = -5.439 \text{ kJ/mol}$ ) and endothermic ( $\Delta H = 23.404 \text{ kJ/mol}$ ). The hydrogel composite also exhibited pH-sensitive swelling behavior, with maximum swelling observed under alkaline conditions. These results highlight potential of SA-g-p(AA-IA)/ TiO<sub>2</sub> nanocomposite as an effective adsorbent for Pb (II) removal from solutions.

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#### INTRODUCTION

The enduring presence of organic and inorganic pollutants, particularly heavy metals lead to polluting air, soil, and water [1]. Nonbiodegradable pollutants like heavy metals can \* Corresponding Author Email: layth.alhayder@gmail.com accumulate in living organisms through the food chain, drinking water, and air [2, 3], posing risks to human health, aquatic life, and the overall ecosystem [4, 5]. Water, a vital natural resource, is increasingly affected by the industrial release

of chemicals, especially heavy metals [5]. Among various heavy metals, lead stands out due to its severe toxicity. It can lead to kidney problems, destroy red blood cells, damage the central nervous system, and ultimately cause death [6-12]. Therefore, addressing and eliminating such heavy metal pollution is crucial. Adsorption stands out as a highly favored and widely researched method for eliminating heavy metals from wastewater due to its operational simplicity, high removal efficiency even at low pollutant concentrations, cost-effectiveness, and the possibility of adsorbent regeneration and reuse, supporting sustainability [13-15]. The process relies on the transfer of adsorbate from liquid to surface of a solid adsorbent, where they are held by various physical and chemical interactions [16, 17]. These interactions encompass electrostatic forces, van der Waals forces, hydrogen bonding, and potentially chemical bond formation. Consequently, current research heavily emphasizes the synthesis of new adsorbents with optimized properties to maximize metal i.e., Pb (II) removal, including exploring different materials, refining their synthesis, and modifying their surface characteristics to enhance their affinity for Pb (II) removal [18, 19].

Hydrogels, capable of significant swelling in water, can be synthesized from various natural polysaccharides like sodium alginate (SA), chitosan, cellulose, starch, gellan gum, and guar gum, as well as synthetic polymers as poly (acrylic acid) (AA), poly (Itaconic acid) (IA), and poly (N-vinyl pyrrolidone) (PNVP) [20, 21]. Polysaccharide-based hydrogels are especially appealing for environmental cleanup due to their biocompatibility, biodegradability, low cost, and natural availability and sustainable development principles [22-25]. Their swelling ability generates a large network structure with extensive surface contact, promoting pollutant molecule diffusion and enhancing adsorption. Moreover, their chemical and physical properties can be precisely controlled during synthesis to introduce specific functional groups with high affinity for particular pollutant types, enabling the synthesis of selective and efficient adsorbents [26]. Current research focuses on advanced synthesis techniques like graft copolymerization, interpenetrating polymer networks, and nanomaterial incorporation to improve mechanical strength and adsorption capacity, aiming to develop more effective hydrogel adsorbents for practical wastewater treatment [27].

This study centers on synthesis and application of SA-g-p(AA-IA)/TiO<sub>2</sub> nanocomposite hydrogel to harness synergistic benefits of a natural polysaccharide (SA), synthetic monomers (AA and IA), and inorganic nanoparticles (TiO<sub>2</sub>) for removal of (Pb (II)) ions from solutions [28]. Sodium alginate, an anionic polysaccharide derived from seaweed, is a readily available, cost-effective, and biodegradable biopolymer providing a biocompatible matrix for hydrogel formation [29]. Grafting acrylic acid onto alginate backbone introduces pH-responsive carboxylic acid functional groups, which enhance adsorption of cationic metal ions like Pb (II), particularly under alkaline conditions [30]. The incorporation of Itaconic acid, a non-ionic monomer, into polymer network contributes to hydrogel's overall structural integrity, mechanical strength, and water swelling capacity, facilitating accessibility of adsorption sites within hydrogel matrix [31]. The inclusion of TiO<sub>2</sub> nanoparticles within hydrogel matrix introduces a component known for its high surface area and potential interactions with metal ions [32, 33].

The specific objectives of this research include: i. Synthesizing and thoroughly characterizing the SA-g-p(AA-IA)/TiO<sub>2</sub> hydrogel nanocomposite using appropriate analytical techniques to confirm its successful formation and to understand its structural and morphological properties. ii. Systematically evaluating the influence of key operational parameters, as time, pH of solution, temperature and Pb (II) concentration on efficiency of process. iii. Determining the adsorption kinetics by applying established kinetic models, including pseudo-first-order and pseudo-second-order models, to experimental data obtained under various conditions to elucidate the rate-controlling steps and mechanism of adsorption. iv. Calculating the thermodynamic parameters, as Gibbs free energy change ( $\Delta G$ ), enthalpy change ( $\Delta H$ ), and entropy change ( $\Delta S$ ), to assess spontaneity of adsorption process and to determine whether it is endothermic or exothermic in nature. v. Analyzing the adsorption equilibrium data using various isotherm models as Langmuir, Freundlich, and Temkin models, to determine maximum adsorption capacity of nanocomposite for Pb (II) ions and to gain insights into nature of adsorption mechanism at solid-liquid interface. A comprehensive investigation of isothermal,

kinetics, and thermodynamics of Pb (II) removal on SA-g-p(AA-IA)/ TiO<sub>2</sub> will provide a thorough understanding of adsorption mechanism, enabling the optimization of its performance for efficient and sustainable removal of this heavy metal from wastewater.

## MATERIALS AND METHODS

## Chemicals used

This study employed a range of chemical reagents sourced from various suppliers, each with specific purities. Sodium alginate with a purity of 99.0% was obtained from Himedia. Titanium dioxide with 99.7% purity was supplied by Merck. Itaconic acid at 99% purity and N,N'-methylene-bisacrylamide at 99% purity were both acquired from Himedia Laboratories. Potassium persulfate with 99% purity was purchased from Riedel-dehaenag seclze hannover. Ethanol with a purity of 95% was obtained from Merck ENERGY, while acrylic acid at 99% purity was from Himedia. Nitrogen gas was supplied by Xinrui. Sodium hydroxide with 99% purity was acquired from Alpha Chemika, and hydrochloric acid with a concentration of 38% was from J.T. Baker. Calcium carbonate with 99% purity, potassium chloride with 99.5% purity, and tetramethylethylenediamine with 99.0% purity were all sourced from Fluka. Additionally, lead nitrate with 99.0% purity was obtained from Himedia Laboratories.

# Synthesis of SA-g-p(AA-IA)/ TiO<sub>2</sub> Nanocomposite Hydrogel

The SA-g-p(AA-IA)/ TiO<sub>2</sub> underwent synthesis via free-radical copolymerization in an aqueous environment. The process commenced with the dispersion of 0.12 g of TiO, nanoparticles in 20 mL of deionized water, achieved through 4 hours of continuous stirring followed by an additional 4 hours of ultrasonication. This dispersion was then transferred to a three-necked round-bottom flask fitted with a condenser, a dropping funnel, and a nitrogen gas inlet, and placed in a stirred water bath until complete dissolution was observed. Subsequently, 0.5 g of sodium alginate (SA) was introduced into the reaction mixture and agitated for 1 hour to yield a homogeneous solution. Next, 4 g of acrylic acid (AA) added dropwise to preceding solution under continuous stirring for 15 minutes. Following this, a solution prepared by dissolving 2 g of Itaconic acid (IA) in 2 mL of H<sub>2</sub>O until fully dissolved was added gradually to

the reaction mixture over 15 minutes of constant stirring. In a similar manner, 0.05 g of N, N'-methylenebisacrylamide (MBA) as a cross-linking agent was dissolved in 2 mL of  $H_2O$  and added slowly to the solution with 15 minutes of agitation.

#### Batch adsorption and calibration curve

To assess the synthesized SA-g-p(AA-IA)/TiO capacity as an adsorbent for removing Pb (II) ions from water, batch experiments were conducted. The parameters investigated for Pb (II) adsorption included contact time (0-300 min), temperature (10, 15, 20, 30 °C), initial Pb (II) concentration (100-500 mg/L) and pH (2-10). Each experiment involved agitating 10 mL of Pb (II) solution in a conical flask at a shaking speed of 130 rpm. The pH of the solutions was adjusted using 0.1 M NaOH and HCl solutions, and a pH meter was used for measurement. Following the attainment of equilibrium, the concentration of Pb (II) remaining in the solution was determined using an atomic absorption spectrophotometer (AAS) at its characteristic wavelength. Subsequently, the quantity of Pb (II) adsorbed per unit mass of the adsorbent (mg/g) and the percentage removal of Pb (II) were calculated by Eqs. 1 and 2 correspondingly [34].

$$q_e = \frac{C_o - C_e * V}{m} \tag{1}$$

$$R(\%) = \frac{C_o - C_e}{C_o} x100$$
 (2)

here, and refers to initial and equilibrium concentrations of Pb (II) solution (mg/L), correspondingly while "m" denotes dose of nanocomposite (g) and volume of pollutant solution used is represented by V (L).

A calibration curve for Pb (II) ions (**Fig. 1**) was established by measuring the absorbance of standard Pb (II) solutions at its characteristic wavelength using AAS. This curve demonstrated a strong linear correlation between absorbance and concentration, consistent with Beer-Lambert's law. The resulting linear equation with a high coefficient of determination ( $R^2 = 0.998$ ) provided a reliable method for quantifying Pb (II) concentrations in adsorption studies, enabling calculation of residual Pb (II) concentration, adsorption capacity, and Pb (II) removal efficiency.

## **RESULTS AND DISCUSSION**

Characterization	analysis			
Examining	the	SA-g-p(A	A-IA)/	TiO₂
nanocomposite	using	FTIR	spect	roscopy

before and after Pb (II) ion adsorption reveals significant modifications in characteristic peaks of functional groups. The initial FTIR spectrum of nanocomposite (Fig. 2), prior to Pb (II) exposure,



Fig. 1. Calibration plot for Pb (II) ion solution.



Fig. 2. FTIR of nanocomposite before and after Pb (II) ions adsorption.

J Nanostruct 15(3): 983-996, Summer 2025

displayed peaks corresponding to functional groups of its constituent components: sodium alginate (SA), acrylic acid (AA), Itaconic acid (IA), and TiO2. A broad absorption band observed in range of 3300–3400 cm<sup>-1</sup> indicated the presence of stretching vibrations of -OH and -NH bonds, originating from hydroxyl groups of alginate and the amide groups of IA. A prominent peak around 1650 cm<sup>-1</sup> was attributed to C=O stretching vibration within amide I groups. A band appearing between 1550-1600 cm<sup>-1</sup> likely represented N-H bending (amide II) or asymmetric stretching of carboxylate (-COO<sup>-</sup>) ions from AA. The symmetric stretching of -COO<sup>-</sup> groups appeared in region of 1400–1450 cm<sup>-1</sup>. Furthermore, a strong band around 1020-1080 cm<sup>-1</sup> corresponds to C-O-C stretching. Further, TiO<sub>2</sub> was confirmed by a band in the range of 500–700 cm<sup>-1</sup> of spectra. Following the adsorption of Pb (II) ions, the FTIR spectrum displayed noticeable shifts and changes in intensity of several peaks, suggesting interactions between metal ions and functional groups of composite. The -OH/ -NH stretching band became broader and underwent a slight shift in wavenumber, indicating the potential formation of coordination bonds or electrostatic interactions between the Pb (II) ions and composite's polar groups. The peak at 1650 cm<sup>-1</sup> (C=O stretching) exhibited a minor shift and a decrease in intensity, implying an interaction between Pb (II) and amide or carboxylic acid functionalities. Furthermore, the peak near 1550 cm<sup>-1</sup> showed a shift or a change in intensity, which is attributed to involvement of carboxylate or amine groups in ionic binding with Pb (II) ions. Variations observed in 1400– 1450 cm<sup>-1</sup> region suggested possible interactions between positive charges of Pb (II) and carboxyl groups of nanocomposite, potentially through ionic exchange or complexation. The C–O–C peak near 1020–1080 cm<sup>-1</sup> might have also experienced a change in intensity due to minor rearrangements in polymer backbone upon Pb (II) adsorption [35-38].

XRD analysis revealed the crystalline nature of pure TiO<sub>2</sub> nanoparticles, with sharp peaks corresponding to anatase phase, as evidenced in Fig. 3a. Upon incorporation of TiO<sub>2</sub> into SA-g-p(AA-IA) hydrogel, the resulting nanocomposite (Fig. 3b) exhibited significantly weaker and broader TiO<sub>2</sub> peaks, indicating a reduction in crystallite size and distortion of TiO<sub>2</sub> structure due to dispersion within amorphous polymer network. The presence of a broad halo between 15° and 30° in nanocomposite XRD pattern confirmed non-crystalline nature of hydrogel matrix, while persistence of characteristic anatase TiO<sub>2</sub> peaks, even though with reduced intensity, suggested that the TiO<sub>2</sub> nanoparticles retained their crystalline structure within composite material [39].

FESEM images of nanocomposite prior to Pb (II) ion adsorption (Fig. 4a, 10  $\mu$ m scale) revealed a textured, porous surface with numerous irregularities and openings, indicative of a substantial surface area potentially beneficial for metal ion adsorption. The TiO<sub>2</sub> nanoparticles appeared distributed throughout hydrogel



Fig. 3. XRD of (a) TiO, and (b) SA-g-p(AA-IA)/ TiO, nanocomposite.

J Nanostruct 15(3): 983-996, Summer 2025

matrix, which could contribute to the material's mechanical stability and enhance its affinity for Pb (II) ions via surface interactions. In contrast, FESEM image of nanocomposite following Pb (II) ion adsorption (Fig. 4b, 5  $\mu$ m scale) displayed a considerably smoother and more compact surface morphology. The previously observed pores and openings appeared to be filled or covered,

suggesting that Pb (II) ions had successfully bound to and permeated the nanocomposite's surface. This transformation from a porous to a smoother texture provided visual confirmation of material's adsorptive capabilities for Pb (II) ions [38].

## Kinetic study

The influence of time on adsorption of Pb









(b)

Fig. 4. FESEM of SA-g-p(AA-IA)/ TiO, (a) before and (b) after Pb (II) ions adsorption.

(II) ions by nanocomposite (Fig. 5a-b) was evaluated by examining the adsorption capacity (Q<sub>a</sub>) and percentage removal (% RE). The results demonstrated a rapid initial phase of Pb (II) uptake. Within the first minute, a Q of 815.31 mg/g and a %RE of 65.22% were observed, indicating a fast initial binding of Pb (II) ions to readily available active sites on nanocomposite surface. This was followed by a substantial increase within the initial 15 minutes, reaching a Q of 1093.78 mg/g and a %RE of 87.50%, highlighting a strong initial mass transfer driven by concentration gradient and availability of active sites. Subsequently, the adsorption rate decreased in an intermediate phase (15-90 minutes) as these active sites became progressively occupied. Eventually, system approached equilibrium in later stages (beyond 90 minutes), with minimal further change observed in Q and %RE, suggesting that adsorption saturation was largely achieved by 90 minutes. The maximum Q reached at equilibrium was approximately 1237.66 mg/g, with a corresponding %RE of around 99.01%. This indicates that a contact time of 90 minutes was sufficient for nanocomposite to effectively adsorb Pb(II) ions under studied conditions [40].

To gain insight into the mechanism and ratecontrolling step of Pb (II) ion adsorption onto nanocomposite hydrogel over time, pseudo-firstorder and pseudo-second-order kinetic models were employed. The pseudo-first-order model posits that rate of adsorption site occupancy is proportional to number of unoccupied sites, while pseudo-second-order model suggests that rate-limiting step involves chemisorption, characterized by sharing of electrons between adsorbent and adsorbate (Pb (II) ions). Eqs. 3 and 4 represent mathematical expressions for pseudofirst-order and pseudo-second-order models, correspondingly:

$$\log(Q_{e} - Q_{t}) = \log Q_{e} - \frac{k_{1}}{2.303}t$$
 (3)

$$\frac{t}{Q_{t}} = \frac{1}{k_{2}Q_{e}^{2}} + \frac{t}{Q_{e}}$$
(4)

here,  $q_t$  and  $q_e$  refers to amounts of adsorbate adsorbed at time t and at equilibrium, respectively (mg/g) while  $k_1$  and  $k_2$  refers pseudo-first rate constant (min<sup>-1</sup>) and second order model (g/ mg·min), correspondingly. The kinetic study results (Fig. 6a-b and Table 1) indicated that pseudofirst-order model did not effectively represent adsorption of Pb (II) ions by nanocomposite, as evidenced by its lower regression coefficient compared to pseudo-second model. This suggests



Fig. 5. Effect of time on (a) % removal and (b) Q, mg/g, for Pb (II) adsorption.

J Nanostruct 15(3): 983-996, Summer 2025

that the adsorption process is not solely governed by physical diffusion. Conversely, pseudo-second model provided a better fit to kinetic data, implying that chemisorption is the predominant mechanism. This likely involves interactions as electrostatic forces or complexation between Pb (II) ions and functional groups (e.g., –COOH, –NH<sub>2</sub>) present on nanocomposite [41].

## Isothermal and temperature study

The adsorption capacity ( $Q_e$ ) of nanocomposite for Pb (II) ions was observed to increase with higher initial Pb (II) concentrations across all tested temperatures (10, 15, 20, and 30 °C) (Fig. 7). Specifically, at 10 °C,  $Q_e$  values were 249.82 mg/g at an initial Pb (II) concentration ( $C_o$ ) of 100 mg/L, 374.16 mg/g at 150 mg/L, and 1024.16 mg/g at 500 mg/L. Similar trends were observed at 15 °C (Q values of 249.82, 374.82, and 1081.68 mg/g for C of 100, 150, and 500 mg/L, respectively), 20 °C (Q values of 249.82, 374.82, and 1102.26 mg/g for Co of 100, 150, and 500 mg/L, respectively), and 30 °C (Qe values of 249.82, 374.82, and 1125.93 mg/g for C of 100, 150, and 500 mg/L, respectively). These results indicate that a higher availability of Pb (II) ions in solution, resulting from increased concentrations, provides a greater driving force for Pb (II) ions towards adsorbent surface and their subsequent binding. Furthermore, the effect of temperature on adsorption capacity appears to be complex. While there is a slight increase in Q<sub>a</sub> with rising temperature from 10 °C to 30 °C at higher initial Pb (II) concentrations (e.g., C<sub>o</sub> of 500 mg/L), the adsorption capacity at lower concentrations



Fig. 6. Plot for (a) pseudo first and (b) pseudo second model.

Table 1. Pseudo first and second model parameters. Experimental (mg/g) = 1237.6 mg/g.

Model	Pseudo-first order	Pseudo-second order
q <sub>e</sub> (mg/g)	221.2	1250
$\mathbb{R}^2$ values	0.951	0.999
Constant	0.0364	0.0004
Constant	K <sub>1</sub> (1/min)	$K_2$ (g/mg · min)

(e.g.,  $C_{o}$  of 100 and 150 mg/L) remains relatively consistent across tested temperature range [39, 42, 43].

Thermodynamic parameters such as Gibbs free energy change ( $\Delta$ G), enthalpy change ( $\Delta$ H), and entropy change ( $\Delta$ S) offer essential information



Fig. 7. Effect of Pb (II) concentration at different temperatures.



Fig. 8. Van't Hoff plot.

Table 2. Thermodynamic parameters.

T (°C)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol K)	Kc
20	-5.439	23.404	97.897	9.326



Fig. 9. Graphs for (a) Langmuir, (b) Freundlich and (c) Temkin models.

J Nanostruct 15(3): 983-996, Summer 2025

regarding feasibility and spontaneity of adsorption process, as well as changes in order or disorder at solid-solution interface. Utilizing data obtained at 20 °C (from the Van't Hoff plot, Fig. 8), the thermodynamic parameters for Pb (II) adsorption were determined (Table 2). These values include a ΔG of -5.439 kJ/mol, indicating that the adsorption of Pb (II) ions onto nanocomposite is a spontaneous process under these conditions. Furthermore, the ΔH value of 23.404 kJ/mol reveals that adsorption process releases heat, and is therefore exothermic. Additionally, ∆S value of 97.897 J/mol·K suggests feasibility of process. This reduction in randomness is likely attributed to ordering of Pb (II) ions as they become immobilized on specific sites of adsorbent surface, resulting in a decrease in their degrees of freedom compared to their state in the aqueous solution [44].

To better understand the mechanisms governing the interaction between Pb (II) ions and nanocomposite surface, three adsorption isotherm models were applied: Langmuir, Freundlich, and Temkin isotherms. The Langmuir model (Eq. 5) operates on assumptions of monolayer adsorption, where a single layer of adsorbate ions forms on a uniform adsorbent surface with a limited number of identical adsorption sites. The Freundlich isotherm (Eq. 6) describes multilayer adsorption on heterogeneous surfaces, where enthalpy of adsorption changes with extent of surface coverage. In contrast, the Temkin model (Eq. 7) considers interactions between adsorbed ions and assumes that enthalpy of adsorption decreases linearly with surface coverage.

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_o b C_e}$$
(5)

$$\log q_e = \log K_f + \frac{1}{n} \log C_e$$
(6)

$$q_{eq} = BlnA_{T} + BlnC_{eq}$$
(7)

where,  $q_e$  is equilibrium adsorption capacity,  $R_{L}$  is Langmuir constant, Ce is equilibrium adsorbate concentration,  $K_f$  is Freundlich constant, 1/n is an empirical parameter of surface heterogeneity, R is gas constant, T is absolute temperature, b is Temkin constant related to heat of adsorption, and A is Temkin isotherm constant. Isotherm analysis (Fig. 9a-c) indicated that the adsorption behavior of Pb (II) ions onto nanocomposite could be effectively described by both Freundlich and Temkin models, as evidenced by their high R<sup>2</sup> values. This suggests that the composite surface likely presents



Fig. 10. Effect of pH on (a) swelling ratio and (b) adsorption capacity of nanocomposite for Pb (II) ions.

J Nanostruct 15(3): 983-996, Summer 2025

energetically diverse sites, facilitating multilayer adsorption characterized by varying adsorption energies and interactions between adsorbed Pb (II) ions [45].

# Effect of pH

he swelling behavior of the SA-g-p(AA-IA)/TiO<sub>2</sub> nanocomposite hydrogel is markedly dependent on pH, as surrounding ionic conditions influence charge of functional groups within its polymeric framework, and this is quantified by Eq. 8:

Swelling ratio (%) = 
$$\frac{W_S - W_d}{W_d} \times 100$$
 (8)

Under acidic conditions (Fig. 10a), the hydrogel exhibited limited swelling. At pH 2, the swelling percentage was 130%, gradually increasing to 240% at pH 3 and 330% at pH 4. This constrained swelling in acidic environments is primarily due to the protonation of carboxylic (-COOH) and amide groups, which reduces the repulsive forces between the polymer chains and limits the hydrogel's water uptake capacity. As the pH increased to 5 and beyond, a significant enhancement in swelling capacity was observed. At pH 5, the swelling percentage reached 470%, continuing to rise substantially to 670% at pH 6, 850% at pH 7, and reaching a high of 1060% at pH 8. This considerable increase is attributed to the deprotonation of -COOH groups, forming -COO<sup>-</sup> ions, which leads to stronger electrostatic repulsion within the polymer network. The increased density of negative charges causes the hydrogel structure to expand more extensively, enabling greater water absorption. Beyond pH 8, the swelling capacity continued to increase, but at a slower pace, reaching 1100% at pH 9, 1120% at pH 10, and eventually plateauing at 1155% at pH 12. This leveling off indicates that ionizable groups within polymer have largely lost their protons, and hydrogel structure has approached its maximum expansion in alkaline conditions [46].

The adsorption capacity  $(Q_e)$  of nanocomposite for Pb (II) ions was significantly influenced by pH of solution (Fig. 10b). At a highly acidic pH of 2,  $Q_e$  was 958.44 mg/g. This relatively high adsorption under acidic conditions suggests a strong interaction between Pb (II) ions and protonated functional groups on adsorbent surface. As pH increased to 4 and then to 6, adsorption capacity showed a slight improvement, reaching 970.30 mg/g and 981.45 mg/g, respectively. This increase indicates that as concentration of H<sup>+</sup> ions decreased, more favorable binding sites for Pb (II) ions became available. A further increase in adsorption was observed at pH 7 and 8, where Q values were 989.59 mg/g and 994.02 mg/g, respectively, suggesting that near-neutral to slightly alkaline conditions are optimal for Pb (II) adsorption onto this nanocomposite. Further increases in pH to 9 and 10 resulted in a minimal additional increase in adsorption capacity to 996.50 mg/g and a peak of 998.62 mg/g, indicating that a weakly basic pH provides the most effective adsorption of Pb (II) ions. These results highlight pH-dependent surface charge of nanocomposite and its impact on electrostatic interactions with Pb (II) ions [46].

#### CONCLUSION

In this study, a SA-g-p(AA-IA)/ TiO<sub>2</sub> hydrogel nanocomposite was successfully synthesized and its effectiveness in removing Pb (II) ions from aqueous solutions was evaluated. The material exhibited favorable structural and surface properties, as confirmed by FTIR, XRD, and FESEM analyses, which supported its strong affinity for Pb (II). The adsorption was significantly influenced by operational factors, particularly pH, where weakly basic conditions enhanced adsorption. Kinetic modeling indicated that adsorption process followed pseudo-second-order kinetics, suggesting a chemical adsorption mechanism. Isotherm analysis revealed that adsorption behavior was best described by both Freundlich and Temkin models, indicating multilayer adsorption on a heterogeneous surface. The thermodynamic parameters confirmed spontaneity and exothermic nature of adsorption process. The composite also demonstrated pH-sensitive swelling behavior, with high adsorption capacity achieved under specific conditions. These findings suggest that the SA-gp(AA-IA)/ TiO<sub>2</sub> hydrogel nanocomposite shows promise as an efficient adsorbent for removal of Pb (II) ions from contaminated water.

## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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J Nanostruct 15(3): 983-996, Summer 2025

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