Article

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pb(II) ion Removal from contaminated water samples using a New Nanopolymer by spectrophotometric technique

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Abstract:

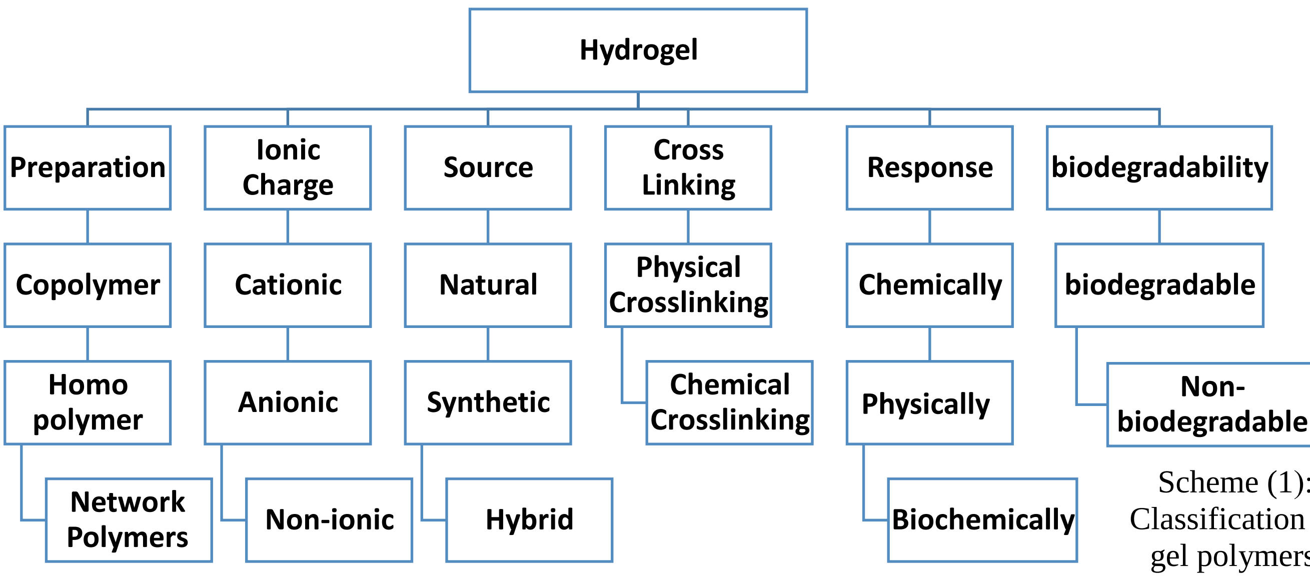
This study involved the preparation of a new nanopolymer with outstanding environmental properties and high stability. The polymer was manufactured through a copolymerization process involving hydrophilic and hydrophobic monomers. The starting materials used were acrylic acid (AA), methyl cellulose (MC), and polylactic acid (PLA).To achieve the nanopolymerization, a free-radical photopolymerization method was employed. This process involved the polymerization of methyl cellulose, acrylic acid, and polylactic acid, with 1,6-hexanediol diacrylate (HDODA) as a cross-linking agent and 1-hydroxycyclohexylphenyl ketone as a photoinitiator.The resulting nanopolymer combines the properties of the starting materials, demonstrating its high environmental friendliness and structural stability. This makes it a promising candidate for applications requiring high-performance and sustainable materials. The nanopolymer was characterized using FTIR, TGA, DSC, SEM, and TEM techniques. The swelling ratio of this polymer was measured as a type of gel polymer. The study applied the nanopolymer to the spectroscopic determination of lead(II) ions. The optimal conditions for the determination process were determined, such as determining the wavelength of maximum absorption. A spectroscopic reference method was used to calculate the remaining ion using a new standard method for estimating the ion is characterized by the use of the reagent (2-[(6-Methoxy-2-benzothiazolyl) azo]-4-methoxyphenol) 6-MBTAMP at a wavelength of 670 nm, pH 7, reagent volume of 0.5 ml, and ion volume of 2 ml. A standard calibration curve for the lead(II) complex was also prepared before adsorption. Studying the best conditions for the adsorption process and estimating the effect of pH, shaking time, mass of the adsorbed surface, and volume of the ion solution on the adsorption process. A calibration curve was also prepared after the adsorption process to estimate the remaining lead(II) ions. Linearity range was 0.5 to 10 $mg.L^{-1}$, and Sandell's sensitivity was 0.1666 $\mu g.cm^{-2}$, molar absorptivity(ε) was $1.2432\times10^{3} L.mol^{-1}.cm^{-1}$.

Key words: Nanopolymer, Hydrogels, Acrylic Acid, Polylactic Acid

Introduction

Water is a vital element for life, and its quality is greatly affected by pollutants, most notably heavy metals. Some metals, such as zinc and copper, are essential in small quantities, while others, such as lead and cadmium, are toxic even at low concentrations [1]. Increased contamination with these toxic metals poses a serious health problem for humans, plants, and animals [2]. Heavy metals enter the human body through water and food, and travel through the circulatory system to negatively affect vital organs and various cells [3]. Igneous and sedimentary rocks are the main natural sources of heavy metals in soil [4]. Heavy metal sources can be basically classified into two main categories: natural geological sources and anthropogenic sources [5,6]. Before selecting any treatment technology, the precise quantification of heavy metal concentrations in the water must be determined. This process requires the use of a cost-effective, environmentally friendly, and highly sensitive analytical technique to ensure reliable results and select the optimal solution for pollution control [7]. Adsorption is an effective separation process in which unwanted molecules or ions from a liquid or gaseous medium accumulate at the interface of a solid or liquid [8]. Adsorption technology is one of the most common and effective Ways to get rid of and separating the elimination of heavy metals from aqueous solutions, as it is environmentally friendly [9]. Interlocking and semi-interlocking nano-polymer gel networks have received increasing research interest as adsorbents, especially for the elimination of heavy metals and toxic dyes, due to their excellent swelling properties and high adsorption efficiency [10]. Composed of repeating units called monomers, polymers are vast molecules. The process of polymerization is what typically forms a polymer, giving it physical and chemical characteristics that are different from its individual monomers. [11]. Polymers can be classified according to their source and preparation methods into [12] . Natural polymers [13,14]. Semi-synthetic polymers [15,16]. Synthetic polymers [17]. Polymers can also be classified based on the structural structure of the molecular chain, as this

classification depends on how the monomers are arranged and linked within the polymer chain [18]:
Linear polymers [19], Branched polymers [20], Cross-linked polymers [21],and Network Polymers [22]. Hydrogel polymers are among the most prominent types of Network polymers, and therefore, they will be discussed in this chapter due to their increasing importance in environmental remediation fields. They are three-dimensional, water-insoluble polymer networks characterized by their ability to absorb and retain large amounts of water. Their insolubility is due to the presence of physical or chemical entanglements between the polymer chains, which are formed either through strong and permanent covalent bonds or through non-covalent interactions [23, 24].
Figure (1) shows a comprehensive classification of hydrogel polymers based on multiple criteria including source, response, crosslinking, biodegradability, preparation, and ionic charge [25, 26].



Scheme (1): Classification of gel polymers based on their different properties

EXPERIMENTAL

APPARATUS

All absorbance measurements were performed using a Biochrome Libra S60 dual-beam spectrometer, used to measure the samples used in this study. A Pioneer analytical balance (Ohaus PA214) was also used. The wavelength of maximum absorption was measured using a Shimadzu UV-1700 spectrometer. The nanopolymers were prepared using the following instruments: a multi-function ultrasonic thermal cleaner, USC-M series, made in Korea, a homogenizer (supplied in Korea and USpicydownlight), and an 18-watt CRESCENT USND-1801 LED lamp, USA. Characterization was performed The study of the material properties included the application of a set of integrated analytical techniques such as FTIR, SEM, TGA, DSC, and TEM..

MATERIALS USED OR COMPOUNDS

All solutions were prepared using distilled water. used in this study, and the materials were of high purity except for the 6-MBTAMP reagent, which was prepared using alcohol solution.

How to prepare standard stock solutions

Preparation of Pb(II) Solution

A crude lead(II) concentrated solution of 100.00 mg/L was prepared by dissolving 0.0159 g of lead nitrate in 100.00 ml of distilled water. A few drops of nitric acid concentrated were added to facilitate the dissolution process. Working solutions of this crude solution were then prepared by subsequent dilution [27].

Preparation of the New Organic Reagent Solution (6-MBTAMP)

A crude solution of the new organic reagent, 6-MBTAMP (g/mol = 315.10 g/mol), was prepared at a concentration of 1×10^{-3} mol/L by dissolving 0.0315 g of the reagent in 100.00 mL of ethanol. Diluted working solutions were then prepared as needed [28].

Interfering solutions

Solutions containing all the interfering ions are prepared in 100 mgL^{-1} concentration, by dissolving amounts(0.0163g of $CdCl_2$),(0.0208g of $ZnCl_2$),(0.0268g of $CuCl_2.2H_2O$),($Bi(NO_3)_3$), in distilled water and completing the volume to 100 mL.

Synthesis and Characterization of(PLA-CO-AA-CO-MC)

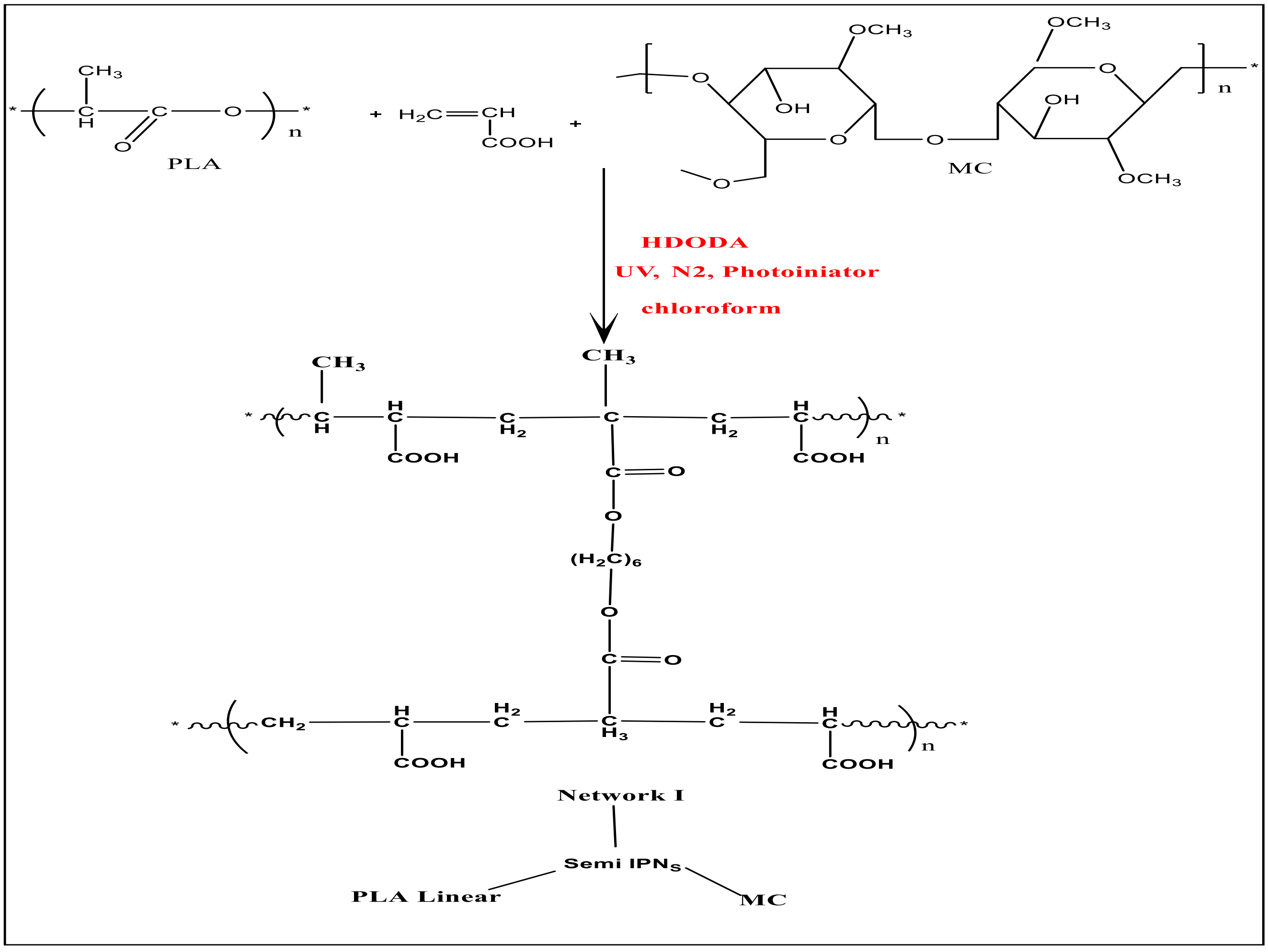
The polymer (PLA-CO-AA-CO-MC) was prepared by reacting 0.2 g of methyl cellulose with 4 ml of acrylic acid, adding 0.2 g of Poly Lactic Acid dissolved in 12 ml of chloroform, adding 0.6 ml of the cross-linking agent HDODA and 0.05 g of the photocatalyst 1-hydroxycyclohexyl phenyl ketone, with nitrogen pumped during the reaction to remove any dissolved oxygen for 30 minutes at room temperature. Then, the mixture was poured into polypropylene molds and exposed to UV light at a wavelength of 365 nm for minutes until it solidified as shown in Figure (2).

SAMPLES COLLECTION

Various water samples were collected from multiple sources, including tap water, well water, fish pond water, and wastewater. All samples, except tap water, underwent a preliminary filtration process to remove suspended impurities. The pH of all samples was then adjusted to neutral (pH = 7). Following this preliminary treatment, the recovery rate of lead(II) ions in each sample was estimated to study the efficiency of extraction or analysis in different aqueous matrices

Preparation steps of a new Nano polymer

The new nanopolymer was prepared according to the scheme (2)



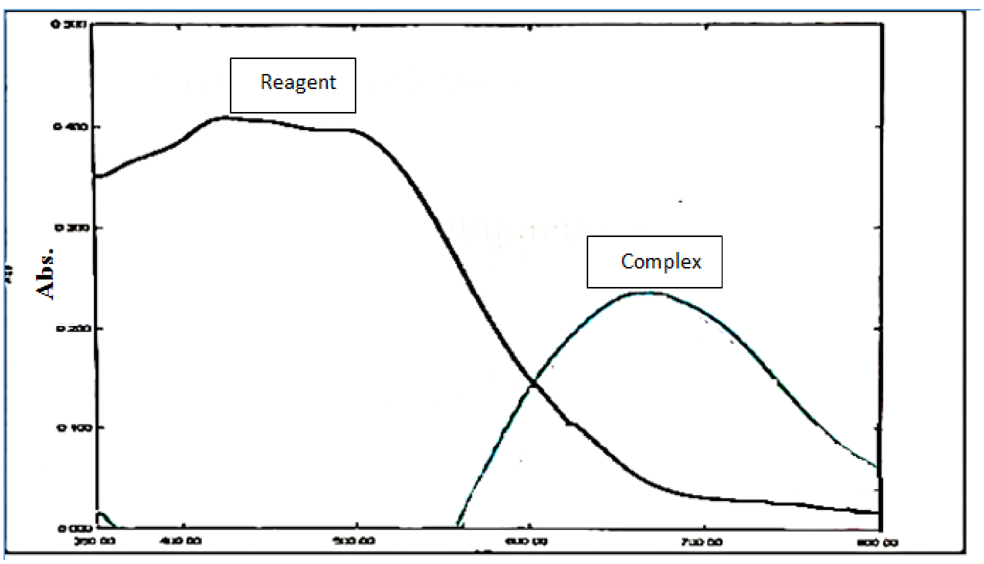
Scheme (2): Synthesis reaction of (PLA-CO-AA-CO-MC)

RESULTS AND DISCUSSION

This study focused on developing a sensitive spectroscopic method to estimate Pb(II) ions at aqueous solutions utilizing a PLA-CO-AA-CO-MC nanopolymer. The optimal conditions for this process were determined by extrapolating the influencing factors. The maximum absorption wavelength (λ max) was found to be 670 nm, while the optimal solution pH (pH) was 7.

The methodology involved using 6-MBTAMP reagent to form a colored complex with lead ions, allowing for absorbance measurements. Optimal volumes of reagent (0.5 mL) and lead(II) ion solution (2 mL) were used to ensure maximum reaction efficiency.

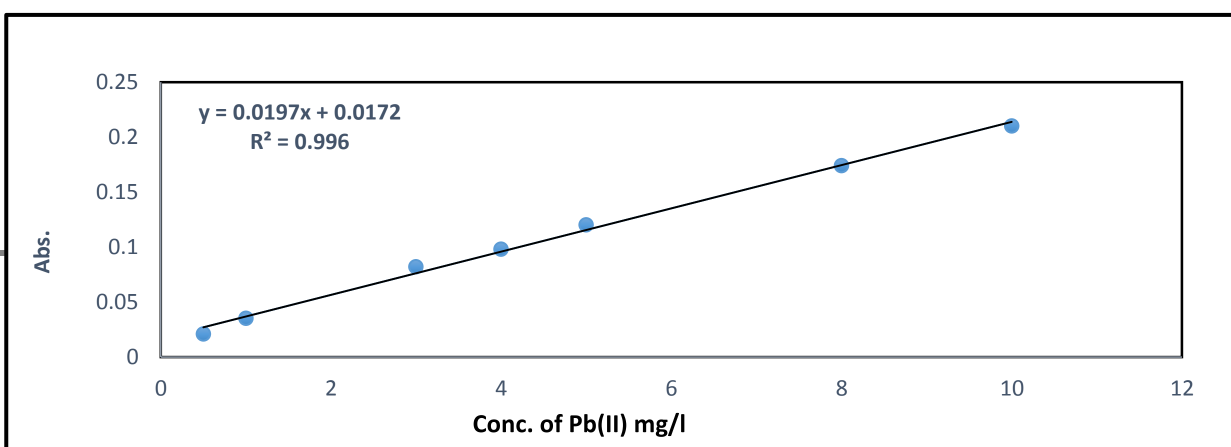
To ensure the accuracy of the results, the Spectrophotometric Reference Method was applied as a standard. This method allowed for the calculation of the amount of ions remaining in the solution, providing an accurate assessment of the efficiency of the quantification process. as shown in Figure(1)



Figure(1): The wavelength of maximum absorption Determining of Pb(II) complex with 6-MBTAMP as reagent

Standard calibration curve of lead(II) complex before the adsorption process

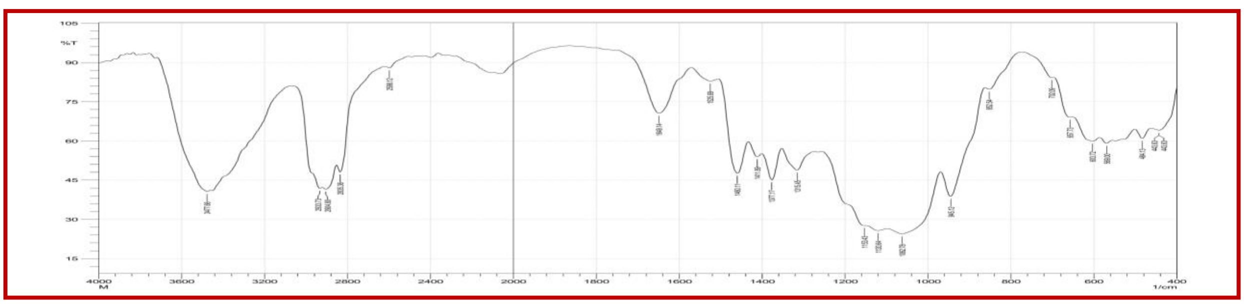
A calibration curve was prepared at a wavelength of 670 nm **before the adsorption** process, by preparing and measuring the absorbance for a series of concentrations (0.5 - 10) mg/L as shown in Figure(2).



Figure(2):Standard calibration curve of lead(II) complex before the adsorption process

INFRARED SPECTRUM

The PLA-CO-AA-CO-MC nanopolymer, methylcellulose, acrylic acid, and polylactic acid were characterized using Fourier transform infrared spectroscopy. A broad peak of carboxylic OH at(3122.75- 3475.73) cm^{-1}, C=O ester at 1737.66 cm^{-1}, and carboxylic C=O at 1712.79 cm^{-1} appeared for_the nanopolymer. In contrast, a narrow peak of alcoholic OH at 3477.66 cm-[1], aliphatic CH at 2933.73 cm^{-1}, and etheric C-O at 1163.43 cm^{-1} appeared for methylcellulose. Meanwhile, acrylic acid showed a broad peak of carboxylic OH at 3518.16 cm^{-1}, carboxylic C=O at 1747.51 cm^{-1}, and alkene C=C at 1635.64 cm-[1], while poly Lactic acid showed an ester group at 1774.51 and an aliphatic CH at 2970.38 as shown in Figure, 3,4,5,6.



Figure(3) FTIR of the nanopolymer

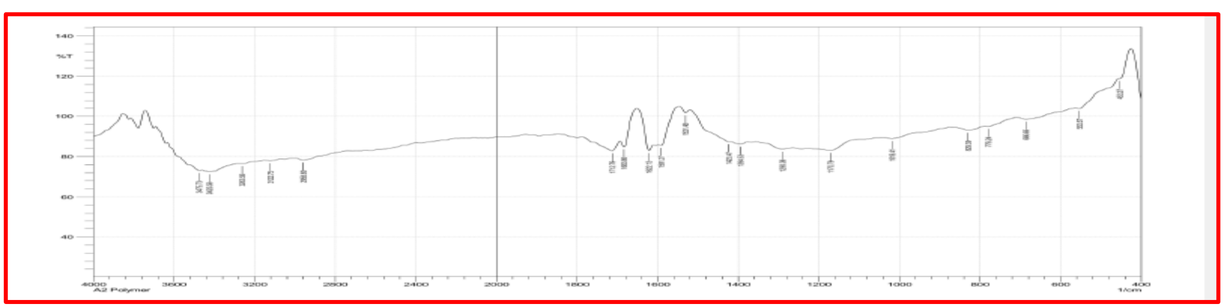


Figure (4): FTIR of the methylcellulose

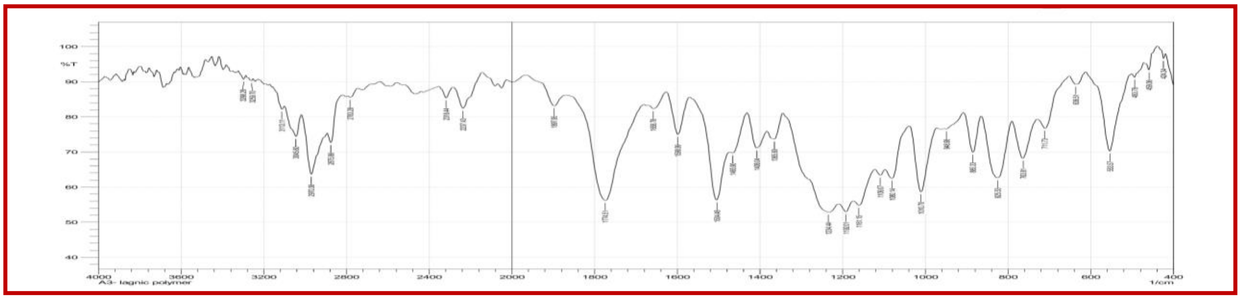


Figure (5): FTIR of the acrylic acid

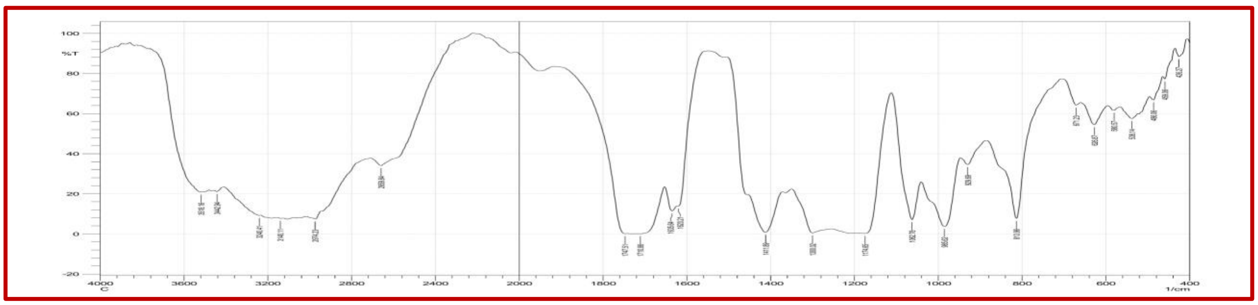
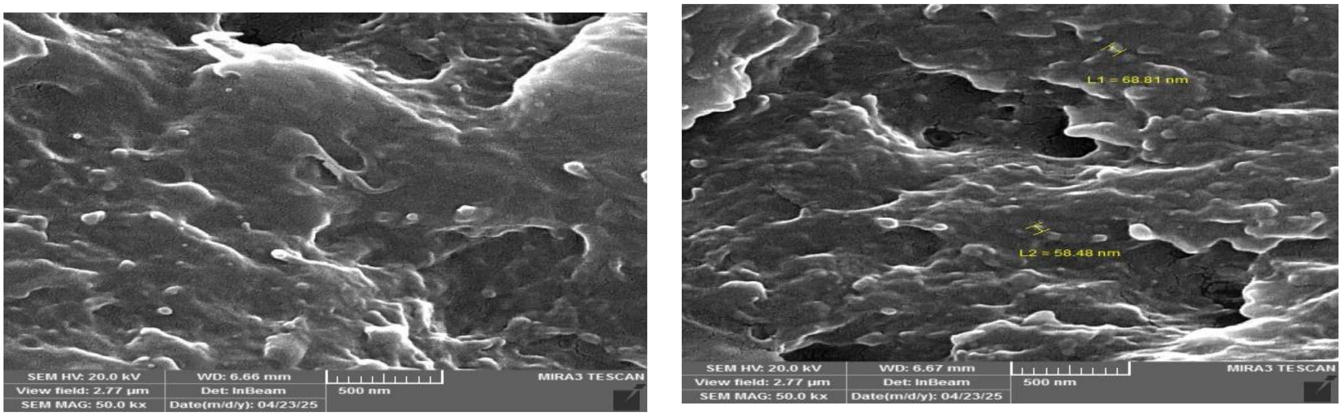


Figure (6):FTIR of the poly Lactic acid

Field Emission Scanning Electron Microscopy (FE-SEM)

The surface was examined using a scanning electron microscope (SEM). topography of the research material. This examination enabled the characterization of the overall shape and surface morphology from the prepared material nanopolymer. From the results, it can be noted that the nanopolymer formed from (PLA-CO -AA-CO-MC) consists of spherical and homogeneous nanoparticles, with an average diameter of approximately 63.645 nm. The accumulation of these particles contributed to the filling of the porous channels, resulting in a more cohesive structure. The microscopic images also revealed a clear network and a smooth surface extending across the entire structure. As shown in Figure(7) , the porous surface was visible in the polymers, particularly in



the MIP polymer**.**

FIGURE (7): FESEM micrograph of (PLA-co-AA-CO-MC) Transmission Electron Microscopy

Transmission Electron Microscopy(TEM)

Transmission electron microscopy (TEM) is a pivotal analytical tool in advanced microscopic studies, enabling the detection of the internal structure of materials with extreme precision. This technique relies on directing a beam of very high-energy electrons passing through an extremely thin specimen, typically 100 nanometers or less in thickness.This method aims to investigate the structural properties of materials at the atomic and nanoscale, enabling researchers to understand the relationship between structure and function in various scientific fields(29)as shown in Figure(8).

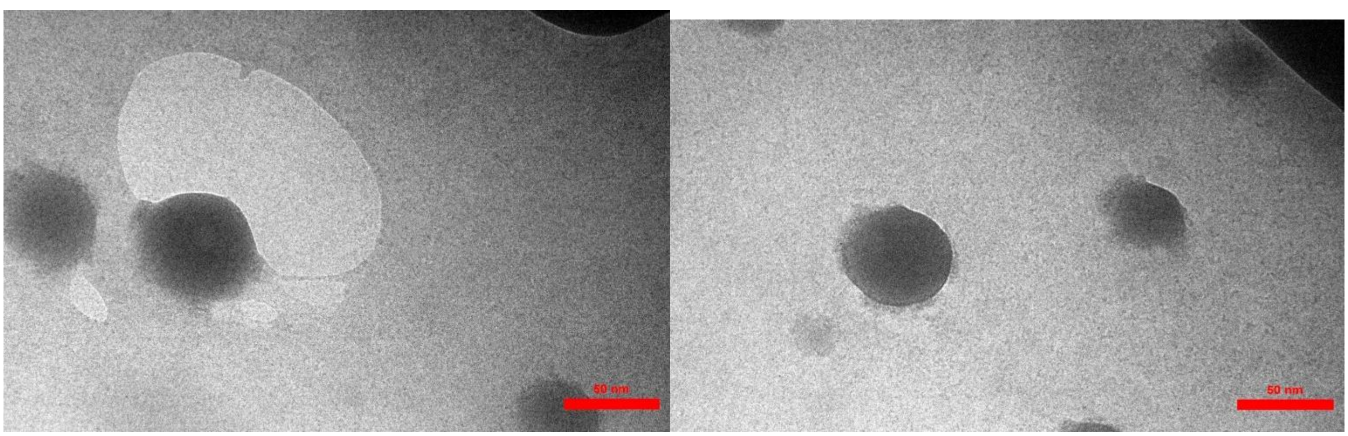


Figure (8): Transmission Electron Microscopy(TEM)

Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) techniques:

Differential thermal analysis (DSC) is a fundamental technique that involves subjecting a sample to controlled thermal cycles of heating and cooling. The results can be interpreted by analyzing the peaks shown in the Figure(9,Endothermic peaks: These peaks represent processes that absorb energy, such as melting, evaporation, and glass transitions.Exothermic peaks: These peaks represent processes that release energy, including crystallization and chemical reactions.In contrast, thermogravimetric analysis (TGA) measures the change in sample mass as a function of temperature, expressed as the percentage weight loss(30).

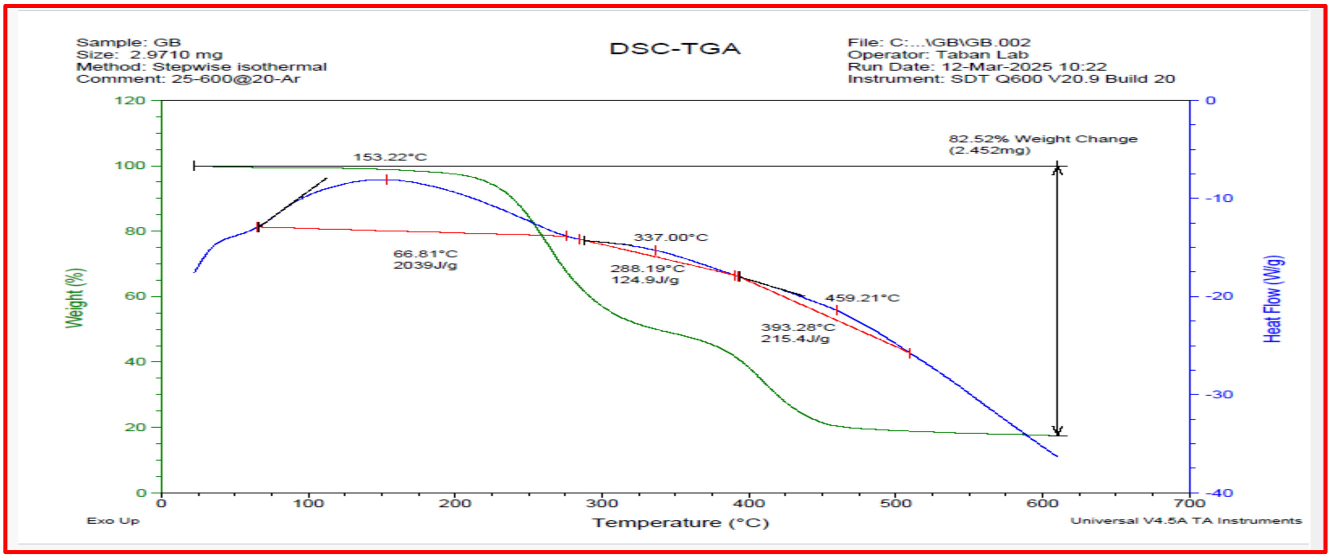


Figure (9): Analysis using TGA and DSC techniques

Swelling ratio : A sample of the prepared polymer with a weight of 0.3450 was placed in 100 ml of distilled water for seven hours. The swelling ratio As in Figure(10)was measured for each hour after drying it from excess water using filter paper, using the following relationship(31):

$$SR=M-M^0/M^0 *100\%$$

Where M = the mass of the swollen hydrogel. , M^0 = the mass of the hydrogel before swelling.

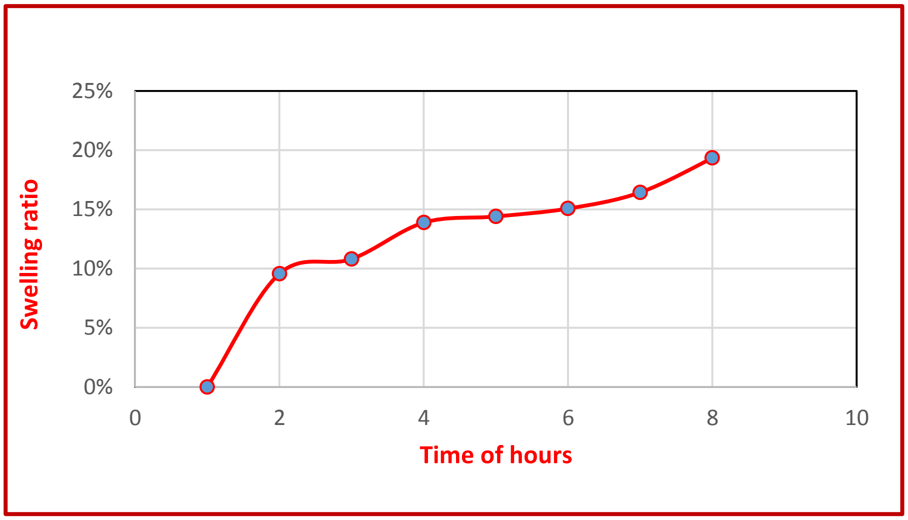


Figure (10): Swelling ratio of (PLA-CO –AA-CO-MC) polymer

Study of the optimum conditions for the adsorption process and estimation:

Study of the effect of pH on the adsorption process.

The effect of pH on the adsorption process was studied, where the weight of the adsorbed surface was 0.01, the shaking time was 5 minutes, the lead(II) ion concentration was 20 $mg.L^{-1}$, the solution volume was 5 ml, and the pH was (3, 5, 7, 9). It was found that the best adsorption occurred at pH 7, as it yielded the highest recovery rate, as shown in Figure (11)

Study the effect of shaking time on the adsorption process

A study was conducted to evaluate the impact of shaking time on the adsorption efficiency of lead(II) ions at a concentration of 20 $mg.L^{-1}$ using 0.01 g of adsorbent. Shaking time ranged from 5 to 15 minutes at a pH was 7 of the medium. It was observed that increasing the shaking time to 10 minutes resulted in achieving the maximum adsorption rate, as shown in Figure (12)

Study the effect of the mass of the adsorbed surface on the adsorption process

The effect of different masses of the nanopolymer (PLA-CO-AA-CO-MC), within the range of 0.001 to 0.2 g, on lead(II) ion adsorption was studied. 0.1 g was found to be the optimal mass that achieved the highest ion removal rate under the same conditions as the previous two experiments. Figure (13)

Study the effect of solution volume on the adsorption process

The effect of solution volume on the adsorption rate was studied, with solution volumes of 3, 5, and 10 mL. The optimal volume of 3 mL was chosen to achieve a higher recovery percent as shown Figure(14).

Study the effect of Pb(II) ion solution concentration on the adsorption process.

Under the optimal conditions for study, a calibration curve(Figure(15) was obtained at a wavelength of 670 nm after the adsorption process for a series of lead(II) ion concentrations, from the result the linearity was 1 to 10 $mg.L^{-1}$ and Sandell's sensitivity was 0.1666 $\mu g.cm^{-2}$, molar absorptivity(ε) was $1.2432\times10^{3} L.mol^{-1}.cm^{-1}$.

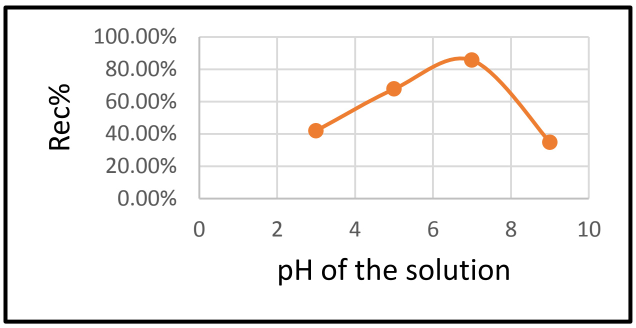


Figure 11. The effect of pH on the adsorption process

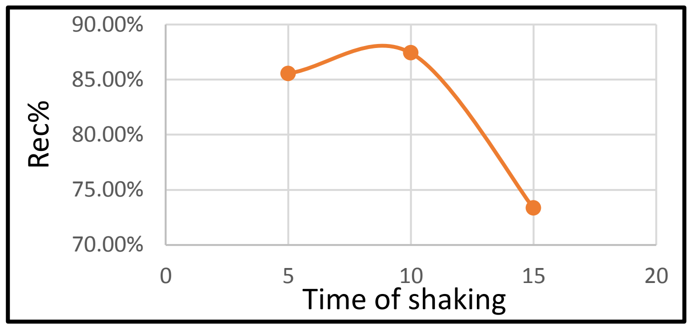


Figure 12: Effect of shaking time on the adsorption process

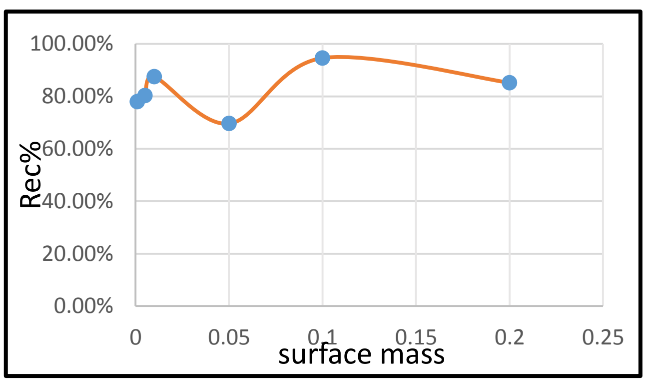


Figure 13. the effect of the mass of the adsorbed surface on the adsorption process

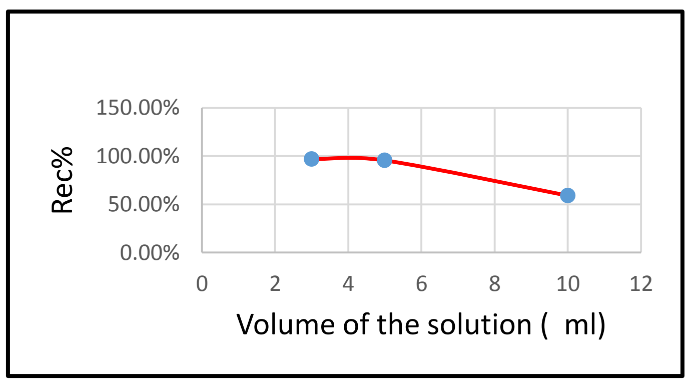


Figure 14. the effect of solution volume on the adsorption process

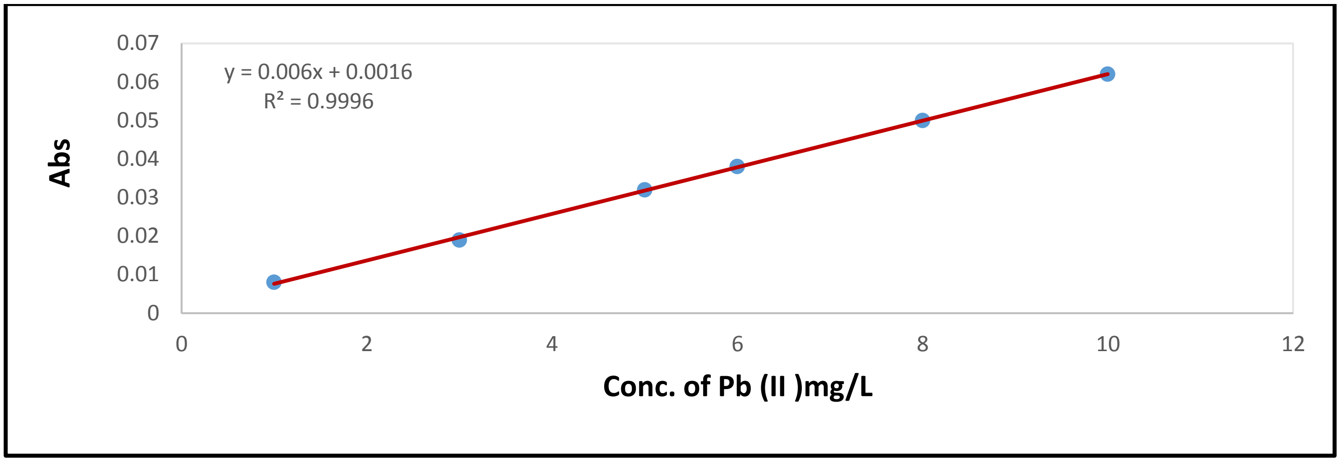


Figure 15: Calibration curve after adsorption to estimate the remaining lead(II) ion

Interferences:

The effect of zinc (II), copper (II), cadmium (II), and bismuth (III) ions on the removal efficiency of lead (II) from an aqueous solution was studied. These interactions were evaluated at two different concentrations of interfering ions, 10 mg/L and 50 mg/L, while the lead ion concentration was fixed at 10 mg/L.

The results showed that the studied ions had no significant effect on the lead ion removal process, The chemical was tested at varying concentrations to observe its effect concentrations. This indicates the absence of significant competitive interactions between the studied cations and lead ions at the reaction sites, confirming the selectivity of the analytical method used and demonstrating its effectiveness in the determination of lead even in the presence of other ions.

Applications

To evaluate the sorbent's efficiency under realistic environmental conditions, four real water samples were tested: tap water, lake water, well water, and wastewater.

This experiment was conducted under pre-determined optimal conditions, aiming to measure the sorbent's ability to remove lead (II) ions from complex matrices. Although the lead concentrations in the samples were low, the flame atomic absorption (FAA) technique was used to ensure accurate measurements and determine the actual lead ion concentration.These applications demonstrate the sorbent's effectiveness in removing lead from various water sources, demonstrating its potential for environmental remediation.

Conclusion

In this study, a new nanopolymer was prepared by combining acrylic acid (AA), methyl cellulose (MC), and polylactic acid (PLA), using a photopolymerization technique. These components were selected for their dual properties, combining hydrophilic and hydrophobic monomers. The chemical structure of the prepared nanopolymer was verified using a range of available analytical techniques.The efficiency of the prepared nanopolymer as an adsorbent for the removal of lead(II) ions,which are classified as hazardous environmental pollutants, was evaluated. The results demonstrated that this method is highly effective, rapid, easy to implement, and accurate for environmental remediation applications.

TABLE 1. Recovery value of pb(II) in different water samples

Sample	True value mg.L^{-1} by FAA	Measured value mg.L^{-1} By suggested method	E%	Recovery %
Tap water	0.001	Nil	……	……
Lake water	0.041	0.040	-2.43	97.56
Well water	0.0205	0.019	-7.31	92.68
Drainage water	0.790	0.800	1.26	101.26

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