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RESEARCH ARTICLE

Determination of Pb(II) Ion with a Novel, Highly Efficient Immobilized Arsenazo III in a Polymer Matrix

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ABSTRACT

In this study, a novel analytical reagent based on arsenazo III immobilized on a polymer matrix composed of polyacrylonitrile, polyethylene polyamine, and dichloroethane (PPD) was developed and applied for the spectrophotometric determination of Pb(II) ions. The proposed method demonstrated standard analytical and metrological characteristics for the highly accurate determination of Pb(II) ions, as evidenced by a relative standard deviation (Sr) of 0.06 and a minimum detectable concentration (C_{min}) of 0.24 μg/L. The accuracy and reproducibility of the method were evaluated using Student's *t*-test and Fisher's *F*-test. Comparison of the experimental and tabulated values (experiment=1.09; t_{table}=2.83, $F_{experiment}$=2.52; Ftable=4.47) confirmed the reliability of the proposed analytical procedure. At pH 5.8 in an acetate buffer medium, the Pb(II)–arsenazo III complex immobilized on the PPD matrix exhibited a maximum analytical signal at a wavelength of 640 nm. The measurement results followed the Beer–Lambert–Bouguer law, showing a linear relationship in the Pb(II) concentration range of 0.04–2.4 μg/mL. The method is simple, rapid, cost-effective, and allows direct Pb(II) ion determination in water samples with high sensitivity and selectivity.

Keywords: Beer-Lambert-Beer law, Highly selective sorption spectrophotometry, Lead detection, PPD sorbent, Water samples

Introduction

Lead (Pb) is one of the most commonly detected heavy metals in contaminated water sources, frequently exceeding the World Health Organization (WHO) guideline of 10 μg/L, which poses severe ecological and health risks.[1,2] Recent studies have focused on developing highly selective and sensitive methods for Pb(II) detection.[3,4] Flor de Liss Meza Lópe et al was use select three functional monomers—acrylic acid (ACRY), 2-acrylamido-2-methylpropane sulfonic acid (AMPSA), and allylamine (ALLY)—for the design of ion-imprinted polymers (IIPs). Adsorption studies under optimized conditions demonstrated that IIP-AMPSA exhibited the highest Pb^{2+} adsorption capacity (51.84 mg g^{-1}), outperforming IIP-ACRY (31.02 mg g^{-1}) and IIP-ALLY (12.90 mg g^{-1}), consistent with the computational predic-

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tions.[5,6] Ashirova, M. A. et al. studied sulfarsazen (SFN) immobilization on polyethylene polyamine-modified polyacrylonitrile (PPA) analytical matrix for Hg(II) determination.[7–9] Optimal analytical conditions were observed at pH 9.8 and 520 nm, with a linear range of 2–42 mg L^{-1}, a detection limit of 0.4 mg L^{-1}, and an adsorption efficiency of ~98%, SFN/PPA matrix demonstrated strong selectivity even in the presence of common interfering ions (Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+}, Na^{+}, Cl^{-}, I^{-}), enabling accurate Hg(II) determination in wastewater samples.[10–12]

The aim of the work is to immobilize arsenazole III on a fibrous sorbent and to develop a new economical, rapid sorption-spectrophotometric method for the determination of lead ions using immobilized arsenazole III, with application to the determination of Pb(II) ions in natural objects.

Materials and methods

Materials

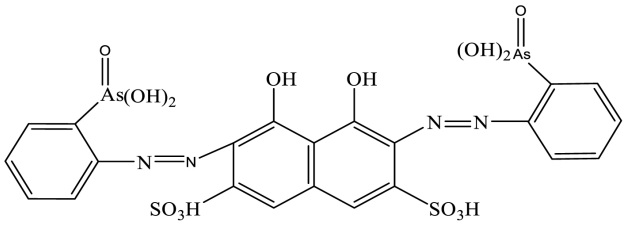
All chemicals used in this study, including polyacrylonitrile, polyethylene polyamine, arsenazo III(3,6-bis-[(2-arsonophenyl)azo]-4,5-dihydroxy-2,7-naphthalenedisulfonic acid) and 1,2-dichloroethane, $Pb(CH_3COO)_2{\cdot}3H_2O$ salt were obtained from Sigma-Aldrich MilliporeSigma (Merck Group) and Ximreaktivinvest LLC, and were used without further purification.

Methods

IQ analysis- In order to determine the reaction mechanisms of the immobilized compounds, as well as the resulting complex formation, IR spectra were recorded on a Bruker Fourier spectrometer (Invenio S-2021, in the range of 4000–400 cm^{-1}. During the research, a pH meter called “BANTE pH meter-210” was used to measure the optimal environments of solutions. In the solution medium, optical densities and wavelengths of the reagent and complex were measured by absorption spectroscopy using an EMC-30PC-UV spectrophotometer. In the sorption determination, the optimal conditions for complex formation were found using an X-Rite Eye One Pro mini spectrophotometer.

Experimental part

In this research work, a 0.02% solution of the organic reagent 3,6-bis-[(2-arsonophenyl)azo]-4,5-dihydroxy-2,7-naphthalenedisulfonic acid ($C_{22}H_{18}As_2N_4O_{14}S_2$, Mr = 776.37 g/mol) was prepared for the determination of lead (II) ions. Preparation of the



Scheme 1. Molecular structure of the analytical reagent (3,6-bis-[(2-arsonophenyl)azo]-4,5-dihydroxy-2,7-naphthalenedisulfonic acid).

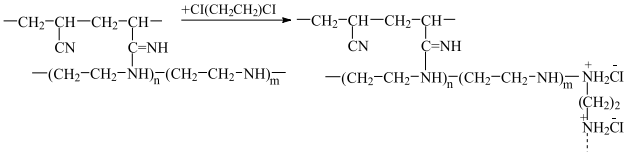
standard lead (II) solution: To prepare a standard 1 × 10^{-4} M solution of $Pb(CH_3COO)_2{\cdot}3H_2O$, 0.0325 g of the dry salt was weighed into a 200 mL volumetric flask using an analytical balance with an accuracy of 0.0001 g, and then dissolved in distilled water up to the mark of the flask Scheme 1.

Buffer solutions

Acetate buffer solution was used to provide the medium for the solutions. To prepare a pH=5.5 solution, 100ml of 1N CH_3COOH and 50ml of 1N NaOH were added to 500ml flasks, and to prepare a pH=5.8 solution, 79.5ml of CH_3COOH and 50ml of NaOH were added and made up to the mark with distilled water.

Selection of a polymer carrier sorbent

For lead salt and Arsenazo III reagent, PPD sorbent was used, which is obtained by sequentially chemically modifying polyacrylonitrile in the presence of polyethylenepolyamine and dichloroethane. The synthesis reaction of the sorbent is shown in Scheme 2 below:



Scheme 2. Synthesis reaction of PPD (Polyethylenepolyamine) sorbent.

In this study, PPD fiber was used as the carrier sorbent, and 0.2 g was taken and exposed to a standard solution of 0.1 molar HCl hydrochloric acid for 24 hours to activate the sorbent and neutralized with distilled water.

Immobilization process into a polymer matrix

The mechanism of immobilization of the selected arsenazo III analytical reagent on polymer supports is presented below. First step. PPD polymer was activated in 0.1 N hydrochloric acid (~R-NH_3 + Cl-). In this process, 0.2 g of polymer support was placed in

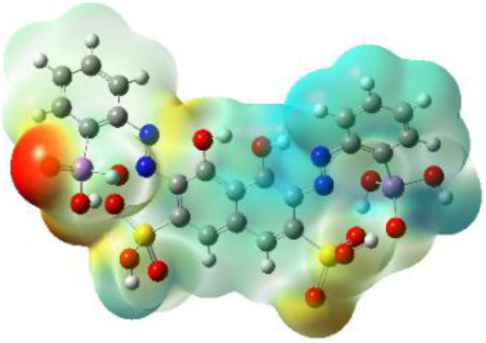


Fig. 1a. Arsenazo III electrostatic potential surface.

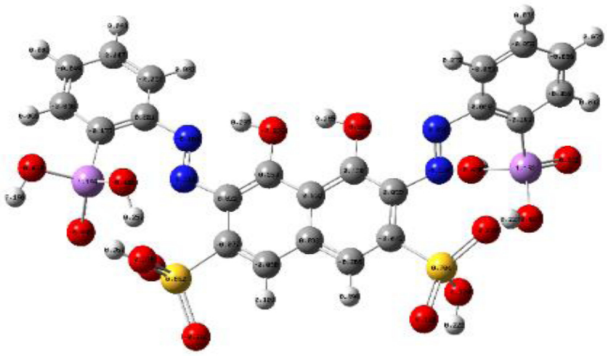


Fig. 1b. Electron cloud density of arsenazo III.

a 25 ml flask, then 10 ml (0.1 N) HCl solution was added, and the resulting solution was activated at room temperature for 1 day. Then the support matrix was washed with distilled water to a neutral state (pH=7). As a result, $\sim R\text{-}NH_3^+Cl^-$ was formed.

$$\sim P\text{-}NH_2 + HCl \rightarrow \sim P\text{-}NH_3^+ + Cl^-$$

The second step is to obtain an immobilized form of the carrier polymer using the Arsenazo-3 analytical reagent. In this process, 10.0 mL of a 2.57×10^{-4} M (or 0.02%) Arsenazo-3 solution and 0.2 g of the ionic form of the carrier polymer ($\sim P\text{-}NH_3^+Cl^-$) are added to a 25.0 mL flask, after which the mixture is stirred for 9–10 minutes. The carrier matrix immobilized with Arsenazo-3 ($\sim R\text{-}NH_3^{+-}O_3S\text{-}R$) is then washed with distilled water and dried at room temperature. During this immobilization process, the sulfo functional groups of the Arsenazo-3 reagent form bonds with the amino functional groups of the sorbent through electrostatic interactions.

$$\sim P\text{-}NH_3^+Cl^- + HO_3S\text{-}R \rightarrow \sim P\text{-}NH_3^{+-}O_3S\text{-}R + HCl$$

Third stage The process of complexing the lead ion with the arsenazo III ion chemically sorbed to the polymer matrix in a static process. The reaction of lead (II) with the arsenazo III reagent in $\sim R\text{-}NH_3^{+-}O_3S\text{-}R$ was carried out as follows: a $1*10^{-4}$ M standard solution of lead (II) was mixed with the immobilized carrier polymer ($\sim R\text{-}NH_3^{+-}O_3S\text{-}R$) in a flask; as a result, $\sim R\text{-}NH_3^{+-}O_3S\text{-}R\text{-}Pb$ was formed. Pb (II) ions are strongly bound to the hydroxyl functional groups through an ionic bond and to the –N= functional group through a coordination bond. [13,14]

Results and discussion

Quantum chemical calculation

Electrostatic potential surfaces are important in identifying the reaction centers of molecules, especially in noncovalent interactions. These surfaces indicate the nucleophilic and electrophilic centers of molecules. In Figs. 1a and 1b, the red dots represent regions with a high electron cloud density and a negative electrostatic potential, i.e., regions with more electrons. The purple centers represent regions with a positive potential and fewer electrons. [15]

Electrostatic potential.

On the electrostatic potential surface of arsenazo III, Figs. 1a and 1b, red spherical centers represent minima, and purple spherical centers represent maxima. Large maxima and small minima are visible in the figure. White spherical points represent minima, and red spherical points represent maxima. Electrostatic potential analysis showed that the coupling reactions proceed with the help of -OH and -N= groups.

It was found that the highest electron cloud density of the arsenazo III reagent is O-0.758, which belongs to the SO_3H group. Therefore, it can be assumed that the immobilization of the arsenazo III reagent on the fibers occurs through the sulfo (SO_3H) group of the reagent Fig. 1b.

HOMO-LUMO.

In computational chemistry, HOMO (Highest occupied molecular orbital) and LUMO (Lowest unoccupied molecular orbital) are key concepts in analyzing the electronic structure of a molecule. They describe the highest occupied and lowest unoccupied orbitals in a molecule.

HOMO-LUMO energy gap: The energy difference between HOMO and LUMO is important in determining the reactivity and stability of a molecule. This difference is also called the energy gap. A small HOMO-LUMO gap makes a molecule reactive towards chemical reactions, while a large gap increases stability Fig. 2a and b.

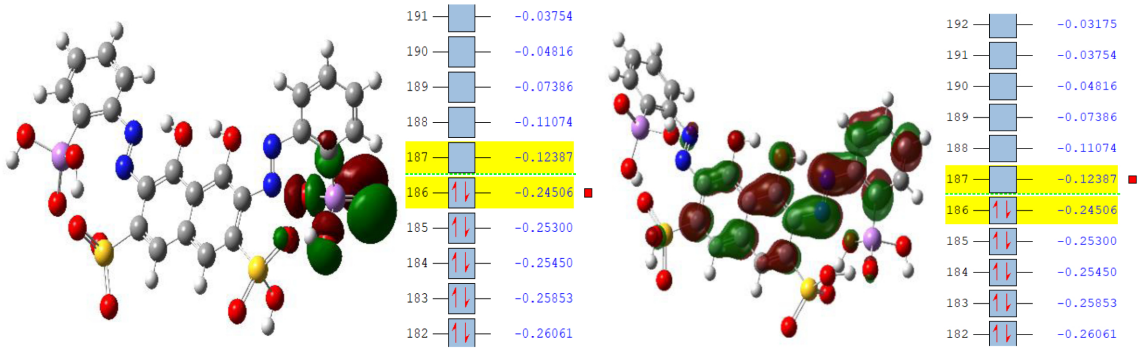


Fig. 2. a) The HOMO, the highest occupied molecular orbital of arsenazo III; b) The LUMO, the lowest unoccupied molecular orbital of arsenazo III.

Reactivity:

1. If it is between 0–2 equiv, it reacts easily and quickly
2. If it is between 2–4 equiv, it reacts under certain conditions
3. If it is greater than 4 equiv, it is inert
4. Soft if it is less than 1 equiv, hard if it is greater than 1 equiv
   EL=–0.12387 equiv E_H=–0.24506 equiv
   $\Delta E=E_L-E_H$=–0.12387–(–0.24506) = 0.12119 equiv

Conclusion: the substance reacts easily, and it is soft.

Spectrophotometric analysis.

The EMC-30PC-UV-1800 spectrophotometer was used to measure the maximum absorption of the Arsenazo III reagent. Measurements were taken against a reference solution of Arsenazo III, and the absorption values were compared. The maximum absorption wavelength of the Arsenazo III reagent was observed at 540 nm. This information was important for determining the maximum absorption wavelengths of the reagents, and subsequent experiments were based on changes in these wavelengths. [16]

Optimal carrier

To select the optimal carrier, 0.2 g of several types of sorbents was weighed on an analytical balance with an accuracy of 0.0001 g and treated with a 0.1 N HCl standard solution. The matrices were then washed, dried, and stored in special Petri dishes. The carriers were placed in a glass, and 0.02% solutions of the selected organic reagent, Arsenazo III, were added to them. The optical density of the solutions before and after immobilization was measured using an EMC-30PC-UV/UV-5100 UV-VIS spectrophotometer. After immobilizing the selected activated carrier with the organic reagent under optimal conditions, their spectra were measured using an X-Rite Eye-One Pro reflectance spectrophotometer, calibrated against the original polymer matrices based on a $BaSO_4$ matrix. The results are presented in Figs. 3 and 4. [17] The reflectance spectra of the best immobilized organic reagents on the sorbents were measured and analyzed using an X-Rite Eye-One Pro Mini spectrophotometer. The results are presented in Fig. 3 and Fig. 4.

From Fig. 3, which depicts the light reflectance spectra, it is evident that the PPD carrier exhibited the highest reflectance change upon immobilization of Arsenazo III, indicating more effective binding of the reagent compared to other carriers. This superior performance can be attributed to the presence of reactive amino groups in the PPD polymer, which form strong electrostatic interactions and hydrogen bonds with the sulfonic and hydroxyl groups of Arsenazo III. In contrast, carriers such as SMA2 and PPM showed comparatively lower reflectance changes, suggesting less efficient immobilization due to a reduced number of available functional groups or steric hindrance in the polymer matrix. Fig. 4, which represents the Kubelka-Munk transformed reflectance spectra, corroborates these observations. The Kubelka-Munk function allows a more quantitative comparison of the amount of immobilized reagent. Here, PPD again demonstrates the highest signal, confirming that it provides the most favorable environment for Arsenazo III attachment. Ipak fibroin and PPA carriers show moderate immobilization, likely due to partial accessibility of reactive sites or differences in surface area. PPF shows better immobilization than SMA2 and PPM but remains inferior to PPD.

The data suggest that the chemical composition and functional group availability of the carrier play a critical role in reagent immobilization. PPD was selected as the optimal carrier because it ensured maximum reagent loading, strong interaction with

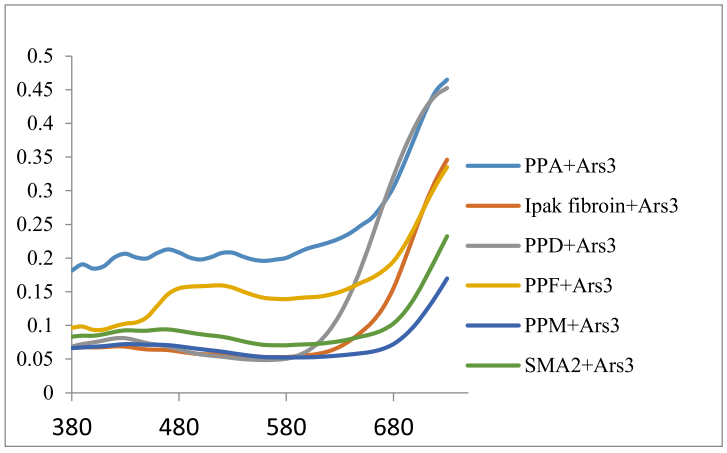


Fig. 3. Light reflectance spectra of Arsenazo III reagent immobilized on several types of sorbents.

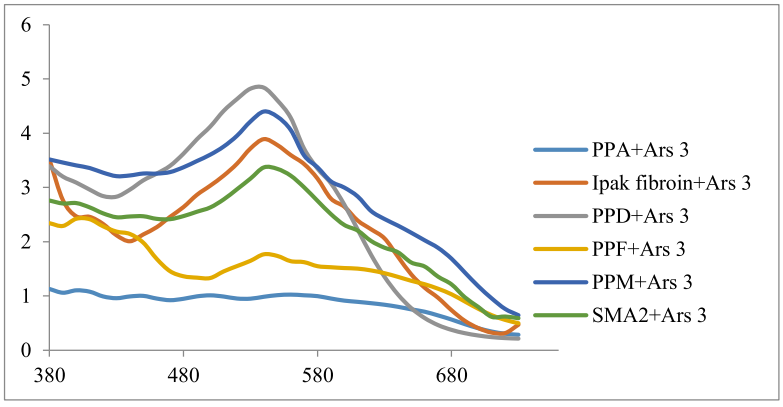


Fig. 4. Kubelka-Munka function representation of the reflectance spectra of arsenazo III reagent immobilized on several types of sorbents.

the reagent, and thus enhanced analytical sensitivity for subsequent Pb(II) ion determination.

Time dependence of reagent immobilization.

To study the time dependence of immobilization of the ADND-3 reagent on PPD fiber, the fiber was incubated in a $1*10^{-3}$ M solution of the reagents for 1–50 minutes, and the analytical signals were measured. The results obtained are presented in Fig. 5.

We can see from the graph above that 15 minutes is enough to completely immobilize the Arsenazo III reagent. Fig. 4 presents the kinetics of Arsenazo III immobilization on PPD fiber. The analytical signal increases rapidly within the first 15 minutes, after which it reaches a plateau, indicating complete immobilization. This rapid immobilization ensures that the method is efficient and time-effective, reducing waiting times while maintaining consistent analytical performance. In summary, the combined

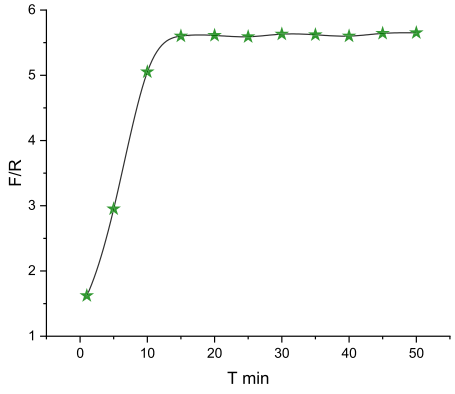


Fig. 5. Time dependence of reagent immobilization.

analysis of Figs. 3 and 4 demonstrates that the chemical composition and surface functionality of the carrier significantly influence the immobilization efficiency of Arsenazo III. PPD fiber not only provides the highest loading of the reagent but also ensures rapid and stable immobilization, making it

Table 1. Selection of optimal buffer solution.

R + Me	Buffer solution	pH 2	3	4	5	5,5	5,8	6	7	8	9
Arsenazo III + Pb^{2+}	Acetate (A)		-	0,18	0,35	0,68	0,8	0.54	-	-	-
	Universal(A)	-	0,15	0,35	0,43	0,46	0,5	0.48	-	-	-
	Phosphate(A)	-	-	-	0,25	0,28	0,32	-	-	-	-

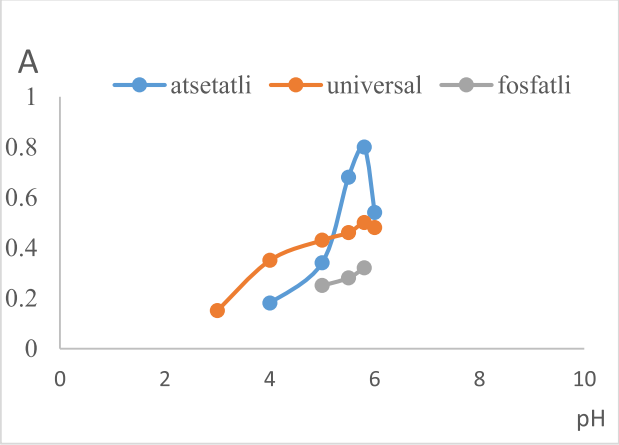


Fig. 6. Dependence of complex formation on the environment.

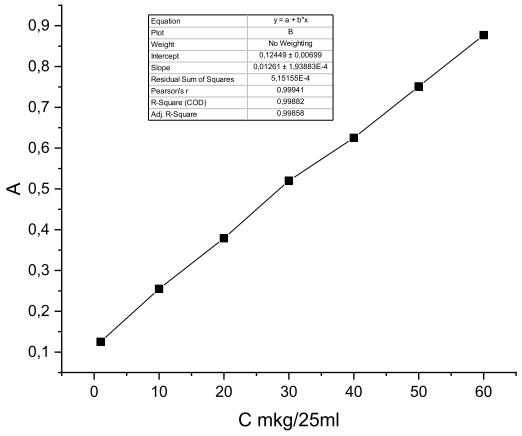


Fig. 7. Graph of the dependence of optical density on the amount of lead (II) ion added.

the most suitable carrier for the developed sorption-spectrophotometric determination of Pb(II) ions.

The dependence of complex formation on the pH of the medium and the nature of the buffer.

In a 25 ml standard volumetric flask, 2 ml of 0.001% aqueous solution of ADND-3 reagents, 5.0 ml of a buffer solution of known pH, and 1 ml of Pb^{2+} solutions (60 μg/ml) were added, and double-distilled water was added to the mark of the flask. The pH of the resulting solutions was redetermined using a PHS-3E pH meter (China), and the optical densities (λ_{max}=540, l=1.0 cm) were measured relative to standard reference solutions. The results obtained are presented in Fig. 6, and Table 1.

From Fig. 6 and Table 1, we can conclude that the complex formation with immobilized ADND-3 and Pb^{2+} ions leads to the formation of a stable complex between pH=5–6. The highest analytical signal was observed at pH=5.8. For the subsequent analysis processes, the acetate buffer solution was used at pH(Pb^{2+})=5.8 for the formation of lead complexes. [18,19]

Observance of the Bouguer-Lambert-Beer law.

Experiments were conducted to study the obedience of the Bouguer-Lambert-Beer law in the determination of lead (II) ions using immobilized arsenazo III salt reagents Fig. 7.

Fig. 7 shows that the Beer-Lambert-Boer law is valid for lead (II) ion concentrations in a 25 mL solution in the range of 1 to 60.0 μg/mL, with a deviation from a straight line observed at higher concentrations, which is large enough to detect metal ions in environmental objects.

Spectral characterization of the complex formation of lead(II) ion with immobilized arsenazo III

The blue curve corresponds to the free Arsenazo III reagent in aqueous solution, which exhibits a characteristic maximum absorption band at $\lambda = 540$ nm, associated with $\pi \rightarrow \pi^*$ electronic transitions within the azo chromophore. The red curve represents the Pb(II)–Arsenazo III complex formed at pH 5.8 in acetate buffer solution. Upon complexation with Pb(II) ions, a pronounced bathochromic shift of the absorption maximum to $\lambda = 610$ nm is observed, indicating strong coordination interactions between Pb(II) ions and the azo (-N=N-) and hydroxyl (-OH) functional groups of the Arsenazo III ligand. The significant shift in the absorption maximum and the increase in absorption intensity confirm the successful formation of a stable Pb(II)–Arsenazo III complex and provide the basis for selective spectrophotometric determination of Pb(II) ions in Fig. 8. [20,21]

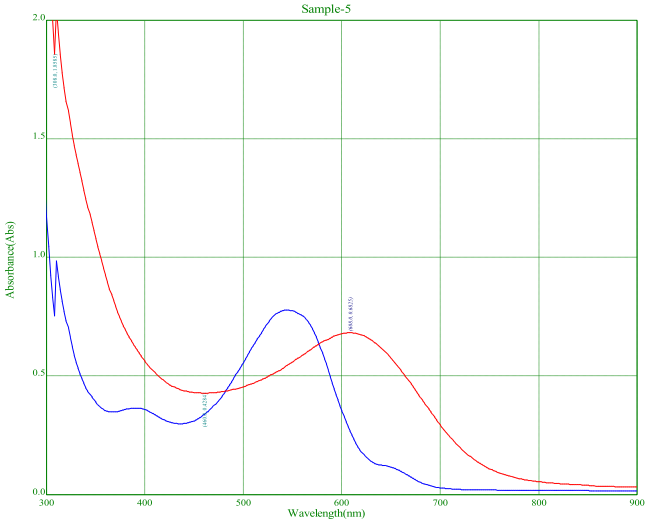


Fig. 8. Absorption spectra of ADND-3 (λp=540 nm) and its complex with lead (II) ion in solution (λk=610).

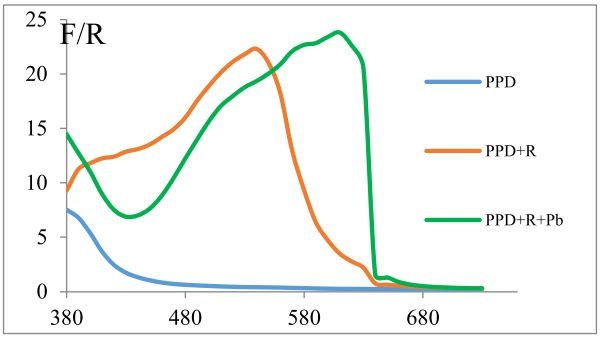


Fig. 9. The light reflection spectrum of the carrier sorbent PPD, arsenazo III immobilized on it, and the complex.

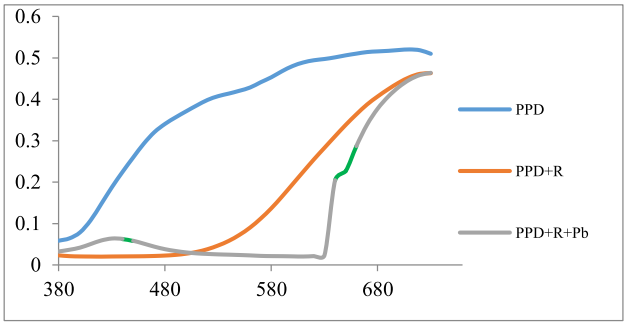


Fig. 10. Representation of the Kubelka-Munk function of the light reflection spectrum of the carrier sorbent PPD, arsenazo III immobilized on it, and the complex.

As shown in Fig. 9, the immobilized reagent exhibits significant changes in the light reflection spectrum after complex formation with Pb^{2+} ions. Fig. 10 demonstrates the variation of the KubelkaMunk function of the carrier sorbent before and after immobilization of the arsenazo III complex.

The increase in reaction contrast on a solid substrate indicates that the structure of the immobilized reagent becomes rigid due to steric hindrance, there are no rotational and vibrational movements, therefore, the analytical signal is maximum during immobilization Table 2.

IR spectrum analysis

The structure of the complex formed by lead (III) and the arsenazo III reagent immobilized on the polymer matrix and the mechanism of immobilization of the reagent on the fiber were determined using IR spectrometry. The infrared spectra of the complex were studied in the frequency range of 4000–400 cm^{-1} using an IR-Fourier-spectrometer "Bruker Invenio S-2021". To study the mechanism of immobilization of the arsenazo III reagent on the fiber, the IR spectra of the complex formed with the fiber, the reagent, and the metal ion of the immo-

Table 2. Spectral classification of the complex formed by the lead (II) ion with the arsenazo III reagent ℓ =1,0 sm, C_{Pb2+}= 60 μg).

Complex color, phase	pH	λ, HR nm	λ, MeR nm	Δλ	C_{Pb}^{2+} mkg/ml
Purple, solution	5–6	540	610	70	60
Purple, solid	5–6	540	610	70	60

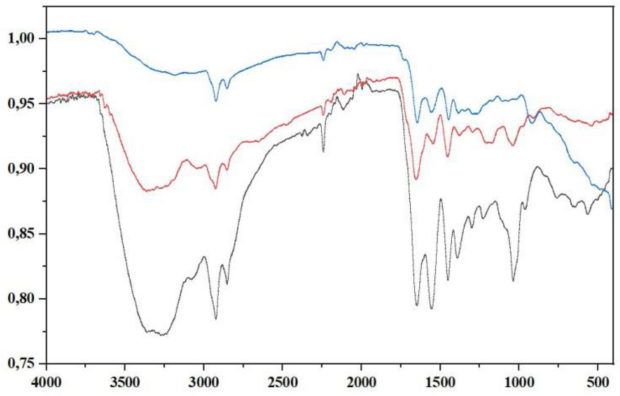


Fig. 11. IR spectrum of the complex of immobilized arsenazo III with Pb(II) ion.

bilized reagent were measured and are presented in Fig. 11.[22,23]

In the IR spectrum of Arsenazo III, an absorption band corresponding to the valence vibrations of the -OH bond is observed at around 3369 cm^{-1}. Absorption bands due to the valence vibrations of the -C-H (sp^3) bond appear at 2916 cm^{-1}, and those of the =C-H (sp^2) bond appear at 3081 cm^{-1}. An absorption peak of the -C-N=N bond is observed at 1620 cm^{-1}, while bands belonging to the -SO_2 group appear in the 1413–1478 cm^{-1} region. Absorption bands corresponding to the C-O-H bond are observed in the 1169–1297 cm^{-1} region, those of the -C-S- bond at 785 cm^{-1}, and bands formed by the vibrations of the As-O bond in the 423–626 cm^{-1} region.[24] The broadening of absorption peaks in the IR spectrum of the PPD fiber immobilized with the Arsenazo III reagent, observed at 3342 cm^{-1} (due to -O-H vibrations) and 3273 cm^{-1} (due to hydrogen bond and donor-acceptor bond formation), indicates that the reagent is attached to the fiber. These peaks are absent in the IR spectrum of the starting materials. Furthermore, the peaks in the 478–909 cm^{-1} region are significantly broadened compared to those in the spectra of the initial products, indicating an increase in intermolecular and intramolecular hydrogen bonding within the product structure. The appearance of absorption bands in the 478–499 cm^{-1} region, corresponding to As-O vibrations that are not present in the IR spectrum of the fiber, also confirms the immobilization of Arsenazo III on the fiber.[25]

In the IR spectrum of the immobilized Arsenazo III complex with Pb(II) ions, the most significant changes are observed in the 502 cm^{-1} and 554 cm^{-1} regions, while absorption bands corresponding to the main functional groups are preserved. In particular, we can observe that absorption lines belonging to the -O-Pb- and -N-Pb- bonds are formed in these regions, Table 3.

X-ray fluorescence analysis of the complex

The X-ray fluorescence spectrum of immobilized arsenazo III on the PPD sorbent shows the appearance of a spectrum characteristic of the arsenic element, which is absent in the spectrum of the sorbent Fig. 12. The X-ray fluorescence spectrum of the complex shows the highest characteristic of iron ions, which indicates the interaction of the immobilized reagent with lead Fig. 13.

SEM analysis

SEM analysis of immobilized arsenazo III and its complex with Pb (II) ion. Morphological studies and elemental composition of fibers were carried out using a scanning electron microscope “VO-MA 15” (Zeiss, Germany). To obtain information about the composition of elements, electron photographs with selected locations, composition tables, and graphic spectra of fiber samples, immobilized arsenazo III, and its complex with lead ions were presented. The reagent immobilized on the fiber in an appropriate medium was immersed in the studied lead solution, prepared for analysis, and after 15 minutes, it was removed and examined under a scanning electron microscope.[26,27] The results are presented in Fig. 14a to Fig. 15c.

When the PPD fiber matrix with the arsenazo III reagent immobilized was analyzed using a scanning electron microscope, it can be seen from the spectrum and table Figs. 15a to 15c that the chlorine in the carrier was replaced by arsenazo III, since the spectra corresponding to arsenic atoms were clearly visible.

Table 3. IR spectrum analysis of PPD fiber, immobilized arsenazo III, and the complex of immobilized arsenazo III with Pb(II) ion.

Compound	ν = SO_2OR	ν = N=N and (C-N)	ν = O-As	ν = OH	ν = C-H (Ar)	ν = O-Pb	ν = C-H (sp^3)	ν = CN	ν = N-H
PPD	-	1646	-	-	-	-	2852–2923	2242	3185
Immobilized (Arsenazo III)	1453	1654	478	3342	3042	-	2926–2853	2192	3273
Immobilized arsenazo III Pb(II) complex	1455	1647	-	3373	2923	502	2852	2242	

Analyzed result

Sample Information

Sample name	M. PPD tola Arsen. (III)
File name	M. PPD tola Arsen. (III)
Application	Umumiy.
Date	2024/ 2/23 10:03
Analyzed by	
Counts	1
Comment	

Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	14.6	mass%	0.0093	0.0054	0.0161
2	Br	0.0005	mass%	<0.0001	0.0001	0.0004
3	P	4.26	mass%	0.0090	0.0038	0.0115
4	S	0.375	mass%	0.0036	0.0089	0.0266
5	K	0.0296	mass%	0.0017	0.0027	0.0080
6	Ca	0.164	mass%	0.0027	0.0011	0.0033
7	Ti	0.0044	mass%	0.0003	0.0006	0.0019
8	Cr	0.0010	mass%	0.0001	0.0003	0.0008
9	Fe	0.0248	mass%	0.0006	0.0006	0.0019
10	Cu	0.0031	mass%	0.0001	0.0001	0.0004
11	Zn	0.0028	mass%	<0.0001	0.0001	0.0003
12	As	0.0879	mass%	0.0003	0.0002	0.0006
13	Sr	0.0012	mass%	<0.0001	<0.0001	0.0001
14	Zr	0.0884	mass%	0.0009	0.0002	0.0006
15	Ag	0.0006	mass%	<0.0001	<0.0001	0.0003
16	Tb	0.0071	mass%	0.0008	0.0018	0.0054
17	Dy	0.0044	mass%	0.0005	0.0011	0.0033

Spectrum

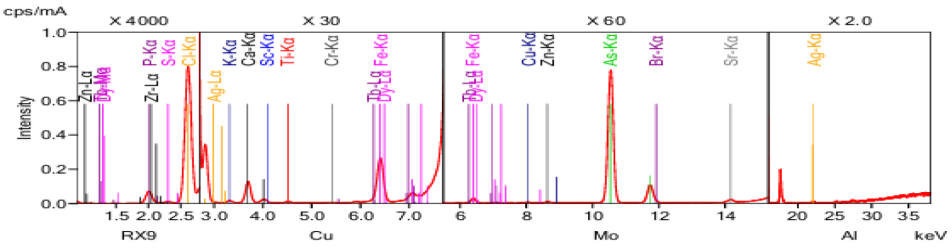


Fig. 12. X-ray fluorescence spectrum of immobilized ADND-3.

Analyzed result

Sample Information

Sample name	M. PPD tola Arsen. (III) Pb
File name	M. PPD tola Arsen. (III) Pb
Application	Umumiy.
Date	2024/ 2/23 10:14
Analyzed by	
Counts	1
Comment	

Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	1.72	mass%	0.0019	0.0012	0.0037
2	Mg	0.780	mass%	0.0207	0.0445	0.133
3	Si	0.329	mass%	0.0034	0.0064	0.0191
4	P	1.83	mass%	0.0040	0.0045	0.0135
5	S	3.23	mass%	0.0053	0.0119	0.0358
6	Ca	0.0442	mass%	0.0013	0.0014	0.0041
7	Ti	0.0024	mass%	0.0003	0.0005	0.0016
8	Cr	0.0007	mass%	<0.0001	0.0002	0.0006
9	Fe	0.0138	mass%	0.0004	0.0008	0.0025
10	Ga	0.0226	mass%	0.0006	0.0015	0.0046
11	Ge	0.0025	mass%	0.0001	0.0003	0.0010
12	As	0.216	mass%	0.0011	0.0040	0.0121
13	Se	0.0026	mass%	0.0002	0.0005	0.0014
14	Zr	0.103	mass%	0.0011	0.0004	0.0011
15	Re	0.0113	mass%	0.0004	0.0007	0.0021
16	Ir	0.0862	mass%	0.0013	0.0035	0.0104
17	Pb	2.59	mass%	0.0032	0.0045	0.0135
18	At	0.0035	mass%	0.0003	0.0007	0.0021
19	Tb	(0.0051)	mass%	0.0009	0.0024	0.0071
20	Dy	(0.0046)	mass%	0.0007	0.0019	0.0058

Spectrum

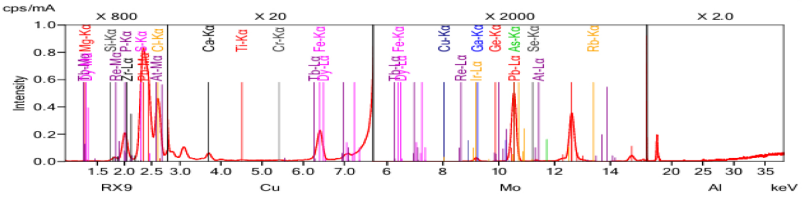


Fig. 13. X-ray fluorescence spectrum of the complex of lead (II) ions with immobilized arsenazo III reagent.

Then, SEM analysis of the complex formed with Pb showed that the percentage concentration of N and O atoms decreased, and a signal corresponding to Pb was formed. As can be seen from the obtained X-ray fluorescence and SEM results, it can be concluded that arsenazo was immobilized on the PPD fiber, and the Pb (II) ion interacted with the immobilized organic reagent. This indicates the correctness of the developed sorption-spectrophotometric method, Scheme 3.

The sensitivity index of the developed method according to Sendel was found to be 0.001 $\mu g/cm^2$, with light absorption as follows:

$$S.b.s. = \frac{60 \cdot 1.0 \cdot 0.001}{0.7 \cdot 25} = 0,00342 \frac{mkg}{sm^2}$$

The results of the analysis were summarized and presented in Table 4 below.

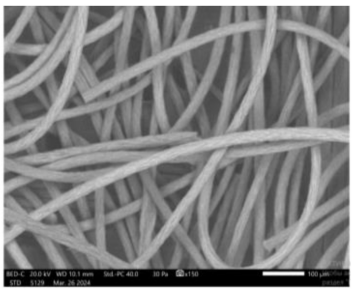


Fig. 14a. SEM image of arsenazo III immobilized on PPD fiber.

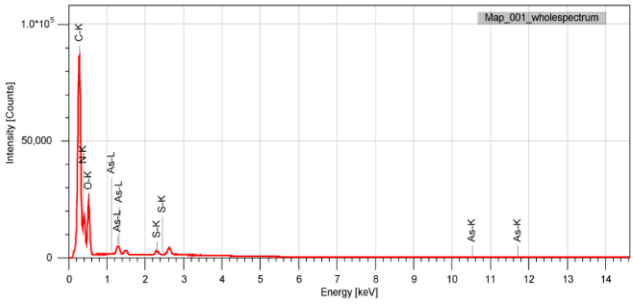
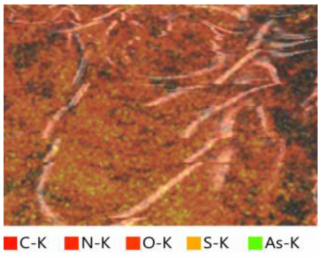


Fig. 14b. SEM spectrum of arsenazo III immobilized on PPD fiber.



Element	Line	Mass%	Atom%
C	K	49.04±0.03	54.73±0.04
N	K	24.91±0.09	23.84±0.08
O	K	25.32±0.07	21.22±0.06
S	K	0.35±0.00	0.15±0.00
As	K	0.38±0.02	0.07±0.00
	Total	100.00	100.00

Fig. 14c. Morphological study of arsenazo III reagent immobilized on PPD.

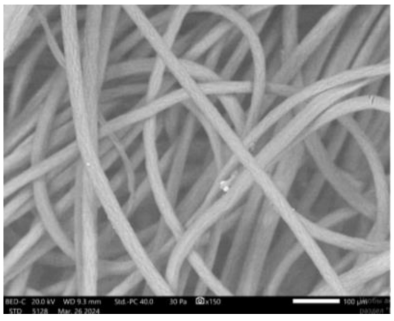


Fig. 15a. SEM image of the complex formed by Pb(II) ion with arsenazo III immobilized on PPD fiber.

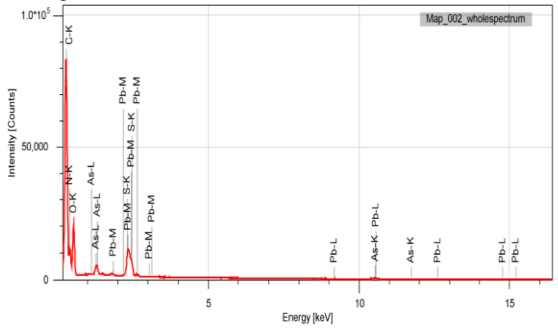
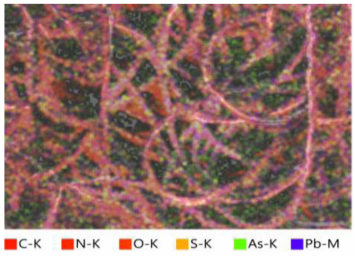


Fig. 15b. SEM spectrum of the complex formed by Pb(II) ion with arsenazo III immobilized on PPD fiber.



Element	Line	Mass%	Atom%
C	K	55.15±0.04	65.81±0.05
N	K	14.62±0.08	14.96±0.08
O	K	20.30±0.06	18.19±0.06
S	K	0.46±0.01	0.21±0.00
As	K	1.47±0.05	0.28±0.01
Pb	M	8.00±0.04	0.55±0.00
Total		100.00	100.00

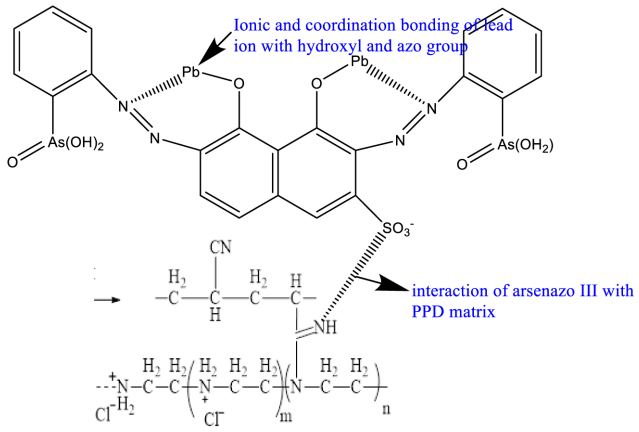
Fig. 15c. Morphological study of the complex of arsenazo III reagent immobilized on PPD with lead (II).

Application of the developed sorption-spectrophotometric method for the determination of Pb (II) to the analysis of complex model mixtures.

To apply the developed method to the analysis of real objects, a complex model mixture close to the composition of the natural object was initially prepared, and the sorption-spectrophotometric determination of Pb (II) ion in this solution was carried out using the same method and conditions as for individual solutions.[28] The analysis results are presented in Table 5.

Competitiveness assessment of the sorption-spectrophotometric method for the determination of Pb (II) ion.

To assess the competitiveness of the proposed sorption-spectrophotometric method for the determi-



Scheme 3. Probable structure of the complex formed by Pb(II) ion with arsenazo III in a solid matrix.

Table 4. Comparison of optimal conditions for complexation of Pb(II) with Arsenazo III in solution and on a PPD polymer carrier.

	Conditions for the formation of the complex	
Parameters	in solution	PPD in a polymer matrix
λ nm	610	610
Optimal pH	5,8	5,8
mkg/ml Lower detecton limit	0,04	0,04
Time to manifestation of complex formation, min	15	7–10
Selectivity	Co(100), Ni(50), Cu(50), Al(100), Zr(10), Mo(10), W(50), Cd(100)	Co(100), Ni(50), Cu(50), Al(100), Zr(10), Mo(10), W(50), Cd(100)

nation of Pb (II) ion, as well as to determine the reliability of the results obtained, some metrological and analytical parameters of other alternative methods were compared. The results are presented in Table 6.

The data in Table 7 demonstrate the accuracy of the developed method, and its comparison with other independent methods shows that the proposed methods are in no way inferior to existing and widely used methods in chemical analysis for some metrological characteristics. Thus, the developed method can be used in the analysis of technical and real objects.

To assess the accuracy of the developed method, we checked it with tabular data using Student's coefficient and Fisher's criteria, and we observed that the obtained analysis results were correct.

Table 5. Results of the determination of Pb (II) ion in artificial mixtures with immobilized arsenazo III (P=0.95 n=5).

	Found Me, mkg (P=0,95; n=5)		
Composition of the mixture being analyzed, μg	x + ΔX	S	S_r
Pb(2,0) + Fe(5,0) + Zn(15)	1,99 ± 0,02	0,017	0,008
Pb(1,0) + Cr(10,0) + Cd(13,0) + Cu(10,0)	0,99 ± 0,03	0,025	0,026
Pb(3,0) + Mn(5,0) + Fe(15,0) + Mn(10,0)	2,98 ± 0,02	0,021	0,007
Pb(5) + Ca(3,0) + Mg(2,0) + Cu(10,0) + Fe(30,0)	4,98 ± 0,02	0,02	0,004

Table 6. Results of competitiveness assessment by comparison with data obtained using other independent methods for the determination of Pb (II) ion in natural waters (n=5; P=95).

	Developed method		spectrophotometry		inversion voltammetry	
№Samples	x ± ΔX	S_r	x ± ΔX	S_r	x ± ΔX	S_r
1	4,99 ± 0,02	0,004	4,97 ± 0,04	0,007	4,97 ± 0,03	0,006
2	9,97 ± 0,05	0,005	10,03 ± 0,18	0,016	9,96 ± 0,06	0,072
3	14,97 ± 0,04	0,002	14,96 ± 0,003	0,003	14,97 ± 0,026	0,002

Table 7. Comparison of the competitiveness of the sorption-spectrophotometric method and the inversion-voltammeterometric method for the determination of Pb (II) ion in technogenic waters (n=11; $f_1 = f_2 = 11$, P=0.99).

	SSF method		IV method	
Object analysis	sample, μg/ml	S_r	sample, μg/ml	S_r
Technogenic water	0,4	0,07	0,4	0,05
t- criteria	$T_{calc.} = 1{,}65$; $t_{table}=3{,}17$		$t_{calc} < t_{table}$	
F- criteria	$F_{calc} = 1{,}5$; $F_{table} = 4.46$		$F_{calc} < F_{table}$	

Conclusion

As a result of this study, the analytical and metrological parameters of the determination of lead ions using Arsenazo III immobilized on a PPD fiber carrier were studied by a novel sorption–spectrophotometric method for the determination of Pb(II) ions, which was successfully developed using Arsenazo III immobilized on a PPD polymer fiber. The immobilization process, confirmed by SEM, XRF, and IR analyses, demonstrated that the sulfo groups of Arsenazo III effectively interact with the amino groups of the polymer, while Pb(II) ions form stable complexes via ionic and coordination bonds. Optimal conditions for complex formation were established at pH 5.8 in acetate buffer, with a maximum analytical signal observed at 610 nm. The method exhibited high sensitivity, selectivity, and rapid complexation, with complete immobilization achieved within 15 minutes and a linear response over the Pb(II) concentration range of 0.04–60 μg/mL, following the Beer–Lambert–Bouguer law. Validation with artificial mixtures and comparison with established spectrophotometric and voltammetric methods confirmed the accuracy, reproducibility, and competitiveness of the proposed approach. Overall, the developed method provides a simple, reliable, and efficient tool for determining trace Pb(II) in environmental and technogenic water samples.

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Authors' declaration

- Conflicts of Interest: None.
- I/We hereby confirm that all the Figures and Tables in the manuscript are mine/ours. Furthermore, any Figures and images that are not mine/ours have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript
- Ethical Clearance: The project was approved by the local ethical committee at Tashkent Institute of Chemical Technology and Termez State University of Engineering and Agrotechnologies, Uzbekistan.

Authors' contributions statement

All authors conceived of the presented idea. K.M. and S.Z. collected the necessary materials (chemical substances, tools etc.) and methods for conducting this research. M.O. and Y.B. studied the reaction, optimal conditions for Determination of Pb(II) ion with a novel, highly efficient immobilized arsenazo III in a polymer matrix and other parameters. N.A.K and S.F studied the mathematical calculations for the determination of copper(II) ions in this arsenazo III in a polymer matrix reagent. J.F. discussed all the results obtained with the authors and they contributed greatly to the preparation of the research work. D.O. helped review and correct grammatical errors and minor issues in the paper. All authors read the manuscript carefully and approved the final version of this article.

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تعيين أيون الرصاص الثنائي Pb(II) باستخدام أرسينازو III مثبَّت عالي الكفاءة داخل مصفوفة بوليمرية

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الخلاصة

في هذه الدراسة، جرى تطوير كاشف تحليلي جديد قائم على أرسينازو III المثبَّت على مصفوفة بوليمرية مكوَّنة من بولي أكريلونتريل، وبولي إيثيلين بولي أمين، وثنائي كلورو الإيثان (PPD)، وتطبيقه في التقدير الطيفي لأيونات الرصاص الثنائي Pb(II). وقد أظهرت الطريقة المقترحة خصائص تحليلية وقياسية متميزة للتعيين الدقيق لأيونات Pb(II)، كما يتضح من قيمة الانحراف المعياري النسبي (Sr) البالغة 0.06، وأدنى تركيز قابل للكشف (Cmin) البالغ 0.24 ميكروغرام/لتر. تم تقييم دقة الطريقة وقابليتها للتكرار باستخدام اختبار t لستودنت واختبار F لفشر. وأكدت مقارنة القيم التجريبية بالقيم الجدولية (t التجريبي = 1.09؛ t الجدولي = 2.83، وF التجريبي = 2.52؛ F الجدولي = 4.47) موثوقية الإجراء التحليلي المقترح. وعند قيمة pH مقدارها 5.8 في وسط منظم الأسيتات، أظهر معقد Pb(II)-أرسينازو III المثبَّت على مصفوفة PPD أعلى إشارة تحليلية عند طول موجي بلغ 640 نانومترًا. وقد اتبعت نتائج القياس قانون بير–لامبرت–بوغير، حيث أظهرت علاقة خطية ضمن مدى تراكيز Pb(II) الممتد من 0.04 إلى 2.4 ميكروغرام/مل. وتمتاز الطريقة المقترحة بالبساطة والسرعة وانخفاض التكلفة، فضلًا عن قدرتها على التعيين المباشر لأيونات Pb(II) في عينات المياه بحساسية وانتقائية عاليتين.

الكلمات المفتاحية: قانون بير–لامبرت–بوغير، المطيافية الامتصاصية بالامتزاز عالية الانتقائية، الكشف عن الرصاص، المازّ PPD، عينات المياه.