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Structural and Sorption Characteristics of Nano-Structured Polysaccharide Biopolymers Derived from Plants of the Asteraceae Family

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ABSTRACT

This study investigates the structural and sorption characteristics of nanostructured polysaccharide biopolymers isolated from plants of the Asteraceae family (Dahlia tubers and Helianthus tuberosus) toward Pb^{2+} ions in model aqueous solutions. The biopolymers were obtained by acid extraction at pH 1.5-2.5 and 80-90 °C, followed by ethanol precipitation, with a yield of 10-14%.

Surface morphology examined by scanning electron microscopy revealed a hierarchically organized nano- and micro structured matrix with particle agglomeration and developed interaggregate porosity. The presence of lamellar structures and a porous network promotes diffusion of metal ions to active functional centers.

The interaction mechanism between the biopolymer matrix and Pb^{2+} was studied by FTIR spectroscopy. Shifts and intensity changes of absorption bands corresponding to carboxyl and hydroxyl groups confirmed a coordination-chelate complexation mechanism without destruction of the polysaccharide backbone.

Sorption experiments were performed using model Pb^{2+} solutions prepared from analytical-grade lead (II) acetate. The residual Pb^{2+} concentration after contact with the biopolymer was determined by atomic absorption spectrometry at 283.3 nm using calibration. Additional control measurements were conducted by complexometric titration in acetate buffer medium (pH 5.5) with 0.01 M Trilon B.

Up to 76-77% of Pb^{2+} was removed within the first 10 min, and sorption equilibrium was reached after 60 min. The maximum removal efficiency reached 87%. The Freundlich model best described the process, indicating heterogeneous surface structure and multilayer adsorption. These results demonstrate the potential of Asteraceae-derived polysaccharide biopolymers as environmentally safe sorbents for heavy metal removal from aqueous media.

1. Introduction

The contamination of aquatic ecosystems with heavy metal ions remains one of the most serious environmental problems of our time. Lead ions (Pb^{2+}) are highly toxic, bioaccumulative, and persistent in the environment, which necessitates the development of effective methods for their removal from aqueous solutions [1, 2]. In this regard, water purification technologies based on the use of environmentally friendly and renewable materials have been actively developed in recent years [3].

The ref. [4] provides a comprehensive description of methods for producing highly efficient porous carbon sorbents based on lignocellulosic waste of agricultural origin. Various biomass materials, such as rice husks, nut shells, straw, and wood shavings, are used as feedstock; these are subjected to carbonization followed by chemical activation, most commonly using potassium hydroxide (KOH). It has been shown that KOH activation promotes the development of a hierarchical porous structure with the formation of micro-, meso-, and macro-pores, as well as an increase in the number of oxygen-containing functional groups on the surface of the materials. This, in turn, leads to a significant increase in the sorption capacity of carbon materials toward various pollutants. The resulting sorbents are considered effective, economically accessible, and environmentally safe materials for water and air purification.

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The review examines the composition and characteristics of plant biomass and its thermal conversion methods into biochar, including pyrolysis, torrefaction, and hydrothermal carbonization. Special emphasis is placed on hydrothermal carbonization, covering the reaction mechanism, transformation of individual biomass components and the whole material, properties of the resulting biochar, and current applications, such as fuel production and the manufacture of adsorbents, catalysts, and other materials [5]. Particular attention is paid to natural polysaccharides, such as pectins, alginates, cellulose, and their derivatives, which are biodegradable, readily available, and contain functional groups (–COO^-, –OH) capable of binding metal cations [6, 7]. Pectin materials demonstrate high affinity for Pb^{2+} ions due to the implementation of ion-exchange and coordination-chelate interaction mechanisms [8].

Modern research shows that the sorption capacity of polysaccharide biopolymers is determined not only by their chemical composition, but also by their structural organization. The degree of methoxylation, distribution of functional groups, molecular weight, and surface morphology have a significant effect on the kinetics and capacity of sorption [9, 10]. Hierarchically organized nano- and microstructured biopolymer matrices are characterized by a developed porous system and increased accessibility of active centers, which contributes to the acceleration of mass transfer and increased efficiency of metal binding [11].

Plants of the Asteraceae family have attracted considerable attention as abundant and renewable sources of polysaccharide biopolymers with diverse biological and functional properties. Polysaccharides isolated from Arctium lappa L. roots using ultrasound-assisted extraction optimized by response surface methodology have been shown to contain monosaccharides such as mannose, glucose, fructose and galactose and to exhibit significant antioxidant activity through efficient scavenging of reactive oxygen species, including DPPH, hydroxyl and superoxide radicals. These findings highlight the potential of Asteraceae-derived polysaccharides as promising natural antioxidant compounds. Similarly, polysaccharides obtained from Helianthus tuberosus have demonstrated notable structural and biological properties, including pronounced anti-inflammatory and bioactive effects associated with their molecular structure and interactions with biological targets. Such characteristics confirm the potential of polysaccharides from Asteraceae plants as promising candidates for the development of functional and biomedical materials [12, 13]. However, comprehensive studies combining morphological analysis, spectroscopic identification of functional groups, and modeling of sorption isotherms remain limited.

The current state of research in this area necessitates a systematic study of the structural and sorption characteristics of polysaccharide biopolymers from plants of the Asteraceae family using modern methods of physicochemical analysis.

The aim of this work is to study the structural and sorption characteristics of nanostructured polysaccharide biopolymers isolated from plants of the Asteraceae family (Dahlia tubers and Helianthus tuberosus) in relation to Pb^{2+} ions in model aqueous solutions.

Despite the growing interest in polysaccharides derived from Asteraceae plants, previous studies have primarily focused on their extraction methods, chemical composition, antioxidant activity, and biological properties. Information on the relationship between the hierarchical structure of these biopolymers and their sorption behavior toward heavy metal ions remains limited. In particular, comparative studies of polysaccharide biopolymers isolated from Dahlia tubers and Helianthus tuberosus using a comprehensive approach that combines morphological characterization, FTIR analysis of metal-binding mechanisms, and sorption isotherm modeling are scarce.

The novelty of the present work lies in the integrated investigation of nanostructured polysaccharide biopolymers obtained from two Asteraceae plant sources as environmentally friendly sorbents for Pb^{2+} removal. For the first time, the structural organization of the obtained biopolymers was correlated with their sorption performance toward lead ions through a combination of scanning electron microscopy, FTIR spectroscopy, and adsorption modeling. The study provides new insights into the role of hydroxyl and carboxyl functional groups in Pb^{2+} binding and demonstrates the applicability of these renewable biopolymers as effective materials for aqueous heavy-metal remediation.

The research objectives include:

– obtaining polysaccharide biopolymers by acid extraction followed by precipitation;

– studying the morphology and porosity of the biopolymer matrix by SEM;

– identifying the functional groups involved in the interaction with Pb^{2+} using IR spectroscopy;

– evaluating the kinetics and efficiency of Pb^{2+}

extraction with analytical control of the residual concentration and interpretation of sorption isotherms.

Unlike most previously published studies on pectin and other polysaccharide sorbents, this study has produced materials derived from plant raw materials of the Asteraceae family that possess a well-developed functional and porous structure, which ensures their high sorption activity toward Pb^{2+} ions. The determined values of maximum sorption capacity and kinetic parameters are at the level of or exceed the corresponding values described for similar biopolymer sorbents in the literature, which indicates the competitiveness of the developed materials. Additionally, it should be noted that the combination of natural raw materials, ease of production, and high sorption efficiency makes the proposed biopolymers promising for practical application in water purification systems for the removal of heavy metal ions.

2. Experimental part

Dahlia tubers and Helianthus tuberosus, belonging to the Asteraceae family, were used as plant raw materials.

The plant raw materials were dried to an air-dry state and ground to a particle size of 1-2 mm. In order to remove lipophilic and low-molecular-weight accompanying compounds (chlorophylls, waxes, phenolic substances), the raw materials were pretreated with 83-85% ethanol while heating. After extraction, the alcohol solution was separated, and the plant residue was dried to a constant weight.

The pre-purified raw material was subjected to acid extraction with an aqueous solution at pH 1.5-2.5 and a temperature of 80-90 °C with constant stirring for 1.0-2.0 hours. The resulting aqueous extract was separated from the insoluble residue by filtration.

The extract was concentrated under reduced pressure, after which the pectin substances were precipitated by adding ethanol to a volume fraction of at least 96%. (1:3) The resulting gelatinous pectin precipitate was separated by centrifugation, sequentially washed with ethanol to remove residual impurities, and dried to a constant weight [14].

The yield of pectin substances was calculated as a percentage of the mass of the initial air-dry plant raw material. The yield of pectins was 10-14%.

SEM. During the study, an Axia Chemi SEM scanning electron microscope (Thermo Fisher Scientific) (USA) with an energy dispersive analysis system (SEM-EDS) was used to obtain micrographs.

IR spectroscopy. IR spectra were recorded using a Bruker ALPHA instrument (Germany) at room temperature with a wavenumber resolution of 1 cm^{-1} in the frequency range 4000-400 cm^{-1}.

Atomic absorption spectrometer. Sorption studies were conducted in model aqueous solutions of Pb^{2+} prepared from analytical-grade lead (II) acetate. The use of a model system is due to the need to create controlled conditions with a given initial concentration of metal ions and to exclude the influence of accompanying ions and organic impurities characteristic of natural and waste waters. This approach allows for an objective assessment of the sorption capacity of the biopolymer matrix.

The residual concentration of Pb^{2+} ions after sorption was determined by atomic absorption spectrometry using a PinAAcle 900T device (PerkinElmer Inc., USA). Measurements were performed at an analytical wavelength of 283.3 nm using a calibration curve constructed from standard lead solutions.

The sorption capacity of the polysaccharide biopolymer matrix was determined by the amount of Pb^{2+} ions bound to 0.15 g of sorbent. The amount of adsorbed metal was calculated from the difference between the initial and residual concentrations of lead ions in the model solution. After contact of the biopolymer with a standard solution of lead (II) acetate, the resulting lead pectinate precipitate was separated by filtration, and the concentration of Pb^{2+} in the filtrate was determined by titrimetric method.

The residual Pb^{2+} ion content was determined by complexometric titration with a 0.01 M solution of Trilon B (Na_2EDTA) in an acetate buffer medium at pH 5.5 in the presence of xylenol orange indicator until the color changed from red to lemon yellow.

Table 1. Organoleptic characteristics of pectins obtained from Dahlia tubers and Helianthus tuberosus [15]

Parameter	Dahlia tubers	Helianthus tuberosus	Standard (GOST 29186-91)
Appearance	Amorphous powder	Amorphous powder	Amorphous powder
Odor	Odorless	Odorless	Odorless
Color	Light beige	Light beige	Light beige

Before titration, the solution was brought to a volume of 100 ml with purified water; for analysis, a 10 ml aliquot was taken, 10 ml of acetate buffer was added, and then titrated with constant stirring. The concentration of Pb^{2+} cations was determined both in the initial solution and after sorption, and the sorption capacity was calculated using the generally accepted method [16].

The mass of lead in the solution under investigation was calculated using the following formula:

$$m(Pb^{2+}) = \frac{N \cdot V \cdot Э_{(Pb^{2+})}}{1000}$$

where: N – the normal concentration of the Trilon B solution; V – the volume of the Trilon B solution used for titration, ml; EPb^{2+} – the molar equivalent mass of lead ions, mol/l.

The experimental adsorption value was calculated using the following expression:

$$A_{exp} = \frac{(C_0 - C_e) \cdot V}{m}$$

where: C_0 and C_e – the initial and equilibrium concentrations of Pb^{2+} ions in the solution, mmol/L; V – the volume of the solution, L; m – the mass of the pectin sorbent, g.

The experimental adsorption values obtained were used to construct sorption isotherms (Fig. 4). To analyze the nature of the sorption process, the experimental data were compared with the Langmuir and Freundlich models. The parameters of the corresponding equations were determined graphically, and the calculated values are given in Tab. 3.

The analysis of adsorption isotherms and the calculation of the ratios between experimental and theoretical adsorption values (A_{exp}/A_{theory} and A_{exp}/A_{theory}) were performed similarly to the method previously used for pectin sorbents [17].

Analysis of the isotherm shapes and the relationships between experimental and theoretical adsorption values showed that Freundlich's model most accurately describes the process of Pb^{2+} ion sorption by Jerusalem artichoke and dahlia pectins. This indicates the heterogeneity of the active centers on the surface of the sorbents and the possibility of multilayer adsorption. The Langmuir model was used for a comparative assessment of the maximum sorption capacity of pectins, but the experimental data are less consistent with the assumption of monomolecular adsorption.

Thus, the pectins studied exhibit high sorption capacity for lead ions and can be considered as promising natural sorbents for the purification of aqueous solutions from heavy metals.

To provide a more comprehensive analysis of the sorption behavior of the materials under study, the parameters of the Langmuir and Freundlich isotherms were additionally examined. The values of Kf and n for the Freundlich model indicate a high sorption capacity and the heterogeneous nature of the surface of pectin biopolymers; where n>1 indicates favorable conditions for Pb^{2+} sorption. For the Langmuir model, the qmax parameter reflects the maximum sorption capacity of the sorbent, whereas KL characterizes the affinity of the active sites for Pb^{2+} ions.

Table 2. Change in Pb^{2+} ion concentration during sorption process

Sorbent	Time (min)	Pb^{2+} concentration (mg/L)	Adsorption capacity (mg/g)	Pb^{2+} concentration (mmol/L)	Removal efficiency (%)
DT	0	12.55	0.00	0.0606	0.00
DT	5	4.75	5.20	0.0229	62.15
DT	10	2.92	6.42	0.0141	76.73
DT	20	2.80	6.50	0.0135	77.70
DT	30	2.17	6.92	0.0105	82.70
DT	60	1.58	7.32	0.0076	87.41
HT	0	10.22	0.00	0.0493	0.00
HT	5	3.86	4.24	0.0186	62.23
HT	10	2.38	5.23	0.0115	76.71
HT	20	2.28	5.29	0.0110	77.80
HT	30	1.76	5.64	0.0085	82.80
HT	60	1.60	5.75	0.0077	84.34

Table 3. Comparison of experimental and calculated adsorption values for Pb^{2+} ions

Sorbent	ΔC (mmol/L)	A_{exp} (mmol/g)	$A_{Langmuir}$ (mmol/g)	$A_{Freundlich}$ (mmol/g)	$A_{exp}/A_{Langmuir}$	$A_{exp}/A_{Freundlich}$
DT	0.0377	0.0251	0.0364	0.0260	0.69	0.97
DT	0.0465	0.0310	0.0360	0.0308	0.86	1.01
DT	0.0471	0.0314	0.0403	0.0312	0.78	1.01
DT	0.0501	0.0334	0.0407	0.0330	0.82	1.01
DT	0.0530	0.0353	0.0430	0.0347	0.82	1.02
Average (DT)	—	—	—	—	*0.79*	*1.00*
HT	0.0307	0.0205	0.0302	0.0210	0.68	0.98
HT	0.0378	0.0252	0.0331	0.0250	0.76	1.01
HT	0.0383	0.0255	0.0340	0.0251	0.75	1.02
HT	0.0408	0.0272	0.0358	0.0270	0.76	1.01
HT	0.0416	0.0277	0.0370	0.0280	0.75	0.99
Average (HT)	—	—	—	—	*0.74*	*1.00*

A comparison of the obtained parameters with literature data for pectin and other plant-based sorbents shows that the qmax values are at the level of or exceed the values reported for similar biopolymer materials, which confirms the competitiveness of the developed sorbents. The data in Table 3 further confirm that the Freundlich model provides a more accurate description of the experimental results compared to the Langmuir model, which is consistent with the heterogeneous nature of the surfaces of the materials under study [18].

3. Results and Discussion

The sorption behavior of pectin biopolymers is largely determined by the pH of the medium, since the degree of protonation of the carboxyl groups (–COOH/–COO^-) affects the accessibility of active sites and the nature of the interaction with Pb^{2+} ions. In the low pH range, a decrease in sorption capacity is observed, which is due to competition between protons (H^+) and metal cations for binding to the sorbent's functional groups. As the pH increases to the weakly acidic range (pH 3.0-6.0), carboxyl groups dissociate to form –COO^-, which leads to an increase in the surface negative charge and a strengthening of the electrostatic attraction to Pb^{2+} ions, as well as promoting complex formation.

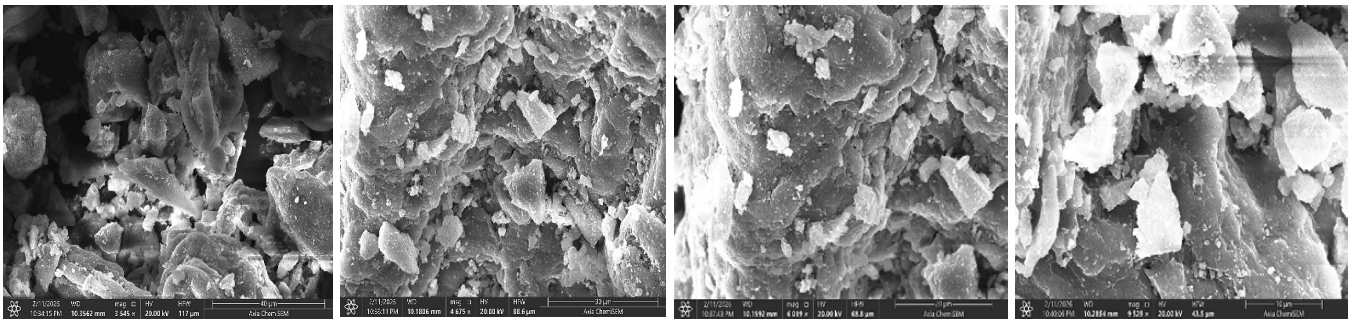
It should be noted that in real aqueous systems, the sorption process is significantly influenced by competing cations, primarily Ca^{2+} and Mg^{2+}, present in natural and wastewater. These ions are capable of interacting with the carboxyl groups of pectin and occupying some of the active sites, which can lead to a decrease in sorption capacity with respect to Pb^{2+}. However, the higher affinity of pectin functional groups for heavy metal ions, due to their ability to form stronger coordination bonds, ensures that significant sorption efficiency is maintained even in the presence of background electrolytes. This indicates the potential for applying the studied materials under conditions approximating real aqueous environments.

As a result of acid extraction of polysaccharide biopolymer fractions from Dahlia tubers and Helianthus tuberosus tubers, differences in the yield of the target product were established. From 10 g of air-dried Dahlia tubers, 1.4 g of biopolymer (14%) was obtained, while from the same amount of Helianthus tuberosus raw material, 1.0 g (10%) was obtained. The data obtained indicate a higher concentration of polysaccharide components in dahlia tubers.

In terms of organoleptic characteristics, the isolated biopolymer fractions of both species were an amorphous light beige powder with no pronounced odor, indicating a sufficient degree of purification and the absence of significant impurities of accompanying compounds.

Thus, both types of plant raw materials studied can be considered as sources of polysaccharide biopolymers, but Dahlia tubers are a more promising object in terms of yield and technological efficiency of obtaining a functional biopolymer matrix.

Degree of agglomeration. All magnifications show pronounced particle agglomeration. At



(a) 3545× (b) 4675× (c) 6019× (d) 9529×

Fig. 1. SEM micrographs of the surface morphology of the pectin sorbent at different magnifications ((a) 3545×, (b) 4675×, (c) 6019× and (d) 9529×).

Table 4. Morphological characteristics of the pectin sorbent surface at different SEM magnifications

Parameter	9529×	6019×	4675×	3545×
Degree of agglomeration	High	High	Moderate	Moderate
Structure density	High	High	Moderate	Moderate
Porosity	Reduced	Reduced	Moderate	Increased
Fine inclusions	Pronounced	Pronounced	Pronounced	Moderately pronounced

magnifications of 9529× and 6019×, the particles are tightly bound together, forming compact aggregates. At lower magnifications (4675× and 3454×), the structure appears somewhat more sparse.

Structure density. At high magnifications, the structure is characterized by high density and compactness. The particles have a lamellar and layered morphology. At lower magnifications, interparticle gaps are visible, indicating moderate aggregate density.

Porosity. At 9529× and 6019×, porosity is reduced due to the close fit of the particles. In the images at 4675× and 3454×, inter-aggregate voids are clearly visible, indicating more pronounced macroporosity.

Small inclusions. Fine inclusions and fragments are present on the surface of large lamellar particles. At high magnifications, they are more clearly visible, which may indicate a secondary phase or the result of mechanical grinding.

The sample under study is characterized by a lamellar layered morphology with a high degree of particle agglomeration. The structure is predominantly dense, but at lower magnifications, interaggregate pores are revealed. The presence of small inclusions on the surface of the particles indicates a heterogeneous structure and the possible presence of secondary phases.

The polysaccharide biopolymer matrix is characterized by a developed porous structure with a predominance of macropores, which is typical for natural carbohydrate polymers. This morphological organization promotes the effective diffusion of metal ions to active functional centers and determines high sorption activity, as well as the ability to form gel-like structures.

From a practical standpoint, an important aspect of the application of pectin biopolymers is their ability to be regenerated and reused. Desorption of Pb^{2+} ions from the sorbent surface can be effectively achieved using dilute acid solutions (e.g., HCl), which protonate the carboxyl groups ($–COO^- \rightarrow –COOH$) and the breaking of metal–ligand coordination bonds, leading to the release of bound metal ions and the regeneration of the sorbent's active sites.

According to the literature, polysaccharide sorbents, including pectin-based materials, are capable of maintaining high sorption activity over several sorption–desorption cycles. Typically, after 3-5 cycles, a moderate decrease in sorption capacity (by 10-20%) is observed, which may be associated with partial blocking of active sites, incomplete desorption of metal ions, or minor structural changes in the sorbent.

Thus, the pectin biopolymers under study have the potential for repeated use, which confirms their technological effectiveness and promising application in water purification processes. At the same time, the experimental assessment of the sorbent's stability during repeated cycles requires further investigation and will be addressed in future studies.

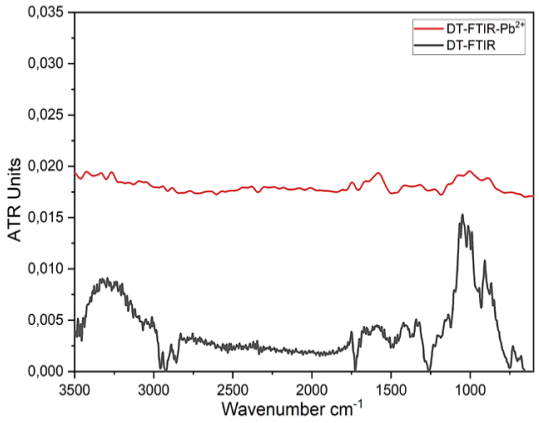


Fig. 2. FTIR spectra of the polysaccharide biopolymer isolated from Dahlia tubers before and after Pb^{2+} sorption.

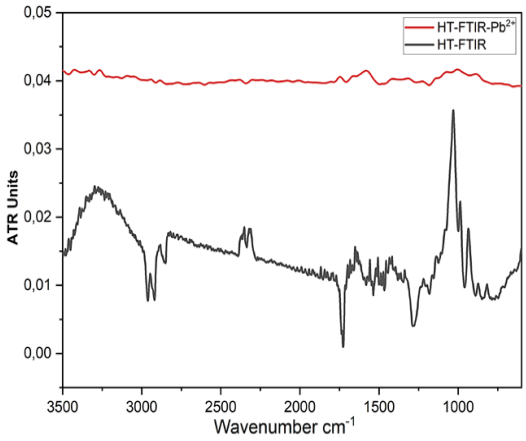


Fig. 3. FTIR spectra of the polysaccharide biopolymer isolated from Helianthus tuberosus tubers before and after Pb^{2+} sorption.

Table 5. FTIR spectral characteristics of polysaccharide biopolymers and changes after Pb^{2+} sorption

Sample	Frequency range (cm^{-1})	Assignment of absorption bands	Changes after Pb^{2+} binding
DT	1750-1735	C=O stretching vibrations of esterified carboxyl groups of pectin ($–COOCH_3$)	Decrease in intensity and slight shift indicating participation of ester groups in complex formation
DT	1650-1600	Asymmetric stretching vibrations of ionized carboxylate groups ($–COO^-$)	Shift to lower frequencies and increased intensity confirming coordination of Pb^{2+} with $–COO^-$ groups
DT	1450-1410	Symmetric stretching vibrations of carboxylate groups ($–COO^-$)	Change in band shape and intensity indicating formation of Pb^{2+}–pectin complexes
DT	1330-1250	C–O stretching and O–H deformation vibrations of carboxyl and phenolic groups	Band weakening due to involvement of oxygen-containing groups in Pb^{2+} sorption
DT	1150-1050	C–O–C stretching vibrations of glycosidic bonds in the polysaccharide matrix	Slight decrease in intensity due to structural rearrangement of pectin during complex formation
DT	1050-1000	C–O stretching vibrations of the pyranose ring of galacturonic acid	Minor changes indicating preservation of the carbohydrate backbone
DT	950-850	Vibrations of glycosidic bonds and C–H deformation vibrations	No significant changes observed
DT	750-700	Deformation vibrations of the polysaccharide chain skeleton	Practically unchanged
HT	3600-3200	O–H stretching vibrations of hydroxyl groups and intermolecular hydrogen bonds	Decrease in intensity and band broadening indicating participation of –OH groups in complex formation
HT	3000-2850	C–H stretching vibrations of the carbohydrate skeleton ($–CH_2$, $–CH_3$)	No significant changes; these groups do not participate directly in Pb^{2+} binding
HT	1750-1730	C=O stretching vibrations of esterified carboxyl groups ($–COOCH_3$)	Decrease in intensity and partial shift indicating involvement of carboxyl groups
HT	1630-1600	Asymmetric stretching vibrations of ionized carboxylate groups ($–COO^-$)	Band shift and increased intensity confirming coordination of Pb^{2+} with $–COO^-$ groups
HT	1450-1410	Symmetric stretching vibrations of carboxylate groups ($–COO^-$)	Change in band shape and intensity indicating formation of Pb^{2+}–pectin complexes
HT	1320-1240	C–O stretching and O–H deformation vibrations of carboxyl groups	Weakening of the band due to participation of oxygen-containing groups in sorption
HT	1150-1000	C–O–C and C–O stretching vibrations of the polysaccharide ring	Minor changes indicating possible structural rearrangement
HT	950-850	Vibrations of glycosidic bonds and pyranose ring	No significant changes; carbohydrate skeleton preserved

The main mechanism of Pb^{2+} binding for both pectins is the same – lead ion coordination occurs mainly due to:

1. Carboxyl groups (–COO^-). This is confirmed by:

– shift of asymmetric bands 1650-1600 cm^{-1} (Dahlia tubers) and 1630-1600 cm^{-1} (Helianthus tuberosus);

– change in the shape and intensity of symmetrical bands at 1450-1410 cm^{-1}.

$$Pb^{2+} + 2(C_6H_7O_6^-) \rightarrow Pb(C_6H_7O_6)$$

2. Ester groups (–$COOCH_3$) in both samples participate in the process: a decrease in the intensity of the 1750-1730 cm^{-1} band is observed. However, for Helianthus tuberosus, possible partial de-etherification is additionally noted, which may contribute to an increase in the number of active carboxyl centers. Hydrolysis of methoxyl groups:

$$C_6H_7O_6 – COOCH_3 + H_2O \rightarrow C_6H_7O_6 – COOH + CH_3OH$$

Formation of additional active centers:

$$2(C_6H_7O_6 – COOH) + Pb^{2+} \rightarrow Pb(C_6H_7O_6)^2 + 2H^+$$

3. Hydroxyl groups (–OH):

– Helianthus tuberosus shows a pronounced broadening and weakening of the broad band at 3600-3200 cm^{-1}, indicating the participation of –OH in complex formation;

– In Dahlia tubers, the participation of –OH is weaker and manifests itself mainly in the range of 1330-1250 cm^{-1}.

$$2(C_6H_7O_6 – OH) + Pb^{2+} \rightarrow (C_6H_7O_6 – O)2Pb + 2H^+$$

4. *The polysaccharide (carbohydrate) skeleton is preserved in both cases:*

– the bands at 1150-1000 and 950-850 cm^{-1} undergo only minor changes,

– no significant destruction of glycosidic bonds is observed.

$$(C_6H_8O_6)n + Pb^{2+} \rightarrow [(C_6H_8O_6)n] \cdot Pb^{2+}$$

Both polysaccharide biopolymer matrices effectively bind Pb^{2+} ions mainly due to carboxylate groups (–COO^-), which act as the main coordination centers of sorption.

At the same time, the biopolymer isolated from Helianthus tuberosus tubers demonstrates a more pronounced involvement of hydroxyl groups (–OH) in the interaction with metal ions, while the biopolymer from Dahlia tubers is characterized by a predominant contribution of carboxyl functional groups to the Pb^{2+} binding process.

In general, the sorption mechanism is coordination-ionic in nature and occurs without destroying the carbohydrate skeleton of the polysaccharide matrix, which indicates that its structural integrity is preserved.

The combination of IR spectral data and sorption study results confirms the implementation of a chelation mechanism of complex formation during the binding of Pb^{2+} ions by polysaccharide biopolymers. The highest sorption efficiency was established for the biopolymer matrix obtained from Dahlia tubers, which allows it to be considered a more promising natural sorbent for heavy metals compared to the material from Helianthus tuberosus.

Experimental studies (Tab. 2) showed that polysaccharide biopolymer matrices isolated from dahlia tubers and Jerusalem artichoke tubers (Helianthus tuberosus) have pronounced sorption activity with respect to Pb^{2+} ions in model aqueous solutions.

The sorption efficiency was evaluated based on the decrease in metal ion concentration in the solution after contact with the biopolymer, as well as on the experimentally determined sorption capacity.

The dependence of the change in the concentration of Pb^{2+} ions in solution on the contact time with polysaccharide biopolymer matrices is shown in Fig. 4. It has been established that at the initial stage of the process, there is an intense decrease in lead concentration, which indicates a high sorption rate and the presence of a significant number of available active centers. Subsequently, the extraction rate decreases, and with an increase in contact time, the concentration of Pb^{2+} changes insignificantly, indicating that sorption equilibrium has been reached in the «solution-biopolymer matrix» system.

The graph shows the change in Pb^{2+} concentration over time during sorption on the polysaccharide biopolymer sorbents DT and HT. During the initial contact stage (the first 10-20 min), a rapid decrease in Pb^{2+} ion concentration is observed, indicating high availability of active functional groups and the predominance of a surface sorption mechanism.

In the subsequent stage, the sorption rate gradually decreases, which is associated with the gradual filling of active sites and the transition of the process to a diffusion-limited regime. Subsequently, the system reaches equilibrium approximately 60 min after the start for both sorbents.

Overall, the HT sorbent demonstrates slightly higher efficiency in the initial stage compared to DT,

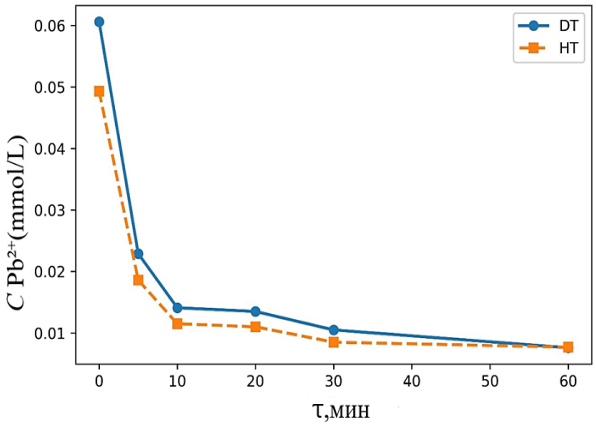


Fig. 4. Change in Pb^{2+} concentration as a function of contact time during sorption on DT and HT polysaccharide biopolymers.

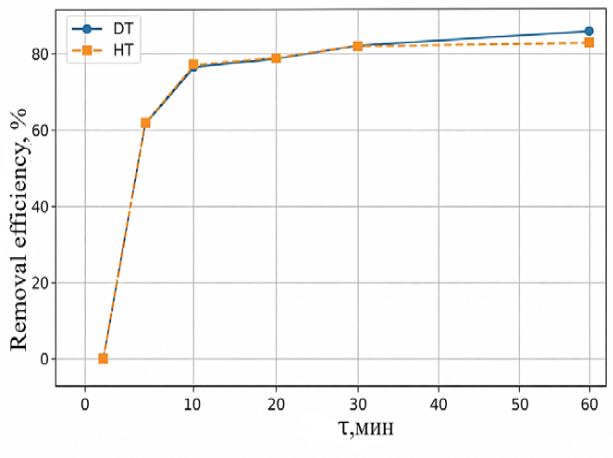


Fig. 5. Degree of Pb^{2+} removal from aqueous solution depending on contact time for DT and HT sorbents.

whereas in the equilibrium region, both materials exhibit similar values of sorption activity. The results confirm the high sorption capacity of the studied polysaccharide biopolymer matrices toward Pb^{2+} ions and their potential for use in water purification processes.

The graph illustrates the change in the degree of Pb^{2+} ion removal (%) from the model solution depending on the contact time with DT and HT polysaccharide biopolymer matrices.

At the initial moment (0 min), the degree of purification for both sorbents is 0%, which corresponds to the absence of contact between the solution and the sorbent. Already in the first 5 min, there is a sharp increase in the degree of purification to ≈ 62%, which indicates a high initial sorption rate. This nature of the process is due to the presence of a large number of free active centers on the surface of pectin.

Within 5-10 min, the degree of purification increases significantly and reaches ≈ 76-77% for both sorbents. Further, in the range of 10-20 min, the increase in the degree of purification slows down, which is associated with the gradual saturation of active centers and a decrease in the concentration gradient.

After 30 min, the degree of purification reaches ≈ 82-83%, after which the process enters a quasi-steady state equilibrium. Between 30 and 60 min, the increase in the degree of purification is insignificant, indicating that the system is approaching sorption equilibrium.

It should be noted that the DT sorbent demonstrates slightly higher purification values in the later stages of the process (up to ≈87% at 60 min) compared to HT (≈84-85%). However, in general, the kinetic curves for DT and HT are similar, indicating a similar nature of the Pb^{2+} ion sorption mechanism.

Thus, we can conclude that:

– the majority of Pb^{2+} ions are removed from the solution within the first 10-20 min;

– further increasing the contact time only leads to a slight increase in the degree of purification;

– DT and HT polysaccharide biopolymer matrices are effective sorption materials for removing Pb^{2+} ions from aqueous solutions.

The graph shows the dependence of sorption capacity A (mmol/g) on the change in Pb^{2+} concentration in solution ΔC (mmol/L) for the DT and HT sorbents. For both materials, a systematic increase in sorption capacity is observed with increasing ΔC, indicating effective binding of Pb^{2+} ions by pectin functional groups.

Sorbent DT exhibits higher sorption capacity values compared to HT across the entire concentration range studied, indicating more pronounced sorption activity.

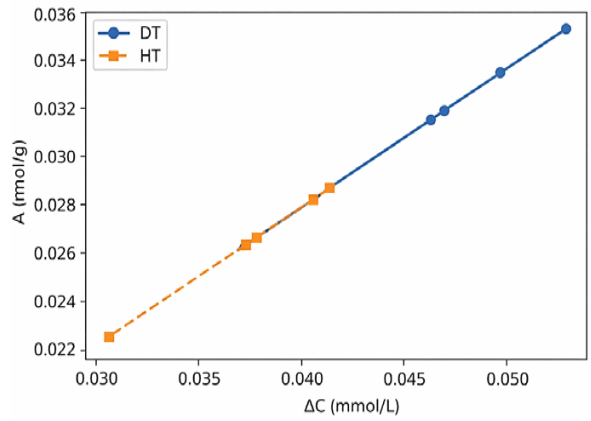


Fig. 6. Dependence of Pb^{2+} adsorption capacity on concentration change (ΔC) for DT and HT sorbents.

The nature of the isotherms indicates a predominantly linear relationship within the studied concentration range, which may suggest the absence of significant surface saturation and high accessibility of active sites.

Minor differences between DT and HT may be attributed to the structural organization of the polymer matrix and the varying accessibility of functional groups involved in complex formation with Pb^{2+}.

Overall, the results confirm an increase in sorption capacity with increasing ΔC and higher efficiency of DT compared to HT.

The graph shows the experimental values of sorption capacity A (mmol/g) as a function of changes in Pb^{2+} concentration (ΔC, mmol/L), as well as the fit of the data using the Langmuir and Freundlich models for the DT and HT sorbents.

For both materials, an increase in sorption capacity is observed with increasing ΔC, indicating effective binding of Pb^{2+} ions by pectin functional groups. At the same time, the DT sorbent is characterized by higher values of A compared to HT across the entire concentration range.

A comparison with theoretical models shows that the Freundlich model better describes the experimental data, indicating the heterogeneous nature of the sorbent surfaces and the non-uniformity of the active sites. At the same time, the Langmuir model exhibits more noticeable deviations, especially at high ΔC values, indicating the limitations of the assumption of a homogeneous surface.

Overall, the results confirm the higher sorption capacity of DT and the heterogeneous nature of the Pb^{2+} sorption process on pectin biopolymers.

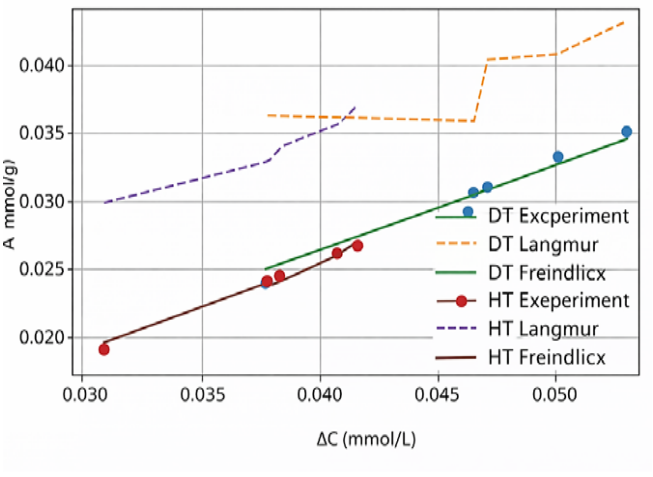


Fig. 7. Sorption isotherms of Pb^{2+} ions on polysaccharide biopolymers: experimental data and fitting by Langmuir and Freundlich models.

The adsorption efficiency of polysaccharide biopolymers is strongly influenced by the pH of the solution. At low pH values, the active functional groups, particularly carboxyl groups, become protonated, which reduces their ability to bind Pb^{2+} ions due to competition with H^+ ions. As the pH increases within the range of 3.0-6.0, deprotonation of the functional groups enhances the coordination interaction between the biopolymer surface and metal ions, resulting in increased adsorption efficiency. However, at higher pH values, the formation of insoluble lead hydroxides may affect the adsorption process.

In addition, the presence of competing ions such as Ca^{2+} and Mg^{2+}, commonly found in real wastewater systems, may influence the sorption behavior of the obtained biopolymers. These ions can compete with Pb^{2+} for available adsorption sites, potentially leading to a decrease in sorption capacity. Nevertheless, due to the high affinity of carboxyl and hydroxyl functional groups toward heavy metal ions, the studied biopolymers are expected to maintain significant adsorption performance in multicomponent aqueous systems.

4. Conclusions

1. It has been experimentally established that polysaccharide biopolymer matrices isolated from Dahlia tubers and Helianthus tuberosus have pronounced sorption activity with respect to Pb^{2+} ions in model aqueous solutions.

2. Scanning electron microscopy (SEM) shows that the studied biopolymers are characterized by a lamellar-layered morphology with a high degree of particle agglomeration. At magnifications of 9529× and 6019×, the structure is highly dense and compact with reduced porosity, while at 4675× and 3454×, interaggregate voids and pronounced macroporosity are revealed. The presence of finely dispersed inclusions indicates structural heterogeneity of the biopolymer matrix.

3. It has been established that developed inter-aggregate porosity contributes to the effective diffusion of Pb^{2+} ions to active functional centers ($–COO^-$, –OH), which has a positive effect on the intensity of the sorption process.

4. Kinetic studies have shown that the sorption process is characterized by a rapid initial stage and the attainment of equilibrium within 20 min, reflecting the high availability of active sites and the efficient transport of Pb^{2+} ions to the sorbent surface.

5. The biopolymer isolated from Dahlia tubers demonstrated higher sorption capacity compared to the material from Helianthus tuberosus, which is associated with greater involvement of ionized carboxylate groups in the coordination binding of Pb^{2+}.

6. Analysis of sorption isotherms showed that Freundlich's model most adequately describes the experimental data, which indicates the heterogeneous nature of the surface of biopolymer sorbents and the presence of active centers with different adsorption energies.

7. The results of IR Fourier spectroscopy confirmed the participation of carboxyl and hydroxyl functional groups in the binding of Pb^{2+} and the implementation of a coordination-chelate complex formation mechanism while maintaining the integrity of the polysaccharide skeleton.

Thus, the combination of SEM data, IR spectroscopy, kinetic analysis, and isotherm modeling confirms the promise of polysaccharide biopolymers from plants of the Asteraceae family as effective and environmentally safe sorption materials for the purification of aqueous media from heavy metals.

Author Contributions

Aliya Kamysbayeva: Investigation; Formal Analysis; Data Curation; Writing – Original Draft. **Mukhametkali Mataev:** Supervision; Methodology; Formal Analysis; Writing – Review & Editing. **Gulbaira Azimbayeva:** Supervision. **Amangeldi Meldeshov:** Validation; Resources. **Uldana Beysenbiyeva:** Investigation; Formal Analysis. **Amirbek Moldabayev:** Investigation.

Conflict of Interest

The authors declare that they have no conflict of interest.

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Структурные и сорбционные характеристики наноструктурированных полисахаридных биополимеров растений семейства Asteraceae

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АННОТАЦИЯ

В работе исследованы структурные и сорбционные характеристики нано-структурированных полисахаридных биополимеров, выделенных из растений семейства Asteraceae (Dahlia tubers и Helianthus tuberosus), по отношению к ионам Pb^{2+} в модельных водных растворах. Биополимеры получали методом кислотной экстракции при pH 1,5-2,5 и температуре 80-90 °C с последующим осаждением этанолом. Выход полисахаридных фракций составил 10-14%.

Морфология поверхности исследована методом сканирующей электронной микроскопии, что позволило выявить формирование иерархически организованной нано- и микроструктурированной матрицы с выраженной агломерацией частиц и развитой межагрегатной пористостью. Наличие пластинчатых структур и пористой системы способствует диффузии ионов металла к активным функциональным центрам.

Механизм взаимодействия биополимерной матрицы с Pb^{2+} изучен методом ИК-спектроскопии. Зафиксированы смещения и изменения интенсивности полос, соответствующих карбоксильным и гидроксильным группам, что подтверждает реализацию координационно-хелатного механизма комплексообразования без разрушения полисахаридного скелета.

Сорбционные исследования проводили на модельных растворах Pb^{2+}, приготовленных из стандартного раствора ацетата свинца (II) аналитической чистоты. Остаточную концентрацию ионов Pb^{2+} после контакта с биополимером определяли методом атомно-абсорбционной спектрометрии на приборе PinAAcle 900T (PerkinElmer, США) при аналитической длине волны 283,3 нм с использованием калибровочной зависимости. Дополнительно контрольные измерения выполняли титриметрическим методом в ацетатной буферной среде (pH 5,5) с применением стандартного 0,01 М раствора трилона Б.

Установлено, что до 76-77% Pb^{2+} удаляется в течение первых 10 мин, а сорбционное равновесие достигается через 60 мин. Максимальная степень очистки составила 87%. Экспериментальные данные наиболее адекватно описываются моделью Фрейндлиха, что указывает на гетерогенный характер поверхности и многослойный механизм адсорбции.

Полученные результаты подтверждают перспективность нано-структурированных полисахаридных биополимеров растений семейства Asteraceae как экологически безопасных сорбционных материалов для удаления ионов тяжелых металлов из водных сред.

Ключевые слова: полисахаридные биополимеры, сорбция; ионы свинца Pb^{2+}, Dahlia tubers, Helianthus tuberosus

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Asteraceae тұқымдасы өсімдіктерінің нано-құрылымданған полисахаридті биополимерлерінің құрылымдық және сорбциялық сипаттамалары

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АҢДАТПА

Жұмыста Asteraceae тұқымдасына жататын өсімдіктерден (Dahlia tubers және Helianthus tuberosus) бөлініп алынған нано-құрылымданған полисахаридті биополимерлердің Pb^{2+} иондарына қатысты құрылымдық және сорбциялық сипаттамалары зерттелді. Биополимерлер қышқылдық экстракция әдісімен (pH 1,5-2,5, температура 80-90 °C) алынып, кейін этанолмен тұндырылды. Полисахаридті фракциялардың шығымы 10-14% құрады.

Бет морфологиясы сканирлеуші электрондық микроскопия әдісімен зерттеліп, бөлшектердің айқын агломерациясымен және күйаралық кеуектілігімен сипатталатын иерархиялық ұйымдасқан нано- және микроқұрылымды матрицаның түзілетіні анықталды. Пластиналы құрылымдардың және кеуекті жүйенің болуы металл иондарының белсенді функционалдық орталықтарға диффузиясын жеңілдетеді.

Биополимерлік матрицаның Pb^{2+} иондарымен әрекеттесу механизмі ИҚ-спектроскопия әдісімен зерттелді. Карбоксильді және гидроксилді топтарға сәйкес жолақтардың ығысуы мен қарқындылығының өзгеруі полисахаридті қаңқаның бұзылуынсыз координациялық-хелаттық кешен түзілу механизмі жүзеге асатынын дәлелдейді.

Сорбциялық зерттеулер Pb^{2+} иондарының модельдік ерітінділерінде жүргізілді (аналитикалық тазалықтағы қорғасын (II) ацетатының стандартты ерітіндісінен дайындалған). Биополимермен әрекеттескеннен кейінгі Pb^{2+} иондарының қалдық концентрациясы 283,3 нм аналитикалық толқын ұзындығында PinAAcle 900T (PerkinElmer, АҚШ) атомдық-абсорбциялық спектрометрінде калибрлеу графигі бойынша анықталды. Қосымша бақылау өлшеулері ацетатты буферлік ортада (pH 5,5) 0,01 М трилон Б ерітіндісін қолдану арқылы титриметриялық әдіспен жүргізілді.

Алғашқы 10 мин ішінде Pb^{2+} иондарының 76-77%-ға дейін жойылатыны, ал сорбциялық тепе-теңдік 60 мин орнайтыны анықталды. Максималды тазарту дәрежесі 87% құрады. Эксперименттік деректер Фрейндлих моделімен жақсы сипатталады, бұл беттің гетерогенді сипатын және адсорбцияның көпқабатты механизмін көрсетеді.

Алынған нәтижелер Asteraceae тұқымдасы өсімдіктерінен алынған нано-құрылымданған полисахаридті биополимерлердің сулы ортадан ауыр металл иондарын жоюға арналған экологиялық қауіпсіз сорбциялық материалдар ретінде болашағы зор екенін дәлелдейді.

Түйін сөздер: полисахаридті биополимерлер, сорбция, Pb^{2+} қорғасын иондары, Dahlia tubers, Helianthus tuberosus