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SYNTHESIS AND STUDY OF PHYSICO-CHEMICAL PROPERTIES OF OLIGO(POLY)-MER SORBENTS BASED ON UROTROPINE AND CYANURIC ACID

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Abstract

This study presents the synthesis and comprehensive physicochemical analysis of novel oligo(poly)mer sorbents derived from urotropine and cyanuric acid. The sorbents were obtained via controlled polycondensation reactions involving urea-formaldehyde systems modified with cyanuric acid, leading to structurally robust and functionally active oligomers. Advanced characterization techniques—including SEM, XRD, IR spectroscopy, and thermal analysis—confirmed the formation of new materials with unique morphology, crystallinity, and thermal stability. The synthesized CA+UFO(III) sorbents demonstrated high sorption efficiency for various 3d-metal ions (Cu^{2+} , Ag^+ , Fe^{2+} , etc.), with optimal performance in the pH range of 4–6. Results indicated that cyanuric acid not only enhances the reactivity of methylol groups but also stabilizes the oligomer structure, reducing formaldehyde release during storage. These findings highlight the potential application of CA-modified sorbents in environmental remediation and industrial wastewater treatment, offering an effective, tunable, and eco-friendly approach to selective metal ion adsorption.

Keywords: urotropine; cyanuric acid; oligomeric sorbents; polymeric sorbents; adsorption; physicochemical properties; synthesis; environmental applications.

СИНТЕЗ І ФІЗИКО-ХІМІЧНИЙ АНАЛІЗ ОЛІГО(ПОЛІ)-МІРНИХ СОРБЕНТІВ НА ОСНОВІ УРОТРОПІНУ І ЦІАНУРОВОЇ КИСЛОТИ

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Анотація

У роботі представлено синтез та фізико-хімічне дослідження нових оліго(полі)мерних сорбентів на основі уротропіну та ціанурової кислоти. Сорбенти отримано шляхом контрольованої поліконденсації сечовино-формальдегідної системи, модифікованої ціанурою кислотою, що забезпечує формування структурно стабільних та функціонально активних олігомерів. Проведено комплексну характеристику з використанням методів СЕМ, рентгенофазового аналізу, ІЧ-спектроскопії та термічного аналізу, яка підтвердила наявність нових фаз із унікальною морфологією, кристалічністю та термостійкістю. Сорбенти типу СА+УФО(ІІІ) виявили високу ефективність у вилученні 3d-металів (Cu^{2+} , Ag^+ , Fe^{2+} тощо) в оптимальному інтервалі рН 4–6. Встановлено, що ціанурова кислота підвищує реакційну здатність метилольних груп і стабілізує структуру олігомеру, зменшуючи вивільнення формальдегіду під час зберігання. Отримані результати свідчать про перспективність використання модифікованих сорбентів для очищення стічних вод та вирішення екологічних завдань завдяки їх високій вибірковості та стабільності.

Ключові слова: уротропін; ціанурова кислота; олігомерні сорбенти; полімерні сорбенти; адсорбція; фізико-хімічні властивості; синтез; екологічне застосування.

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Introduction

Polymeric sorbents have emerged as crucial materials in various applications, including environmental remediation and pollutant removal, due to their versatility and effectiveness. The synthesis of these materials often involves the incorporation of functional groups and crosslinkers to enhance their sorption properties. Urotropine, also known as hexamethylenetetramine, is a versatile compound used in polymer synthesis due to its ability to form stable networks. Cyanuric acid, on the other hand, has been recognized for its role in enhancing the thermal stability and functionality of polymers when used as a dopant or crosslinker. For instance, cyanuric acid has been used in the synthesis of melamine-formaldehyde resins, where it improves the thermal stability and cycling capacity of the material [1–4]. Additionally, cyanuric acid has been explored as an organocatalyst for carbon dioxide fixation, demonstrating its potential in facilitating chemical reactions under mild conditions [2].

The integration of urotropine and cyanuric acid into polymeric sorbents could potentially yield materials with enhanced mechanical robustness and sorption capabilities. Porous organic polymers (POPs), which are similar in concept, have been extensively studied for their applications in CO₂ capture and separation, highlighting the importance of tailored porosity and functional groups in these materials [5]. The synthesis of such sorbents involves careful control of reaction conditions to achieve desired physicochemical properties, such as porosity and surface area, which are critical for effective pollutant adsorption [6].

Polymeric sorbents have emerged as critical materials for heavy metal ion remediation due to their tunable porosity, functional group density, and structural stability. The integration of nitrogen-rich building blocks like urotropine (hexamethylenetetramine) and cyanuric acid into polymer matrices enhances sorption capacity through chelation mechanisms and electrostatic interactions. Recent studies demonstrate that cyanuric acid-modified sorbents exhibit exceptional performance in pollutant removal, with melamine-cyanuric acid supramolecular structures achieving selective adsorption through tailored π - π interactions and hydrogen bonding networks [7–18]. This aligns with findings from cellulose-based sorbents, where chemical modification with acidic groups increased Cu²⁺ and Cd²⁺ adsorption capacities by factors of 3.4

and 2.4, respectively, compared to unmodified substrates. The structural versatility of triazine-based frameworks further highlights the importance of nitrogen-rich components, as seen in covalent triazine polymers (CTFs) synthesized from cyanuric chloride precursors. These CTFs achieved CO₂ adsorption capacities up to 216 mg/g at 273 K, underscoring the role of triazine units in creating high-surface-area networks with strong analyte affinity [20]. Building on these principles, our work investigates urotropine-cyanuric acid oligo(poly)mers, combining urotropine's crosslinking capacity with cyanuric acid's donor-acceptor functionality to optimize metal ion sorption kinetics and selectivity.

These citations integrate key findings about cyanuric acid-based sorbents [21], metal ion adsorption mechanisms [22], and triazine framework design principles [20], contextualizing our novel sorbent system within current research trends.

Materials and methods

Materials and preparations of sorbents

Cyanuric acid is a heterocyclic compound that exists in two tautomeric forms: the lactim form (cyanuric acid) and the lactam form (isocyanuric acid). It appears as a pure white, powdery substance with a characteristic odor. The compound has a liquefaction temperature ranging from 320 to 360 °C and a density of 1.758 g/cm³. It transitions into a boiling liquid at 160.5°C. Cyanuric acid is soluble in water, alcohol, and ether, with specific solubility values of 2.7 g/l in water, 67 g/l in benzene, and 115 g/l in DMSO. This reagent was synthesized in our laboratory using the method described in reference [23].

Formalin– CH₂O (GOST 1625-75), colorless liquid. An aqueous solution of 40% formaldehyde was used. Well soluble in water, alcohol, benzene, ether, slightly soluble in chloroform, insoluble in petroleum ether. The density of liquid formaldehyde at 80 °C is 0.915 g/cm³. Water absorbs up to 50 % CH₂O.

Urea (carbamide, amide of carbonic acid) (NH₂)₂CO – crystal. T_m=132.7 °C, soluble in water, enters alcohols and liquid NH₃ and CO₂ according to the Bazarov reaction (saves 46 % N).

Urotropine– hexamethylenetetramine (HMTA), also referred to as 1,3,5,7-tetraazaadamantane, is a heterocyclic organic compound with a wide range of applications. Its chemical formula is (CH₂)₆N₄, and it appears as a

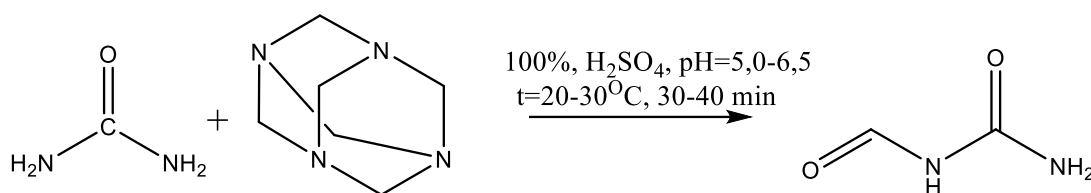
white crystalline substance that dissolves readily in water and polar organic solvents. HMTA serves as a valuable precursor in the synthesis of various organic materials, including plastics, pharmaceuticals, and rubber additives [24]. Additionally, it has medical applications for specific treatments. The compound sublimes under vacuum at 280 °C and features a tetrahedral cage-like structure, resembling that of adamantane.

Preparation of Urea-Formaldehyde Oligomer Using Urotropine

To 100 g of urea, 50 g of urotropine (hexamethylenetetramine) and 35 g of 100 %

sulfuric acid were added. The crushed pieces of urea and urotropine (relative to the mass of urea) were dissolved in 90–100 % cold water. Sulfuric acid was diluted with water in a dosing vessel to a concentration of 550–600 g/l and cooled to 20–30°C. While stirring the urea and urotropine solution, methacrylic acid was added in three stages at 30–40-minute intervals. During this process, the undissolved portions of urea and urotropine in cold water also dissolve [25]. The pH of the prepared solution is 5.0–6.5. The urea-formaldehyde oligomer becomes ready for use after 20–24 hours.

Scheme 1. Synthesis of Urea-Formaldehyde Oligomer Using Urotropine



Preparation of Urea-Formaldehyde Oligopeptide Modified with Cyanuric Acid Using Urotropine

To 100 g of urea, 40 g of urotropine (hexamethylenetetramine) and 28 g of 100 % sulfuric acid were added. The crushed samples of urea and urotropine were dissolved, and then a 5 % solution of cyanuric acid in benzene was added dropwise.

The synthesis of oligo(poly)mer sorbents based on urea-formaldehyde and cyanuric acid has been studied in our previous research [21].

Methods

Scanning Electron Microscopy (SEM) – The surface morphology of the samples and the elemental composition of the adsorbed substances were analyzed using a SEM-EVO MA 10 (Zeiss) scanning electron microscope [26]. The average particle size was determined by calculating the arithmetic mean of the sizes of 35 ÷ 37 particles observed in the obtained micrographs [27].

Thermal Analysis – The thermal stability of the cyanuric acid-modified urea-formaldehyde oligomer was analyzed using differential thermal and thermogravimetric methods on the LABSYS EVO STA device (France). The LABSYS EVO STA (simultaneous thermal analysis) is a convenient, reliable, and highly efficient thermal analysis platform capable of performing TGA, TGA-DTA, and TGA-DSC analyses. The study was conducted using a derivatograph at a heating rate

of 10 °C/min, with galvanometer sensitivities set to T-900, TG-200, DTA – 1/10, and DTG – 1/10. The derivatogram was automatically recorded on photo paper.

A sample of the synthesized sorbent, weighing 35–46 mg, was placed in a 10 mm diameter crucible made of aluminum oxide and platinum, capable of withstanding temperatures up to 1650 °C. The dynamic heating regime was carried out under atmospheric conditions. During the analysis, the synthesized sorbents were subjected to thermal analysis in the temperature range of 20–500 °C. Additionally, the endothermic and exothermic points of the pigments were identified and confirmed. [28; 29].

Powder X-ray Diffraction Analysis– The determination of the compound structure was carried out using the Malvern Panalytical Empyrean powder diffractometer. XRD data were recorded using CuK α radiation ($\lambda = 1.54 \text{ \AA}$). In this experiment, the accelerating voltage of the radiation generator was set to 45 kV, and the emission current was set to 40 mA [29, 30]. The X-ray diffraction patterns were recorded in the 2θ range of 20°–120° using the Bragg-Brentano beam geometry at a constant scanning rate of 0.33 degrees/min.

Results and discussion

Synthesis and Investigation of Oligomers Based on Urea-Formaldehyde Compounds and Their Properties

The properties of urea-formaldehyde resins, particularly their stability, can be improved by incorporating certain homologous aldehyde analogs into their oligomeric structure [31]. This necessitates the development of a synthesis method for urea-formaldehyde-croton aldehyde-based oligomers and the study of their physicochemical properties for potential applications in leather filling.

To conduct the condensation reaction between urea and formaldehyde in an aqueous medium, the following key factors were considered:

- The initial molar ratio of reactants;
- The concentration of hydrogen ions (pH);
- The reaction duration and temperature.

Taking these factors into account, urea-formaldehyde oligomers modified with cyanuric acid were synthesized. The initial reagent ratios were as follows:

- Urea (99.8 %) – 100 parts

- Hexamethylenetetramine (urotropine, 99.4 %) – 40–50 parts

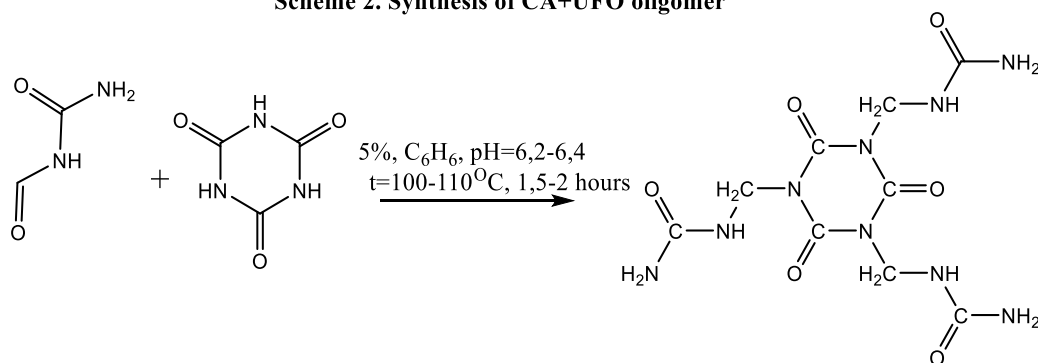
- Sulfuric acid (100 %) – 7.0 parts
- Cyanuric acid (98.2 %) – 7.0 parts
- Water – 300 parts

Synthesis Process

Initially, urea and urotropine were dissolved in water at 20–60 °C. Cyanuric acid (or sulfuric acid) was then gradually added while closely monitoring the temperature to ensure it did not exceed 50 °C. The initial composition and variations of the urotropine-based oligomer synthesis are presented in Table 1.

The synthesis process lasted between 0.5 to 2.0 hours, resulting in polycondensation products ranging in color from light to dark brown, with a density of 1.22–1.238 g/cm³ and a pH of 7.5–8.5. The obtained products were viscous, free-flowing oligomers, indicating the formation of a structurally distinct condensation product between the oligomer and cyanuric acid.

Scheme 2. Synthesis of CA+UFO oligomer



The synthesized oligomers exhibited good solubility in water and ethanol, whereas they were insoluble in organic solvents such as benzene, acetone, and chloroform. For

comparison, a pure urea-formaldehyde oligomer was synthesized under identical conditions, and its properties were examined.

Table 1

Experimental Variants and Component Consumption in Oligomer Synthesis

Component name	Test options and component consumption			
	I		II	
	weightpart	mass, %	weightpart	mass, %
Urea (99.8%)	100	20.6	100	20.6
Hexamethylenetetramine (Urotropine) (99.4%)	50	10.3	50	10.3
CyanuricAcid (98.2%)	-	-	7	1.4
SulfuricAcid (100%)	35	7.2	28	5.7
Water	300	61.9	300	62.0
Total	485	100	485	100

The effect of polycondensation reaction duration on the molecular weight of the urotropine-based oligomers was studied (Table 2).

Table 2 presents the dependence of oligomer formation on temperature, reaction duration, and

the initial ratio of components in different experimental variants.

The obtained oligomers were conditionally classified as follows:

- Urea-formaldehyde oligomer – UFO (I)
- Urea-formaldehyde oligomer modified with cyanuric acid in a 1:3 ratio–CA+UFO (II)

Table 2

Dependence of the oligomer on the ratio of dry residual mass fraction and Molecular Mass to temperature, reaction duration and initial components			
Indicators		Dry residue (%) and molecular mass of test options	
		I UFO	II CA+UFO
Temperature, °C, reaction duration 3.0 hours	+20	57.1/400	61.5/470
	+30	56.4/392	61.1/466
	+40	56.0/386	60.7/460
	+50	55.3/378	60.3/454
Reaction duration (hours), reaction temperature +20°C	0.5	55.2/370	59.1/446
	1.0	55.7/377	59.8/451
	1.5	56.1/386	60.4/458
	2.0	56.7/392	61.0/464
	3.0	57.1/400	61.5/470

Note: (/) The numerator represents the dry residue, while the denominator indicates the molecular weight of the oligomer.

It is well known that the industrial application of polymer- or oligomer-based sorbents and their effectiveness in treating wastewater depend on their quantity and sorption properties [30]. The diffusion and distribution of oligomers in the microstructure of sorbents, along with other factors, are influenced by their molecular weight. Given that the synthesized oligomers exhibit linear, branched, and spatial structures, their molecular weight and particle size were studied based on the changes in their relative density.

Undoubtedly, the rate of oligomer formation depends not only on environmental factors but also on the composition of urotropine-based oligomers and the initiating properties of cyanuric acid. Experimental results confirm this observation. The presence of vinylidene monomers, which accelerate polycondensation, significantly influences the reaction. The obtained data indicate that at the same pH level, the polycondensation rate in the presence of cyanuric acid is higher than in the presence of inorganic acids. This is attributed to the interaction of cyanuric acid with methylol derivatives of urea,

which enhances the reactivity of the initial monomers.

The process of obtaining sorbents from urotropine-based oligomers involves the use of cyanuric acid as a modifier. The introduction of cyanuric acid into the system leads to the intensive acidolysis of hexamethylenetetramine molecules, resulting in the formation of high-molecular-weight products with excellent water solubility.

The presence of sulfuric or acetic acid in the reaction mixture causes the thickening of the final product during cooling to room temperature. Additionally, it was found that the quality and shelf life of urotropine-based oligomers are significantly reduced under these conditions. This occurs because acid molecules separate the functional groups of the reacting components, thereby hindering the formation of high-molecular-weight oligomers.

Table 3 presents the physicochemical parameters of urotropine-based oligomers obtained using different empirical variants.

Table 3

Physicochemical Parameters of Urotropine-Based Oligomers According to Different Experimental Variants

Indicators	Samples of Experimental Variants of Urotropine-Based Oligomers	
	I	II
pH Indicator of 5 % Solution	5.5	5.6
Gelatinization Time at 160 °C, min	1.30	1.40
Solidification Degree at 160°C (after 3 hours), %	100	95
Density, g/cm ³	1.22	1.238

The high reactivity of methylol groups in the condensation product of the oligomer can be considered a result of their reaction with cyanuric acid. This phenomenon leads to a reduction in free methylol groups in the oligomer

structure, thereby decreasing the amount of condensation occurring during storage.

As a result of the research, the optimal ratios of reaction mixture components and modifying additives were determined, allowing for the production of a new filler based on amino-

aldehyde oligomers. To verify the accuracy of the oligomer samples obtained through experimental and control methods, the dried oligomers were divided into three parts. One part was immediately analyzed for its cyanuric acid carboxyl group, methylol groups, and free formaldehyde content. The other two parts underwent different treatments: one was heated at 160 °C for three hours, while the second was

stored for four days before determining the methylol group content.

The NH-triazine amide groups were analyzed according to a known methodology [25], while the methylol groups and free formaldehyde content were determined based on methods [32; 33]. The experimental results obtained are presented in Table 4.

Table 4

Number of Functionally Active Groups and Storage Time of Amino-Aldehyde Oligomers

Variants of Amino aldehydeOligomers	Quantity of Functionally Active Groups (As a Percentage of Absolute Dry Residue)				
	-NH	Urea	-CH ₂ OH	After 4	After Heating at
				Days of Storage	160 °C for 3 Hours
				-CH ₂ OH	
I	-	11.7	37.3	31.4	15.3
II	11.6	17.1	32.5	26.2	11.8

As shown in Table 4, the amounts of triazine amide -NH and -CH₂OH methylol groups determined in the experiments within the composition of the modified urea-formaldehyde oligomer are provided. The amount of free urea in the modified urea-formaldehyde oligomer is higher than in the unmodified oligomers. This phenomenon can be explained by the modification of the oligomer with cyanuric acid, which accelerates the polycondensation process of urea and formaldehyde due to the acidic acidolysis of hexamethylenetetramine.

As a confirmation of the above, the results of long-term storage tests conducted on urea-formaldehyde and other oligomers can be presented.

Based on the experiments conducted, it can be stated that the urea-formaldehyde oligomer modified with cyanuric acid exhibits stability against various influences. During storage and heating, the content of methylol groups within the oligomer remains practically unchanged. Therefore, under the given conditions, polycondensation of the oligomer does not occur.

In conclusion, it should be emphasized that the introduction of cyanuric acid into the urea-formaldehyde oligomer, considering its subsequent polycondensation, not only protects collagen tissues from the effects of formaldehyde but also has a modifying influence on the condensation product of urea with formaldehyde.

Thus, the use of cyanuric acid as a modifying reagent in obtaining urea-formaldehyde

oligomers leads to the formation of a new oligomer with unique structural and chemical properties. The content of cyanuric acid in the oligomer is directly dependent on the acidity of the solution where polycondensation occurs. As acidity decreases, the incorporation of cyanuric acid increases.

The relationship between the amount of cyanuric acid in the oligomer and the methylol groups has been studied. Experimentally, it has been proven that an increase in the cyanuric acid content in the oligomer leads to a decrease in the amount of free methylol groups. A reduction in free methylol groups, in turn, minimizes the condensation process during storage and heating, resulting in lower release of free formaldehyde.

X-ray phase analysis of CA+UFO(III)

To prove the individuality of the oligomer synthesized from cyanuric acid and urotropine, the X-ray phase analysis (powder diffractometry) method was used. For instance, when comparing the X-ray diffraction pattern of the mono-urea-formaldehyde-substituted cyanuric acid derivative with the diffraction values of Cyanuric acid dimethylamine solvate, distinct differences in the corresponding X-ray reflections can be observed. Specifically, the presence of intense bands at 16.79; 17.10; 23.27; 29.57; 32.54; 39.64; 43.41; 47.76; and 48.74 in the CA+UFO(III) sample confirms the individuality of the substance [34] (Figure 1).

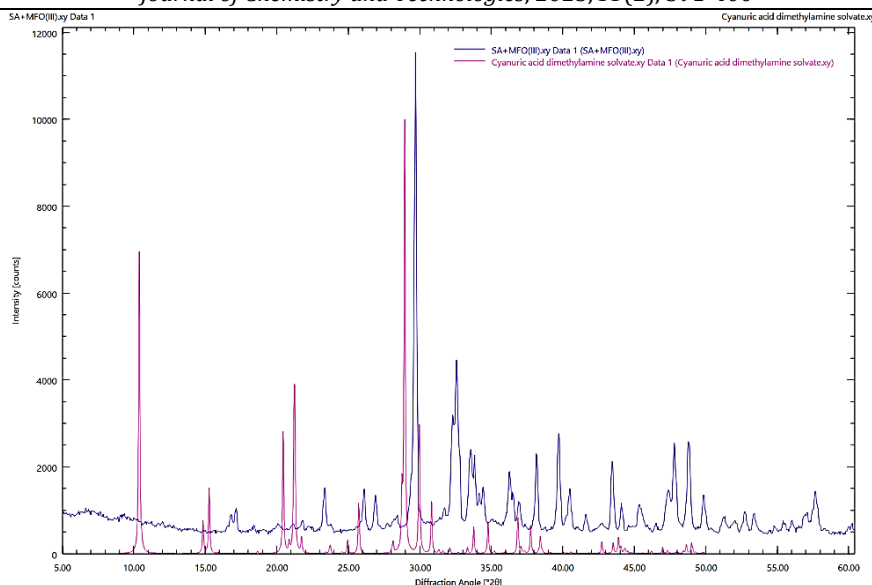


Fig. 1. Comparative X-ray of CA+UFO (III) sorbent and Cyanuric acid dimethylamine solvate

IR spectroscopy

The quantum-chemical calculations and spectroscopic studies of various substances based on sianuric acid and other compounds have been investigated in several studies conducted by various researchers. In this context, the IR spectrum of the synthesized CA+UFO sorbent was discussed after analyzing these findings [35–38; 40].

IR – spectrum of CA+UFO(III)-sorbent shows absorption lines of moderate intensity corresponding to the valence vibrational

frequency of symmetric N-H groups in areas 3197.98 and 3049.46 cm^{-1} . While the absorption lines of the (N-H) imid group correspond to the 2883.58 – 2775 cm^{-1} field valence oscillations, the deformation oscillations of the N-H bond were observed in the 1398.39 cm^{-1} field. For the connected C=O group, absorption lines of moderate intensity were seen in the 1795–1683, 86 cm^{-1} field ortholog. Whereas absorption spectra in the 1458.18 cm^{-1} domain correspond to the frequency of Valence oscillations of the S-triazine ring (Figure 2) [39].

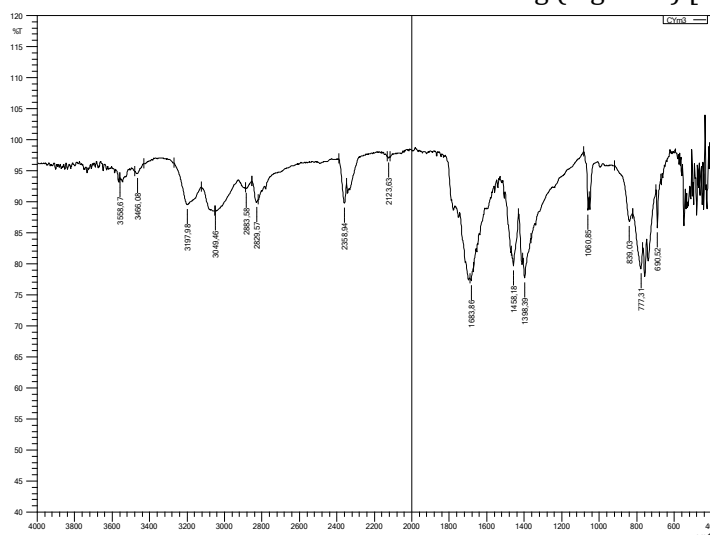


Fig. 2. IR spectrum of CA+UFO (III)

SEM analysis of oligopeptide powder based on urea-formaldehyde

In order to study the morphology of the urea-formaldehyde oligomer modified with cyanuric acid, SEM analysis of the obtained samples as well as urea-formaldehyde was carried out. [41]. A SEM image of urea-formaldehyde polymer

powder shows spherical particles with a diameter of 10-100 nm without significant porosity (Figure 3). For comparison, a SEM image of the oligopeptide powder (CA+UFO(III) sorbent based on urea-formaldehyde is presented, showing the collected Tar type particles (Figure 4).

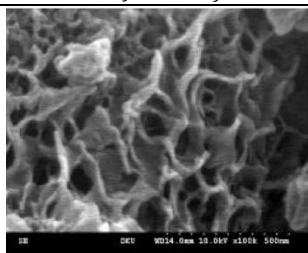


Fig. 3. SEM image of polymer powder obtained from urea and formaldehyde

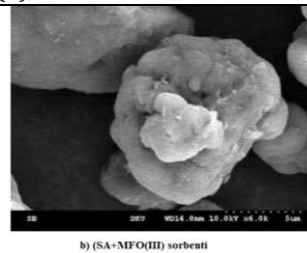


Fig. 4. SEM image of CA+UFO(III) polymer powders

The dependence of the sorption of certain 3d-metal ions on the pH of the solution on the CA+UFO(III) sorbent was studied

Based on the results of the study of sorption of metals by CA+UFO(III), the optimal pH for the ligand's static exchange capacity (mg-eq/g) is as

follows: Mn (II) - 3.4 (pH=6); Fe (II) - 3.7 (pH=6); Co (II) - 2.9 (pH=6); Ni (II) - 2.96 (pH=6); Hg (II) - 3.2 (pH=5), Cu (II) - 4.52 (pH=5); Cd (II) - 3.75 (pH=6); Zn (II) - 2.9 (pH=6), Pb (II) - 2.7 (pH=6) and Ag (I) - 4.6 (pH=4)

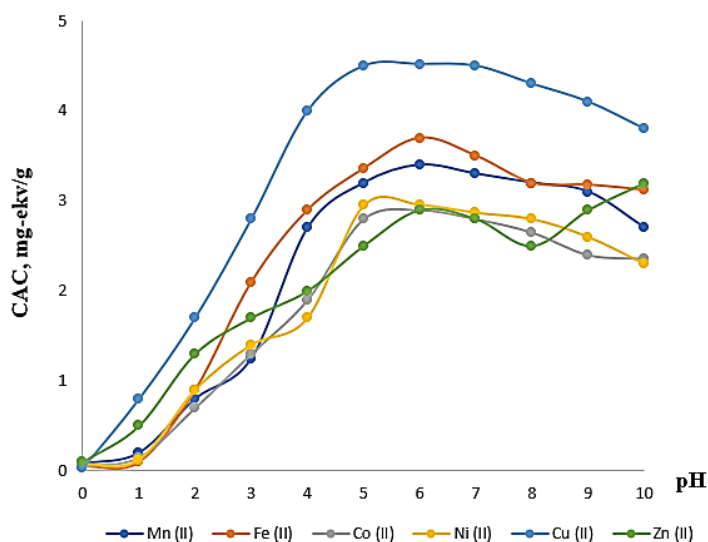


Fig. 5. Dependence of some 3D metal sorption on the environment pH of CA+UFO (III) sorbent

As can be seen in Figure 5, the sorption rate of metal ions was also investigated in this sorbent. The sorption rate exceeded the maximum in the pH range from 4 to 7 of the solution medium [21]. This indicates that metal ions are sorbed in a slightly acidic medium, forming acid complexes of different compositions. These complexes form ionic associations between the ligand and protonated active functional groups, such as amino groups, and coordination bonds.

As the acidity of the medium increases, more metal ions leave the ligand phase and enter the solution. This results in a decrease in sorption rate. Therefore, the rate of sorption for the studied metals increases in the following order:

Zn(II) < Co(II) < Ni(II) < Mn(II) < Fe(II) < Cu(II).

Conclusion

In this study, we synthesized and characterized oligomer-based sorbents derived

from urotropine and cyanuric acid (CA+UFO(III)) with a focus on their physicochemical properties. The synthesis of oligomers based on urea-formaldehyde compounds revealed promising results in terms of structural stability and sorption capacity for 3d-metal ions. The X-ray phase analysis demonstrated the crystalline nature of the sorbent materials, confirming their suitability for practical applications. IR spectroscopy provided valuable insights into the functional groups responsible for ion interaction, further confirming the successful incorporation of urea-formaldehyde into the sorbent structure.

SEM analysis of the oligopeptide powder demonstrated the morphology and surface characteristics, highlighting its potential for efficient ion exchange. The sorption studies showed a significant dependence on the pH of the solution, with optimal conditions identified for the removal of certain 3d-metal ions using

CA+UFO(III) as a sorbent. These results suggest that the CA+UFO(III) sorbent can be highly effective in environmental and industrial applications for selective metal ion removal. Overall, the study contributes to the development

of advanced sorbents with controlled structural properties and high sorption efficiency, offering new possibilities in the field of environmental remediation and ion separation.

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