

NEW COMPOSITE SORBENTS FOR CAESIUM AND STRONTIUM IONS SORPTION

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Abstract. New composite lignocellulose-inorganic sorbents based on vegetal residues, modified with ferrocyanide of different *d*-metals and hydrated antimony pentoxide, were prepared and tested for caesium and strontium ions sorption from aqueous solutions. Physical and chemical methods were used to determine the structural properties of the obtained composite sorbents. Comparative analysis of sorption properties of the obtained materials, with different types of modifiers in the bulk of lignocelluloses-carrier towards radionuclides was done. Obtained results highlight the lignocellulose-inorganic sorbent modified with potassium nickel hexacyanoferrate, being efficient for sorption of caesium ions from solution (maximum efficiency - 98.0% and maximum distribution coefficient $2.1 \cdot 10^4$ mL/g) and lignocellulose-inorganic sorbent based on hydrated antimony pentoxide – efficient for strontium ions sorption from solution (maximum efficiency - 97.3% and the distribution coefficient $1.2 \cdot 10^3$ mL/g). The sorption properties of lignocellulose-carrier and lignocellulose-inorganic samples towards heavy metal ions (Pb^{2+} and Cd^{2+}), methylene blue, gelatine and vitamin B₁₂ were also evaluated.

Keywords: lignocellulose-inorganic sorbent, caesium ion, strontium ion, sorption efficiency, distribution coefficient.

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Introduction

Radionuclides ¹³⁷Cs and ⁹⁰Sr are characterized by a long half-life of about 30 and 29 years, respectively, also by the high toxicity and migration capability; due to high solubility, radionuclides can migrate into the environment and can easily be absorbed by living organisms [1,2]. Nowadays, different methods such as coagulation, extraction, precipitation, membrane filtration, etc., are widely used for removal of radioactive compounds from contaminated water [3]. Among all methods, sorption technique is the simplest, available and effective method.

Different types of sorbents, such as zeolite [4], clays [5], activated carbon [6], hydrous metal oxides [7] or titanate and silicotitanates [8,9], and others, can be used for the radionuclides removal from water solutions. To increase selectivity of natural minerals towards radionuclides, it has been proposed to impregnate the carriers with specific compounds that show selectivity for certain ions.

Ferrocyanides of *d*-metals ([FC]M, where M - metal cations) are characterized by high selectivity towards caesium ions [10], and

hydrated antimony pentoxide (HAP) is characterized by high sorption capacity towards strontium ions [11]. To improve the operational performance of [FC]M and HAP, their immobilization on porous media is applied. For the obtaining of selective sorbents based on [FC]M and HAP it is appropriate to use plant materials as carrier-matrix, for example, vegetal residues from agriculture or food industry. Another advantage of using plant materials is the possibility of their utilization by the thermal degradation that can reduce the volume of solid wastes [12].

Intensive pollution of the environment with radionuclides, heavy metals and other harmful substances is an important environmental problem. Adverse health effects of inorganic toxicants are caused by their ability to accumulate in living organisms, inducing intoxication, pathological changes and a reduction in the average length of life. The adsorption of soluble forms of nuclides by the body tissues is fast and reaches almost 100%. A solution to this problem can be the sorption therapy and prophylaxis with enterosorbents of different nature. Enterosorbents

are distinguished by dosage forms, chemical structure, physical properties, adsorption mechanism and selectivity. Modern enterosorbents are produced as powder, granules, pills, gels, pastes, suspensions, colloids that are mainly divided into 4 types: based on silica, carbon, natural organic polymers (*e.g.* alginate, pectin, chitin, cellulose fibers) and composite (the composition can include two or more of mentioned enterosorbents). Such enterosorbents are not selective towards radionuclides.

Enterosorbents based on vegetal materials that, unlike synthetic ones, are biocompatible and present a soft action on the mucous membrane of the gastrointestinal tract, can be effectively used for sorption therapy. Modification of lignocellulose (LC) sorbents with [FC]M or HAP will allow to obtain enterosorbents for selective binding of caesium and strontium radionuclides.

This study is devoted to the preparation of composite LC-inorganic sorbents derived from vegetal residues of agriculture and food industry, modified with [FC]M and HAP, and investigation of their structural properties using physical and chemical methods and sorption ability towards toxicants. The main idea of using lignocellulose material as a carrier of [FC]M and HAP was to combine the properties of both modifiers and plant material to achieve high radionuclides and other inorganic and organic substances sorption.

Experimental

Materials

The residues of agriculture and food industry such as cherry, apricot and peach stone shells, hazelnut shells, corncob and buckwheat peel, were used as raw material. Chemical analyses of studied materials were carried out according to TAPPI standards, namely: *T-222* for lignin, *T-204* for ethanol-benzene extractable and *T-211* for ash. The cellulose analysis was done according to Kurschner-Hoffer method, based on treatment of plant materials with a mixture of nitric acid and ethanol (1:4) four times in a hot water bath followed by filtration and drying of the cellulose residue. Materials were ground and sieved, and the fraction with a diameter of 0.12 mm and a moisture content of 4% was used for further processing. Plant materials were stored in desiccator to ensure constant humidity and chemical composition.

In order to prepare the LC-carriers and the modifying solutions of [FC]M and HAP, reagents of AR grade were used: hydrochloric acid (HCl), sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$), potassium hexacyanoferrate ($\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$),

iron sulphate ($\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$), nickel sulphate ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$), cobalt sulphate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$), copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) and antimony chloride (SbCl_5). Deionised water from a Millipore-MilliQ system ($\rho=18 \text{ M}\Omega \cdot \text{cm}$) was used throughout the experiments.

Preparation of LC-carriers

The LC-carriers were prepared from vegetal residues by an acid-alkaline treatment with a 3.0 N solution of hydrochloric acid and the subsequent partial delignification of the obtained product with a 0.3 N solution of sodium carbonate. Both steps were carried out for 60 min at 90°C, the ratio of solid to liquid was 1: 5. The LC-product was separated from the solution by filtration, and then washed with distilled water at 80°C to achieve a neutral medium, dehydrated and dried at 50°C to a constant moisture content of 5–7%.

Preparation of composite sorbents

The preparation of LC-[FC]M and LC-HAP sorbents was done by impregnation of LC-carrier with colloidal solutions of [FC]M and HAP at 100°C, during 120 and 10 min, respectively. The initial colloidal solutions of [FC]M (where M = Fe^{3+} , Ni^{2+} , Co^{2+} , Cu^{2+} and Zn^{2+}) were prepared separately from LC-carrier, by mixing the solution of potassium hexacyanoferrate with the *d*-metal salt solution. The concentration of [FC]M and the molar ratio of $\text{M}^{n+}/[\text{Fe}(\text{CN})_6]^{4-}$ were 4 and 1.5 g/L, respectively. The consumption of SbCl_5 was 8%. The ratio of solid to liquid was 1:5. At the end of impregnation process the liquid phase was removed by filtration; modified LC-sorbents were washed with distilled water to achieve neutral medium, dried at 50°C to constant moisture of 5–7%, and analyzed gravimetrically.

Characterization of initial materials and composite sorbents

The infrared (IR) spectra of the samples were recorded on a Specord M80 spectrophotometer (Carl Zeiss, Germany) in the range of 4000–300 cm^{-1} , in KBr pellets (the wt. ratio was 1:9).

The specific surface area of the initial and modified materials was determined from the nitrogen adsorption isotherms at 77 K using the analyzer NOVA 2200 (Quantachrome, USA). Before the experiment, samples were heated at 140°C and outgassed at this temperature under high vacuum for 20 h. The pore volume of the materials was determined by adsorption of benzene vapours in the desiccator [13].

Adsorption properties of composite sorbents

The sorption of ^{137}Cs and ^{90}Sr ions on obtained materials was studied using radiotracer method, under static conditions. The sorbent samples of 0.05 g and 20 mL of simulated solution were used to study caesium sorption; the samples of 0.5 g and 50 mL of simulated solution were used to study strontium sorption. The initial concentration of the caesium and strontium ions was 10^{-9} and 10^{-6} mol/L, respectively; sodium nitrate and sodium chloride solutions of the concentrations 1.0 and 0.001 mol/L were used as a background. The specific activities of ^{137}Cs and ^{90}Sr in the initial solutions and in the equilibrium solutions were determined using a NRG-603 γ -ray analyzer and the liquid scintillation counter LS5000D by Beckman, respectively. Based on the changes of radioactivity before and after adsorption, the removal efficiency S (%), the distribution coefficient K_d (mL/g), the specific distribution coefficient SK_d (mL/g) and the density of ions sorption on the composite LC-inorganic sorbent were calculated by Eqs.(1)-(3):

$$S = \frac{C_0 - C_e}{C_0} \cdot 100 \quad (1)$$

$$K_d = \frac{C_0 - C_e}{C_e} \cdot \frac{V_s}{m_s} \quad (2)$$

$$SK_d = \frac{C_0 - C_e}{C_e} \cdot \frac{V_s}{m_m} \quad (3)$$

where C_0 , C_e – the initial and equilibrium concentrations of radionuclides in model solutions, respectively, mol/L;
 V – the volume of solution, L;
 m_s and m_m – the weight of sorbent and modifier in the composite LC-inorganic sorbent, respectively, g.

Methylene blue adsorption was carried out at the concentration of 100 mg/L. Dye solutions were prepared in 0.15 M phosphate buffer with pH 6.0. Sorbent sample was 0.20 g, the volume of solution – 25 mL.

Vitamin B₁₂ and gelatin sorption were performed from the model solutions with pH 2.0 and 7.5 according to the earlier described procedure [14]. The sorption of metals ions (Pb^{2+} and Cd^{2+}) was done in static conditions using model solutions with the concentration of 50 mg/L and pH value of 6.0 during 24 h at constant stirring (sorbent mass – 0.2 g, solution volume – 50 mL). The metals ions were determined using flame atomic absorption spectrometry (AA-6300 Shimadzu). Each

experiment was repeated five times and then averaged. The relative standard deviation of multiple measurements was less than 5%.

Results and discussion

Different vegetal residues from agriculture and food industry such as buckwheat peel, hazelnut shells, corncob, cherry, apricot and peach stones shells were tested. The treatment of these materials by acid-alkaline scheme allows to obtain LC-carriers or sorbents enriched with lignin and cellulose (as a significant number of carbohydrates, mainly hemicelluloses), mineral components, waxes and fats were removed from the material, as shown in Table 1. The specific surface area and adsorption pore volume increased during such treatment. Sorption properties of LC-materials towards heavy metal ions, also, increase as a result of acid-alkaline treatment. LC-material from apricot stone shells is characterized by the highest removal efficiency of heavy metal ions (Pb^{2+} and Cu^{2+}), so this material was selected for further experiments (Table 1). The LC-material from apricot stone shells with the content of cellulose and lignin of 28.1 and 66.3%, respectively, was used.

Modification of the LC-material with [FC]M colloidal solutions at heating leads to the immobilization of inorganic phase on the organic surface with the formation of composite LC-[FC]M sorbents. The treatment of the LC with SbCl_5 , leads to the hydrolysis of antimony chloride in water solution with the formation of $\text{Sb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ and its sedimentation on the carrier bulk resulting in formation of LC-HAP sorbent. The modifiers content of the resulting composite LC-inorganic sorbents is shown in Figure 1. Composite LC-[FC]M sorbents contain a similar quantity of modifiers in the bulk of the LC-carrier and LC-HAP, with the modifier content of 2.8% (Figure 1). Results of the benzene vapour sorption show that the modification process leads to a reduction of the total pore volume of the obtained sorbents from 0.19 to 0.14 cm³/g for LC-[FC]M sorbents, and to 0.17 cm³/g for LC-HAP sorbent. This indicates the immobilization of the modifiers in the pores of the LC-carrier.

The IR spectra of modifiers formed in the solution are presented in Figure 2. The IR spectra of the initial [FC]M modifiers present vibrations characteristic to $\text{C}\equiv\text{N}$ at 2080–2100 cm⁻¹, Fe-CN bending vibration at 585–606 cm⁻¹ and Fe-C valence vibration at 464–499 cm⁻¹, as well as the motions from crystal water: valence vibration of O-H at 3368–3618 cm⁻¹ and bending vibration of HOH at 1610–1616 cm⁻¹.

Table 1

Material	Content of the main components, %				Surface parameters		Removal efficiency, %	
	Cellulose	Lignin	Ethanol-benzene extractable	Ash	Specific surface area, m ² /g	Adsorption pore volume, cm ³ /g	Pb ²⁺	Cu ²⁺
Cherry stone shells								
- initial material	41.2	51.3	7.8	3.8	1	0.01	53.3	47.0
- LC-material	26.7	63.7	5.0	1.5	14	0.19	64.0	66.3
Apricot stone shells								
- initial material	34.6	48.1	7.1	2.5	1	0.03	46.4	41.7
- LC-material	28.1	66.3	2.6	1.1	23	0.25	90.6	91.0
Peach stone shells								
- initial material	38.3	50.3	5.2	5.1	1	0.02	23.0	26.3
- LC-material	32.0	59.3	4.0	3.8	19	0.21	55.9	56.2
Hazelnut shells								
- initial material	45.0	55.1	2.1	8.0	1	0.01	47.0	43.2
- LC-material	37.2	47.4	1.6	5.3	12	0.19	53.2	58.3
Buckwheat peel								
- initial material	46.7	52.4	13.9	3.5	1	0.03	41.4	44.0
- LC-material	35.2	59.2	9.0	1.2	11	0.15	77.8	80.6
Corn cob								
- initial material	70.7	28.6	8.0	4.3	3	0.16	19.2	25.9
- LC-material	33.9	66.2	4.1	2.2	28	0.29	59.6	60.0

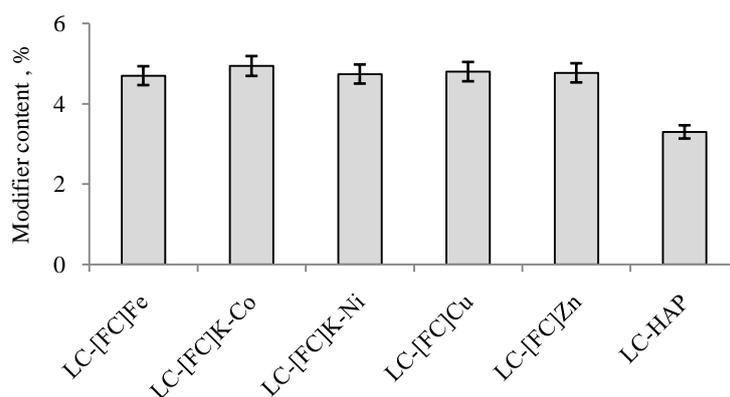


Figure 1. [FC]M and HAP content in LC-inorganic sorbents.

The IR spectrum of individual HAP is characterized by vibration at about 766 and 552 cm⁻¹ attributed to the Sb-O stretching modes of (Sb-O-Sb) and (Sb-OH), respectively [15]. The peak at 1170 cm⁻¹ indicates the presence of (Sb-OH) bending vibration. The peak at 1618 cm⁻¹ is due to the bending vibration of hydroxyl groups of molecular water. The peak at 3402 cm⁻¹ indicates the presence of O-H stretching in the hydrogen bonds.

The IR spectra of the initial LC-carrier and LC-inorganic sorbents are shown in Figure 3. The broad peaks in the region 3000–3700 cm⁻¹ indicate the presence of O–H stretching in the hydrogen bonds. Absorbed water is present in both samples, as can be seen from the peak at

1640 cm⁻¹, which is characteristic for the bending vibration of water molecules. Absorption bands in the range of 2800–3000 cm⁻¹ correspond to asymmetric and symmetric valence vibrations of C–H in the methyl (–CH₃) and methylene (–CH₂–) groups of lignin in both samples. An absorption band at 1215 cm⁻¹ indicates the presence of C–O bonds in the lignin. The IR spectra of the modified LC-[FC]M materials present an absorption band of C≡N groups in the range of 2080–2100 cm⁻¹, after immobilization of the [FC]-salts. Analysis of the IR spectra of modified organic carrier with the HAP does not show differences between the spectra because of the low modifier content, as presented in Figure 3.

The LC-material, LC-[FC]M and LC-HAP sorbents were used for ^{137}Cs and ^{90}Sr sorption, respectively (Table 2). The sample of LC-material is characterized by poor sorption ability with respect to radionuclides ions. The efficiency of ^{137}Cs and ^{90}Sr sorption is 11.2 and 12.6% and the distribution coefficient is 50.5 and 7.2 mL/g, respectively. The results of determination of the sorption efficiency of LC-inorganic sorbents with respect to ^{137}Cs and ^{90}Sr are shown in Table 2. Comparison of the efficiency of caesium sorption on LC-[FC]M shows that at approximately the same content of the inorganic component, the LC-inorganic sorbents are characterized by different absorption properties towards radionuclide.

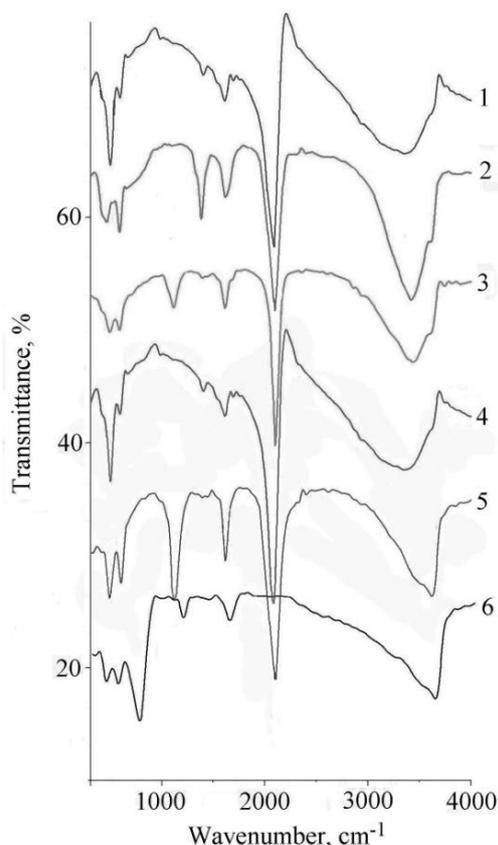


Figure 2. IR spectra of initial modifiers:
(1) – [FC]Fe; (2) – [FC]K-Co; (3) – [FC]K-Ni;
(4) – [FC]Cu; (5) – [FC]Zn; (6) – HAP.

The maximum values of removal efficiency and distribution coefficient on LC-[FC]M correspond to the sorption materials based on [FC]K-Ni and [FC]K-Co. The presented results show that the sorption properties of the sorbents towards caesium ions correspond to the following series:



The distribution coefficient of caesium ions on natural and synthetic zeolites is 20–600 mL/g, on oxyhydrate sorbents is 2–5 mL/g, on phosphates of titanium and zirconium is 700–770 mL/g [16]. So, LC-[FC]M sorbents are more effective in comparison with other inorganic sorbents (Table 2).

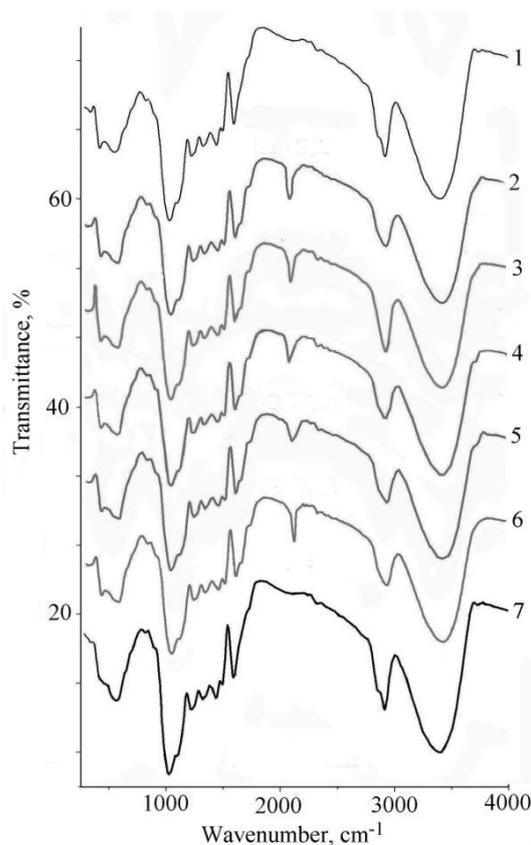


Figure 3. IR spectra of initial LC-carrier (1) and LC-inorganic sorbents modified with:
(2) – [FC]Fe; (3) – [FC]K-Co; (4) – [FC]K-Ni;
(5) – [FC]Cu; (6) – [FC]Zn; (7) – HAP.

Table 2

Sorption properties of composite LC-inorganic sorbents towards radionuclides.

LC-inorganic sorbent	Removal efficiency, %	Distribution coefficient, mL/g	Specific distribution coefficient, mL/g
LC-[FC]Fe	36.9	$2.3 \cdot 10^3$	$4.9 \cdot 10^4$
LC-[FC]K-Co	96.2	$1.0 \cdot 10^4$	$2.0 \cdot 10^5$
LC-[FC]K-Ni	98.0	$2.1 \cdot 10^4$	$4.4 \cdot 10^5$
LC-[FC]Cu	94.6	$7.0 \cdot 10^3$	$1.4 \cdot 10^5$
LC-[FC]Zn	30.9	$0.7 \cdot 10^3$	$1.7 \cdot 10^4$
LC-HAP	97.3	$1.2 \cdot 10^3$	$4.3 \cdot 10^4$

The removal efficiency of strontium ions from model solution and distribution coefficient on LC-HAP are 97.3% and $1.2 \cdot 10^3$ mL/g, respectively. According to the literature data, the distribution coefficient of strontium ions on bentonite is $1.4 \cdot 10^3$ mL/g [17], clinoptilolite – $0.8 \cdot 10^3$ mL/g, synthetic zeolites such as erionite, NaA, NaY and NaX are $0.2 \cdot 10^3$, $0.4 \cdot 10^3$, $0.2 \cdot 10^3$ and $0.5 \cdot 10^3$ mL/g, respectively [18]. The comparison of the distribution coefficient values of strontium ions on LC-HAP with the literature data, allows arguing about high effectiveness of the obtained sorbents. It was shown that pure LC-material does not show high affinity for radionuclides, so the values of the distribution coefficient of radionuclides can be recalculated

based on the mass of modifier in LC-inorganic sorbents. As can be seen, the value of the specific distribution coefficient of caesium and strontium ions on modifiers in the bulk of LC-inorganic sorbents reaches 10^4 – 10^5 mL/g.

The sorption properties of obtained materials towards organic and inorganic toxicants are shown in Table 3. It was found that modification of LC-matrix with [FC]M and HAP, in all cases causes a decrease of the total volume of pores, so the adsorption capacity for methylene blue also decreases. According to the literature data [14], sorption capacity of enterosorbents based on hydrolytic lignin from wood towards methylene blue reaches 28.9 mg/g and the value is lower than for LC-inorganic sorbents.

Table 3

Sorption properties of obtained materials towards different toxicants.

Materials	Sorption capacity, mg/g						
	Methylene blue	Vitamin B ₁₂		Gelatin		Metal ions	
		pH 2.0	pH 7.5	pH 2.0	pH 7.5	Pb ²⁺	Cd ²⁺
LC-carrier	48	1.9	2.6	0	19.7	33	14
LC-inorganic sorbents	39 – 40	1.2 – 1.5	2.0 – 2.3	0	19.6	27–29	11–12

Obtained results also demonstrate that LC-carrier shows the highest affinity to vitamin B₁₂ in a slightly alkaline solution. Overall, obtained data indicate that the studied sorbents have a low adsorptive capacity towards toxicants (with middle molecular weight) in comparison with activated carbon (10.2 mg/g), but also higher in comparison with hydrolytic lignin (1-2 mg/g) [14].

Gelatin was used as a marker for the determination of sorption activity of obtained materials towards toxins and pathogens of protein nature. It was found that, sorbents derived from plant materials do not show sorption activity towards protein compounds in acidic solutions. Sorption capacity of obtained materials towards toxin of protein nature in alkaline medium is nearly 20 mg/g, which is 40% less than for sorbents based on hydrolytic lignin. And this difference can be explained by the content of polysaccharide component. LC-materials, which contain 28% of cellulose, have a partial negative charge in alkaline solution as the protein, so electrostatic repulsion occurs. In this case, sorption of gelatine on sorbents takes place due to the presence of active functional groups of different nature (–NH₂, –COOH, –CONH₂, etc.) in the structure of the peptide chain. Such groups have affinity for oxygen-containing functional groups of lignin.

The investigation of sorption capacity of sorbents towards cadmium and lead ions that induce toxic effects on human body also was studied. Obtained results show that immobilization of modifiers on a LC-carrier leads to slight decrease of sorption capacity towards metal ions. However, in comparison with previous studies on beet pulp based sorbents (sorption capacity for Pb²⁺ and Cd²⁺ is 20.8 and 11.7 mg/g, respectively [19]), the LC-inorganic materials indicate high sorption properties towards metal ions.

Obtained sorbents can be used as materials of multifunctional purposes: as sorbents in radiochemistry and analytical chemistry for the recovery of caesium radionuclides, for effective removal of radionuclides from low-level radioactive solutions and water treatment; in medicine, as enterosorbents with sorption, radioprotective and antioxidant properties; in veterinary as a feed additive that will reduce the adsorption of radionuclides in the gastrointestinal tract of animals with the aim to obtain normative pure products (meat, milk, etc.).

Conclusions

The LC-inorganic sorbents based on lignocellulose modified with [FC]M and HAP were prepared. The study of caesium and strontium ions sorption on LC-[FC]M and

LC-HAP were performed. The efficiency of ^{137}Cs removal from the model solutions with the composite sorbents decreases as follows: LC-[FC]K-Ni>LC-[FC]K-Co>LC-[FC]Cu>LC-[FC]Fe>LC-[FC]Zn. Obtained modified materials are characterized by high sorption properties towards radionuclides.

The sorption of metal cations (Pb^{2+} and Cd^{2+}), dye (methylene blue), gelatine and vitamin B₁₂ from aqueous solutions on LC-inorganic sorbents were investigated. Sorption properties towards different toxicants slightly decrease after modifier immobilization in comparison with the LC-carrier.

Obtained results allow stating that the new composite LC-inorganic sorbents are universal materials for adsorption of radionuclides, heavy metals and organic pollutants from water solutions and can be used in radiochemistry, analytical chemistry, industry, medicine and veterinary.

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