

## УДАЛЕНИЕ ТЯЖЕЛЫХ МЕТАЛЛОВ ТЕКСТИЛЬНЫМИ ОТХОДАМИ НА ОСНОВЕ ЦЕЛЛЮЛОЗЫ

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*Текстильная промышленность является источником значительных количеств целлюлозных отходов, которые можно перерабатывать в перспективные сорбенты. Целью исследования было изучение адсорбционного поведения ионов металлов на целлюлозных поверхностях, полученных из целлюлозных текстильных отходов легкой промышленности. Проведенные ранее исследования показали, что немодифицированные ионообменные смолы на основе целлюлозы, полученные при различных значениях pH, способны удалять ионы тяжелых металлов из водных растворов. В представленной работе ионообменные смолы на основе целлюлозы получали гидролизом исходного отхода серной кислотой с последующей нейтрализацией до различных значений pH и сушкой при 80 °C. Средневзвешенный размер частиц используемых сорбентов был определен на основе фракционного состава порошкообразных сорбентов и составил 0,57 мкм (pH = 1,5 – 3) и 0,14 мкм (pH = 5 – 7). ИК-анализ элементного состава частиц выявил наличие связанных сульфатных групп в порошковой сорбенте с pH = 1,5-3. Результаты экспериментов показали, что модификация поверхности целлюлозного отхода серной кислотой повысила эффект адсорбции ионов Ni, Fe и Pb из водной среды. Сродство сульфокислотных смол к катионам обычно зависит от размера ионов и заряда катиона. Установлено, что значительную роль в адсорбционных свойствах полученных сорбентов играет электростатическое взаимодействие между поверхностными функциональными группами. Показано, что адсорбционная способность изучаемых металлов возрастает в ряду  $Fe^{3+} < Pb^{2+} < Ni^{2+}$ . Результаты исследований позволяют сделать вывод о целесообразности применения модифицированных отходов целлюлозных текстильных отходов легкой промышленности для очистки сточных вод от ионов металлов.*

**Ключевые слова:** текстильные целлюлозосодержащие отходы, катионообменная смола на основе сульфированной целлюлозы, тяжелые металлы, сточные воды

## HEAVY METAL REMOVAL BY CELLULOSE-BASED TEXTILE WASTE PRODUCT

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*The textile industry is a source of significant amounts of cellulosic waste that can be processed into promising sorbents. The aim of study was understanding the adsorption behavior of metal ions on cellulose surfaces obtained from cellulose textile waste of light industry. Previous studies have shown that unmodified cellulose-based ion exchange resins obtained at different pH values were able to remove metal ions from aqueous solution. In present work the cellulose-based ion exchange resins were prepared by H<sub>2</sub>SO<sub>4</sub> hydrolysis of initial waste product with following neutralization up to different pH and drying at 80 °C. Based on the fractional composition of powdered sorbents, the weighted average particle size of the sorbents used is determined: sorbents with pH = 1.5 - 3 ≈ 0.57 mm; sorbents with pH = 5 - 7 ≈ 0.14 mm. The IR analysis of the elemental composition of the particles showed the presence of bound sulfate groups in the powder sorbent with pH = 1.5-3. The results of experiments showed that the modification of the surface of the cellulose waste with sulfuric acid increased the effect of adsorption of Ni, Fe and Pb ions from an aqueous solution. It has been established that the electrostatic interaction between surface functional groups plays a significant role in the adsorption properties of the sorbents obtained. The affinity of sulphonic acid resins for cations generally varies with the ionic size and charge of the cation. This study showed that adsorption capacities of studied metals were in the order of Fe<sup>3+</sup> < Pb<sup>2+</sup> < Ni<sup>2+</sup>. Resulting cellulose particles have sulfate groups on their surface, which have wide range of applications for the removal of heavy metal ions from wastewater.*

**Key words:** textile cellulose-containing waste, sulphonated cellulose cation exchange resins, heavy metals, wastewater

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## INTRODUCTION

Despite the variety of sorbents used [1-3], many of them do not satisfy the entire set of requirements for materials of this type, in connection with which the search and development of new sorption materials is carried out constantly [4-6]. Priority is to obtain relatively cheap sorption materials based on industrial wastes, since waste is reused [7-10].

Cellulose is cheap, renewable, biodegradable, and the most abundant organic raw material in the world [11-15]. During recent years, removal of heavy metal ions, both from waste water and natural waters, has gained importance to solve and minimize the industrial and ecological waste problems [16-18]. Cellulose particles as adsorbents for water purification have been an area of great interest in materials science research in recent years [19]. Owing to the hierarchical structure and tailor able adsorption behavior via surface chemical modification with negative binding groups such as carboxylic, sulfate, and phosphate, cellulose has shown excellent potential to adsorb water pollutants such as dyes pesticides, bacteria and virus, and a wide range of heavy metal ions [20].

It is well known that cellulosic materials can be obtained from various natural sources and can be employed as cheap adsorbents [22]. Their adsorption capacities for heavy metal ions and other aquatic pollutants can be significantly affected upon chemical treatment [21]. In general, chemically modified cellulose exhibits higher adsorption capacities for various aquatic pollutants than their unmodified forms. Numerous chemicals have been used for cellulose modifications which include mineral and organic acids, bases, oxidizing agent, organic compounds, etc. [22].

Application of sulfuric acid results in negative surface charge formation due to incorporation of a number of sulfate groups at the surface of the cellulose nanocrystal. The number of sulfate groups will depend on the hydrolysis time and the sulfuric acid concentration [16]. From other hand, these groups can be removed by washing with dilute sodium hydroxide solutions, or by high temperature [17, 18].

It was shown [20] that the surface modification of cellulose microspheres by sulfate and phosphate functionalities improves the adsorption of metal ions from the aqueous medium.

This paper presents the results of the experiments studying sorption of  $\text{Fe}^{3+}$ ,  $\text{Ni}$  and  $\text{Pb}^{3+}$  ions from aqueous solutions by cellulose-based ion exchange resins, depending on the pH of the resin and duration of the process.

## MATERIALS AND METHODS

Model solution was prepared by dissolving the standard metal salts in distilled water. Concentrations of metal ions in the standard solution were 5 mg/L for each studied metals:  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ .

A quantitative analysis was carried out by atomic absorption spectroscopy (AAS) (spectrometer Spectr AA 200, Varian) in order to evaluate the accurate content of Me in model solutions.

pH measurements were made using PS-2 pH meter which was calibrated with pH 6.86 and pH 4.00 standard buffers before use.

Infrared spectroscopy (FTIR, Impact 410 Nicolet spectrophotometer) was used to characterize the obtained sorbents.

All quantitative analyses were run in triplicate for reproducibility of data and results in the figures and tables were the averaged ones.

The sorbents samples were analyzed using a scanning electron microscope (SEM, JSM-6380 LV).

Wastes product containing cotton fiber were used to produce powdered cellulose-based sorbent.

### PREPARATION OF SORBENT FROM TEXTILE CELLULOSE-CONTAINING WASTE

Wastes containing cotton fiber were used to produce powdered cellulose-based sorbent. In all the experiments, a flask containing cellulose waste and diluted  $\text{H}_2\text{SO}_4$  (30 % wt.) were mixed and placed in thermostatic bath at 70 °C and kept under thermostatic control for 2 h, with following neutral leaching and drying at 80 °C of the resulting pulp.

Neutral leaching tests were carried out at room temperature in a jacketed stirred reactor tank at different cotton waste/distillate water ratios, in order to reach desired pH of leach liquor, by mixing at 100 rpm for 1 h. After what suspensions were filtered and filter cakes were dried at 80 °C for 2 h and grounded up to finer-dispersed state. Depend on resulting leaching liquor pH following sorbents were obtained: KC1 (without neutral leaching step with initial pH = 1.5); KC2 (pH = 3); KC3 (pH = 5); KC4 (pH = 7).

The IR spectroscopy data are presented in Fig. 1. Based on the fractional composition of powdered sorbents, the weighted average particle size of the sorbents used is determined: sorbents with pH = 1.5-3  $\approx$  0.57 mm; sorbents with pH = 5-7  $\approx$  0.14 mm. In turn, the analysis of the elemental composition of the particles showed the presence of bound sulfate groups in the powder sorbent with pH = 1.5-3.

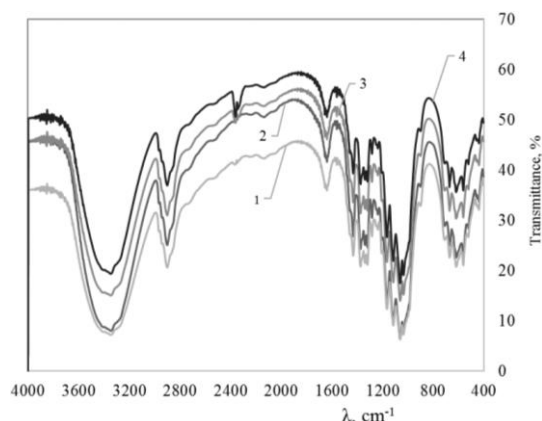


Fig. 1. IR spectra of studied cellulose-containing resins  
Рис. 1. ИК-спектры исследуемых ионообменных смол на основе целлюлозы: 1 – KC1; 2 – KC2; 3 – KC3; 4 – KC4

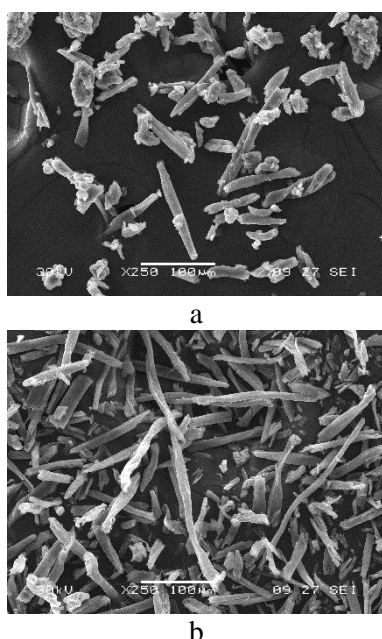


Fig. 2. SEM images of cellulose-based resins obtained at different pH: (a) pH = 1.5 – 3; (b) pH = 5 – 7  
Рис. 2. СЭМ-изображения исследуемых ионообменных смол на основе целлюлозы: (a) pH = 1,5 – 3; (b) pH = 5 – 7

#### ADSORPTION EXPERIMENT

Adsorption experiments were carried in a jacketed stirred reactor tank. The time and mixing speed were set with an automatic controller.

Kinetic study of the heavy metal adsorption was investigated for sorbents dosages 1% wt. The suspensions were mixed under stirring during to 10, 20, 30, 40, 50 and 60 min at 250 rpm. All experiments have been realized at 25 °C.

The suspensions have been constituted by dissolving 0.5 g of powder in 50 mL of standard solution. At the end of the mixing, the suspensions have been

filtrated. The residual concentration of Fe, Ni and Pb in the supernatant has been determined by atomic absorption spectroscopy with inductively coupled plasma emission spectrometer “Agilent Technologies 5100 ICP-OES”.

Removal percentage for each ion based on its initial concentration was calculated according to following equation:

$$\text{Removal percentage (\%)} = [(C_i - C_f) / C_i] \cdot 100\%$$

where  $C_i$  is the initial concentrations of  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$  ions in the solution (mg metal ion/L); and  $C_f$  is the concentrations of  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$  ions in the solution after adsorption (mg metal ion/L).

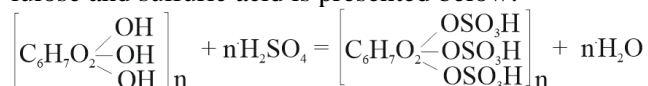
The sorption capacity of the resin were calculated according to equation below:

$$q_e = (C_i - C_b) \cdot V / m$$

where  $C_i$  and  $C_b$  are the initial and final concentrations of  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$  ions, respectively (mg/L);  $V$  is the volume of the solution, L;  $m$  is the sorbent mass, mg.

#### RESULTS AND DISCUSSION

Cellulose nanocrystals prepared by  $\text{H}_2\text{SO}_4$  hydrolysis have sulfate groups on their surface (Fig. 1). The process of sulfuric acid-hydrolysis of cellulose fibers for the preparation of cellulose nanocrystals includes an esterification reaction between acid and cellulose molecules, which induces the covalent coupling of sulfate groups on the surface of prepared sorbents [18]. Negatively charged sulfate groups play an important role in both surface chemistry and physical properties of obtained sorbents. Chemical reaction of cotton cellulose and sulfuric acid is presented below:



In this case, metal removal from model solution can be explained by following reactions:

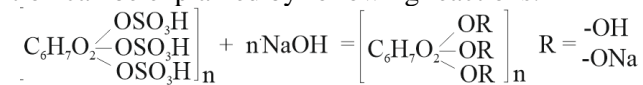


Fig. 3, shows the adsorption rate of Fe, Ni and Pb from model solution after adding studied adsorbents. Concentration of all studied metals reached a minimum after 15 min and then increased for all studied sorbents apart KC4 (pH = 7).

The affinity of sulphonic acid resins for cations generally varies with the ionic size and charge of the cation. Results of experiment show that Fe, Ni and Pb are rapidly adsorbed on the all sorbents. However, the adsorption capacities of studied metals were in the order of  $\text{Fe}^{3+} < \text{Pb}^{2+} < \text{Ni}^{2+}$ . It also was found that no desorption of studied metals occurred in case of sorbent with pH = 7.

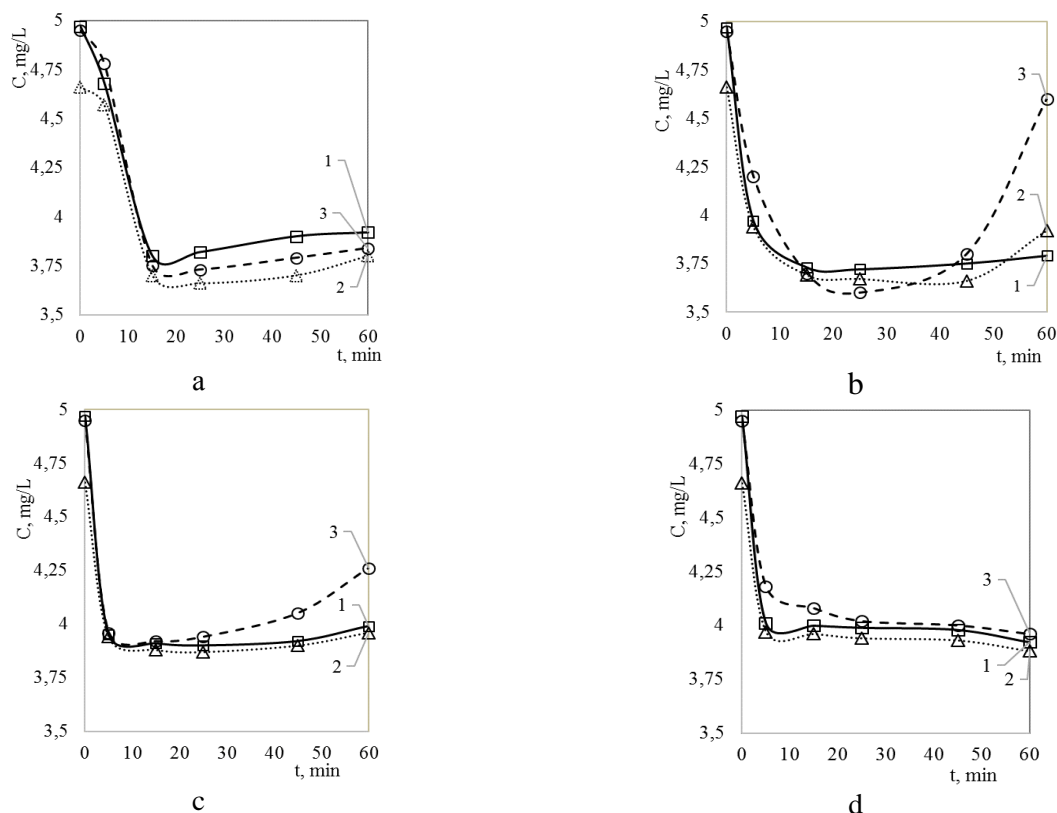


Fig. 3. The effect of process time on metal removing (1 – Ni; 2 – Fe; 3 – Pb) by studied cellulose-based resins prepared at different pH values: (a) – pH=1.5; (b) – pH=3; (c) – pH=5; (d) – pH=7

Рис. 3. Влияние продолжительности процесса на эффект удаления металлов (1 – Ni; 2 – Fe; 3 – Pb) исследуемыми ионообменными смолами на основе целлюлозы, полученными при разных pH: (a) – pH=1.5; (b) – pH=3; (c) – pH=5; (d) – pH=7

### CONCLUSIONS

Unmodified cellulose-based ion exchange resins obtained at different pH values were able to remove  $\text{Fe}^{3+}$ ,  $\text{Pb}^{2+}$ , and  $\text{Ni}^{2+}$  ions from aqueous solution serving as a model for a contaminated water medium.

The surface functionality of cellulose, ligand contents, depended on the pH of the resin, and electrostatic interaction played a significant role in the adsorption properties of the studied cellulose.

It was shown, that the surface modification of cellulose by sulfuric acid improved the adsorption of Ni, Fe and Pb ions from the aqueous medium.

It was also shown that KC4 sorbent provided maximum metal ion removal without any desorption of metal ions.

The modification of studied cellulose-based ion exchange resins will be the subject of further studies in our laboratories.

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