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SPECTROSCOPIC ANALYSIS OF A SORBENT BASED ON UREA, FORMALIN, AND SUCCINIC ACID AND ITS COMPLEXES WITH IONS OF Cu(II), Zn(II), Ni(II)

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Abstract: As a universal solvent, water plays an important role in the existence of all living organisms. Its continuous circulation through the hydrological cycle is essential for maintaining ecosystem balance. Wastewater is distinguished by its pollution and poses a great threat to the environment and all forms of life. Therefore, before discharging polluted water, it is necessary to properly treat it to mitigate its harmful effects. In this work, the sorption of Fe(III), Cu(II), Cd(II), Pb(II) metal ions from separate solutions and mixtures of their components was studied using sorbents obtained from rice husks for wastewater treatment. It is shown that the absorption capacity of sorbents depends on their properties.

In the article, the synthesis of sorbent and the Raman spectra of sorbent obtained on the basis of urea, formalin and succinic acid (KFC) with some 3d-metals were studied. The results of Raman spectroscopic analysis of the metallocomplex formed by KFC sorbent with Cu(II), Zn(II) and Ni(II) ions were analyzed. The obtained spectrum results are presented on the basis of Fig. Absorption frequencies of KFC sorbent and its metallocomplexes for each field (group) were analyzed and included in the table.

Keywords: sorbent, urea, formalin, succinic acid, asymmetric nas vibration, symmetric valence vibrations, deformation d vibrations KFC

Introduction. Targeted synthesis of complex-forming sorbents containing nitrogen, phosphorus, sulfur, study of their physico-chemical and analytical properties, nature of substituents in ligands and acid-base properties of complex compounds formed with metal ions. Research is being conducted on the development of sorption systems that allow the selective separation of metal ions from complex solutions by means of modeling the process of formation of metal-sorbent systems[1]. Water as a universal solvent plays an important role in the existence of all living organisms[2]. Its continuous circulation

through the hydrological cycle is essential for maintaining ecosystem balance. Wastewater is distinguished by its pollution and poses a great threat to the environment and all forms of life. Therefore, before discharging polluted water, it is necessary to properly treat it in order to mitigate its harmful effects [3]. In this article, the sorption of Cd^{2+} , Cu^{2+} , Fe^{2+} , Ni^{2+} , Zn^{2+} ions by polysaccharide biosorbent was studied. The effect of pH on the distribution of metal cations in the heterophase system of an aqueous solution of metal sulfate and a sorbent containing cellulose was determined [4]. The article describes the process of sorption of Cu (II), Ni (II), Fe (III), Cr (VI) ions from model aqueous solutions by production waste (knop) and knop after chemical modification with solutions of organic and mineral acids; sodium sulfide has been studied. [5] In this study, the sorption of Fe (III), Cu (II), Cd (II), Pb (II) metal ions from separate solutions and mixtures of their components was studied using rice husk sorbents. It has been shown that the absorption capacity of sorbents depends on their properties.[6] In this work, the adsorption of metal ions from aqueous solutions by wastes of the woodworking industry was studied under static conditions. The effect of particle size of aspen sawdust on the level of adsorption of Zn^{2+} and Fe^{2+} , Fe^{3+} ions was determined. Adsorption isotherms were obtained and adsorption parameters were calculated using Freundlich and Langmuir equations.[7] The article presents the sorption isotherms of copper (II) ion in a sorbent based on urea, formaldehyde and ethylenediaminetetraacetic acid (EDTA) according to the Langmuir and Freundlich models. [8]. In this research work, the removal of cations from an aqueous solution of wood processing waste-juniper bark sorbent was investigated. It was determined that the dynamic exchange capacity of these sorbents reached 22.52 mg/g at an initial concentration of 52 mg/dm in solution [9][10]. The process of sorption of heavy metals such as Sr, Ni, Zn from surface waters by the natural zeolite (KLT) of the Kholinskoe mine and the organozeolite mineral obtained on its basis was studied. According to the obtained results, internal diffusion coefficients and sorption levels of Sr, Zn, Ni, and Ca are good in pH 6, 7, and 8 [11,12]. Sorption mechanisms with unmodified and modified sorbents have been proposed, including chemisorption, electrochemical, and various variants of donor-acceptor interactions [13,14,15]. Compounds based on N-2-sulfoethyl chitosan of copper (II) and silver (I) ions in the solution were used as sorbents. The sorption selectivity of sorbents based on N-2-sulfoethylchitosan has been shown to increase compared to copper(II) [16,17]. In a number of research works presented below, methods of obtaining sorbents based on various compounds are presented: Dithizone, calmagit[18], ammonium salts of dithiophosphoric acid O,O-diethyl ether[19], 8-hydroxyquinoline[20], 1-(2-pyridylazo) Several complicating agents have been proposed.

Methodology. Synthesis of KFC sorbent. 1.2 g (0.02 mol) of urea was dissolved in 4 ml (0.05 mol) of formalin in a three-necked flask equipped with a reflux condenser and an automatic stirrer for the synthesis of ionite, which forms a complex containing nitrogen and oxygen, and pH=8- Ammonium hydroxide solution was added until 9. The temperature was heated at 70-80°C until a viscous mass was formed. 1.18 g (0.01 mol) of succinic acid solution in 5 ml of ammonium hydroxide was added dropwise to the

resulting viscous mixture and mixed. When the temperature increased to 110-130°C, a solid or gummy mass was formed. The resulting resinous mass was placed in a porcelain bowl and dried in a drying cabinet at a temperature of 100°C for 20 hours. After the dried polymer was crushed, the low molecular weight substances were washed first with 5% sodium alkali solution and then several times with distilled water until it became neutral. As a result, a white granular mass consisting of small pores was formed. Product yield was 90%.

Results.

In the Raman spectrum (HORIBA Scientific) of the sorbent based on urea, formalin and succinic acid (KFC) in the 3128.42 cm⁻¹ and 1176.01 cm⁻¹ regions, valence symmetric ν_s and deformation δ vibrations of the n(NH₂) group and 1116.43 cm⁻¹ In area, the valence ν vibration frequency of the n(C-N-) group was observed. Together with this, the frequency of valence asymmetric ν_{as} , valence symmetric ν_s and deformation δ vibrations of the ν (CH₂) group was formed in the fields of 2945.46 cm⁻¹, 2828.42 cm⁻¹ and 745.46 cm⁻¹. In the regions of 1725.93 cm⁻¹ and 1519.24 cm⁻¹, the valence asymmetric vibration of the ν (C=O) and ν (COO-) group was formed, as well as the deformation δ vibration frequency in the region of 828.42 cm⁻¹. Also, the valence ν vibration frequencies of the ν (COOC) group were observed in the region of 1282.58 cm⁻¹. The obtained spectrum results are presented in picture-1.

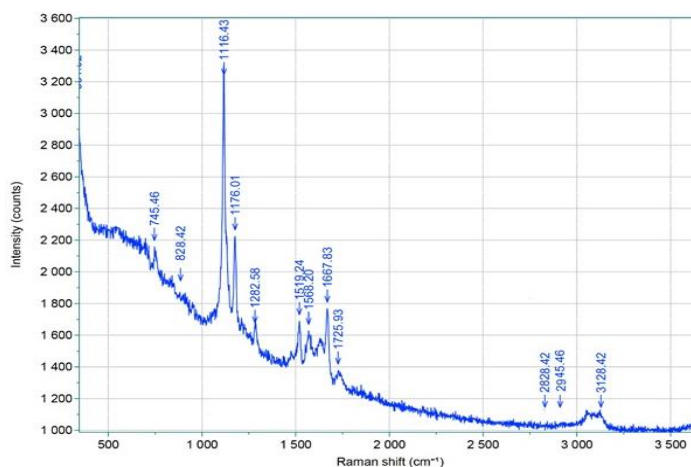


Figure 1. Raman spectrum of KFC sorbent.

The Raman spectrum (HORIBA Scientific) obtained as a result of Cu(II) sorption of KFC sorbent is presented in picture-2, according to the results of the IR spectrum, the valence symmetric ν_s of the ν (NH₂) group at 3195.93 cm⁻¹ and 1188.11 cm⁻¹ deformation δ vibrations and the valence δ vibration frequency of the ν (C-N-) group were observed in the region of 1116.43 cm⁻¹. Together with this, the frequency of valence asymmetric ν_{as} , valence symmetric ν_s and deformation δ vibrations of the ν (CH₂) group was formed in the fields of 2985.31 cm⁻¹, 2895.2998 cm⁻¹ and 785.82 cm⁻¹. In the regions of 1765.11 cm⁻¹ and 1511.24 cm⁻¹, the valence asymmetric vibration of the ν (C=O) and ν (COO-) group was formed, as well as the deformation δ vibration frequency in the region of 825.93 cm⁻¹.

¹. Also, the valence ν vibrational frequencies of the $\nu(\text{COOC})$ group were observed in the region of 1231.58 cm^{-1} . The spectrum results are presented in picture-3.2

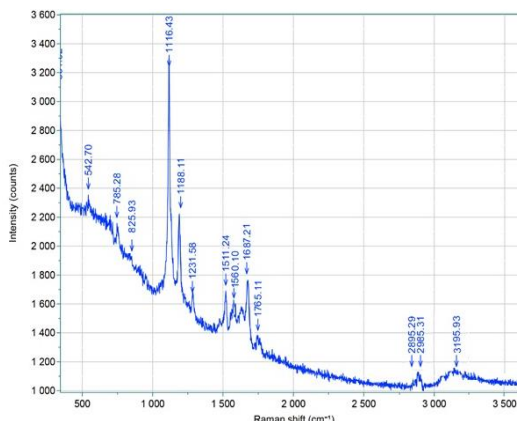


Figure 2. Raman spectrum of the complex formed by Cu(II) with KFC sorbent.

The complex Raman spectrum (HORIBA Scientific) obtained as a result of the sorption of Zn(II) of KFC sorbent is presented in picture-3. According to the results of the IR spectrum, the $\nu(\text{NH}_2)$ group is valence symmetric in the areas of 3268.94 cm^{-1} and 1192.50 cm^{-1} ν_s and deformation δ vibrations and the valence ν vibration frequency of the $\nu(\text{C-N-})$ group were observed in the region of 1182.82 cm^{-1} . Together with this, the frequency of valence asymmetric ν_{as} , valence symmetric ν_s and deformation δ vibrations of the $\nu(\text{CH}_2)$ group was formed in the fields of 2935.29 cm^{-1} , 2865.33 cm^{-1} and 765.82 cm^{-1} . In the 1745.94 cm^{-1} and 1516.24 cm^{-1} regions, the valence asymmetric vibration of the $\nu(\text{C=O})$ and $\nu(\text{COO-})$ group was formed, as well as the deformation δ vibration frequency in the 835.29 cm^{-1} region. Also, the valence ν vibrational frequencies of the $\nu(\text{COOC})$ group were observed in the region of 1233.58 cm^{-1} . The complex Raman spectrum (HORIBA Scientific) obtained as a result of Ni(II) sorption of KFC sorbent is shown in picture-4.

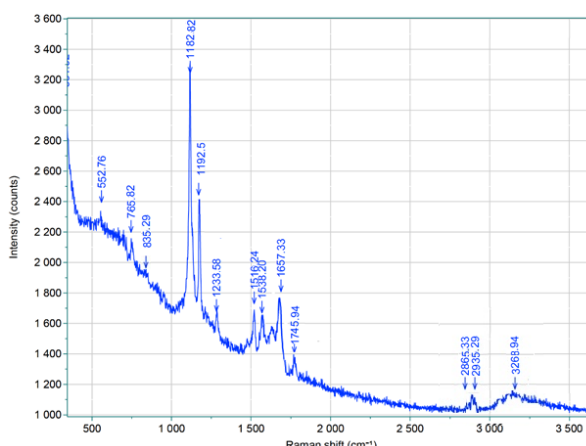


Figure 3. The complex formed by Zn(II) with KFC sorbent

According to the results of the IR spectrum, the $\nu(\text{NH}_2)$ group is valence symmetric in the fields of 3461.88 cm^{-1} and 1196.91 cm^{-1} ν_s and deformation δ vibrations and the

valence ν vibration frequency of the $\nu(\text{C-N-})$ group were observed in the region of 1146.41 cm^{-1} . Together with this, the frequency of valence asymmetric ν_{as} , valence symmetric ν_s and deformation δ vibrations of the $\nu(\text{CH}_2)$ group was formed in the fields of 2967.81 cm^{-1} , 2861.18 cm^{-1} and 735.55 cm^{-1} .

In the 1738.83 cm^{-1} and 1533.21 cm^{-1} regions, the valence asymmetric vibration of the $\nu(\text{C=O})$ and $\nu(\text{COO-})$ group was formed, as well as the deformation δ vibration frequency in the 861.87 cm^{-1} region. Also, the valence ν vibrational frequencies of the $\nu(\text{COOC})$ group were observed in the region of 1283.64 cm^{-1} .

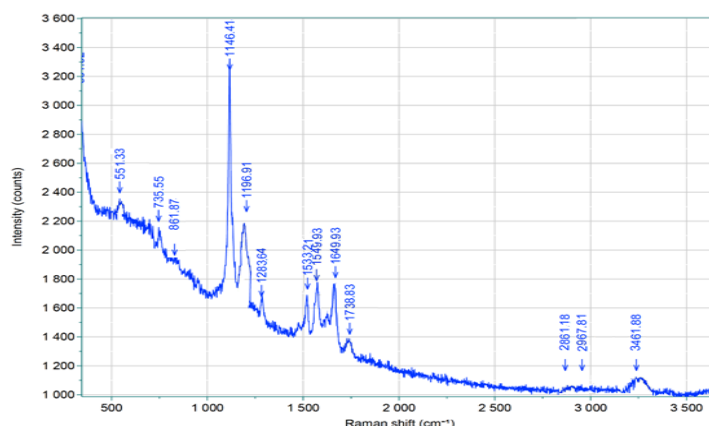


Figure 4. Raman spectrum of Ni(II) complex formed with KFC sorbent.

Table 1

Absorption frequencies in Raman spectra of KFC sorbent and its coordination compounds with Cu(II), Zn(II), Ni(II) ions, cm^{-1}

Vibration classifications	KFC	KFC+Cu(II)	KFC+Zn(II)	KFC+Ni(II)
$\nu(\text{NH})$	3128,42	3195,93	3268,94	3461,88
$\nu_s(\text{CH}_2)$	2945,46	2985,31	2935,29	2967,81
$\nu_{as}(\text{CH}_2)$	2828,42	2895,29	2865,33	2861,18
$\nu(\text{C=O})$	1725,93	1765,11	1745,94	1738,83
$\nu(-\text{COC=O})$	1282,58	1231,58	1233,58	1283,64
$\nu_{as}(-\text{COO-})$	1519,24	1511,24	1516,24	1533,21
$\nu(\text{C-N})$	1116,43	1116,43	1182,82	1146,41
$\delta(\text{NH})$	1176,01	1188,11	1192,50	1196,91
$\delta(\text{CH}_2)$	745,46	785,28	765,82	735,55
$\delta(-\text{COO-})$	828,42	825,93	835,29	861,87

cm^{-1}

As can be seen from table-11, the $\nu(\text{NH})$ vibration frequency and $\delta(\text{C=O})$ vibration frequency in KFC sorbent are relatively shifted to other areas. It can be concluded as follows that the secondary amine and carbonyl groups in the KFC sorbent ensure the coordination of the metal ion and a chelate is formed.

Discussion. Certain scientific and practical results have been achieved in our republic in the production of chemical industry products, in particular, in the selective extraction of non-ferrous and rare metals from the composition of solutions, and in the production of sorbents used for the purification of wastewater from heavy metals. In this article presented by the authors, the problem of pollution and self-cleaning of water

bodies from heavy metal compounds is considered. The process of sorption of heavy metal compounds (Cu (II), Zn (II), Pb (II), Cd (II)) by bottom sediments of different composition was studied. Equilibrium constants and rates of sorption of heavy metals by bottom sediments were obtained during laboratory studies. It was determined that the obtained data are necessary for predicting water quality as a result of pollution of a water body with industrial wastewater.

Conclusion. The article contains a brief annotation of the work, key words. In the introduction, the relevance of the topic and the work of scientists on the topic are analyzed. The absorption spectra of the sorbent based on urea, formalin and succinic acid (KFC) and its complexes with some 3d metals were studied by Raman spectroscopy (HORIBA Scientific). Raman spectroscopic analysis of metal complexes formed by Cu(II), Zn(II) and Ni(II) ions with KFC sorbent was performed. From the results, it can be concluded that the secondary amine and carbonyl groups in KFC sorbent are coordinated with the metal ion and a chelate is formed.

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C O N T E N T S

PRIMARY PROCESSING OF COTTON, TEXTILE AND LIGHT INDUSTRY

Nabidjanova N., Azimova S.	
Study of physical-mechanical properties of fabrics used for men's outer knit assortment	3
Nabidjanova N., Azimova S.	
Development of model lines of men's top knitting assortment	7
Noorullah S., Juraeva G., Inamova M., Ortiqova K., Mirzaakbarov A.	
Enhancing cotton ginning processing method for better fibre quality	12
Kamalova I., Inoyatova M., Rustamova S., Madaliyeva M.	
Creating a patterned decorative landscape using knitted shear waste on the surface of the paint product	16
Inoyatova M., Ergasheva Sh., Kamalova I., Toshpo'latov M.	
State of development of fiber products – cleaning, combing techniques and technologies	21
Vakhobova N., Nigmatova F., Kozhabergenova K.	
Study of clothing requirements for children with cerebral palsy	30
Mukhametshina E., Muradov M.	
Analysis of the improvement of pneumatic outlets in the pneumatic transport system	37
Otamirzayev A.	
Innovative solutions for dust control in cotton gining enterprises	45
Muradov M., Khuramova Kh.	
Studying the types and their composition of pollutant mixtures containing cotton seeds	50
Mukhamedjanova S.	
Modernized sewing machine bobbin cap hook thread tension regulator	53
Ruzmetov R., Kuliyeu T., Tuychiev T.	
Study of effect of drying agent component on cleaning efficiency.	57
Kuldashov G., Nabiev D.	
Optoelectronic devices for information transmission over short distances	65
Kuliev T., Abbasov I., F.Egamberdiev.	
Improving the elastic mass of fiber on the surface of the saw cylinder in fiber cleaning equipment using an additional device	73
Yusupov A., Muminov M., Iskandarova N., Shin I.	

On the influence of the wear resistance of grate bars on the technological gap between them in fiber separating machines **80**

Kuliev T., Jumabaev G., Jumaniyazov Q.

Theoretical study of fiber behavior in a new structured elongation pair **86**

GROWING, STORAGE, PROCESSING AND AGRICULTURAL PRODUCTS AND FOOD TECHNOLOGIES

Meliboyev M., Ergashev O., Qurbonov U.

Technology of freeze-drying of raw meat **96**

Davlyatov A., Khudaiberdiev A., Khamdamov A.

Physical-chemical indicators of plum oil obtained by the pressing method **102**

Tojibaev M., Khudaiberdiev A.

Development of an energy-saving technological system to improve the heat treatment stage of milk **109**

Turg'unov Sh., Mallabayev O.

Development of technology for the production of functional-oriented bread products **115**

Voqqosov Z., Khodzhiev M.

Description of proteins and poisons contained in flour produced from wheat grain produced in our republic **120**

CHEMICAL TECHNOLOGIES

Choriev I., Turaev Kh., Normurodov B.

Determination of the inhibitory efficiency of the inhibitor synthesized based on maleic anhydride by the electrochemical method **126**

Muqumova G., Turayev X., Mo'minova Sh., Kasimov Sh., Karimova N.

Spectroscopic analysis of a sorbent based on urea, formalin, and succinic acid and its complexes with ions of Cu(II), Zn(II), Ni(II) **131**

Babakhanova Kh., Abdukhalilova M.

Analysis of the composition of the fountain solution for offset printing **138**

Babakhanova Kh., Ravshanov S., Saodatov A., Saidova D.

Development of the polygraphic industry in the conditions of independence **144**

Tursunqulov J., Kutlimurotova N., Jalilov F., Rahimov S.

Determination zirconium with the solution of 1-(2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfate **151**

Allamurtova A., Tanatarov O., Sharipova A., Abdikamalova A., Kuldasheva Sh.

Synthesis of acrylamide copolymers with improved viscosity characteristics **156**

Amanova N., Turaev Kh., Alikulov R., Khaitov B., Eshdavlatov E., Makhmudova Y.	
Research physical and mechanical properties and durability of sulfur concrete	165

MECHANICS AND ENGINEERING

Abdullaev E., Zakirov V.	
Using parallel service techniques to control system load	170
Djuraev R., Kayumov U., Pardaeva Sh.	
Improving the design of water spray nozzles in cooling towers	178
Anvarjanov A., Kozokov S., Muradov R.	
Analysis of research on changing the surface of the grid in a device for cleaning cotton from fine impurities	185
Mahmudjonov M.	
Mathematical algorithm for predicting the calibration interval and metrological accuracy of gas analyzers based on international recommendations ILAC-G24:2022/OIML D 10:2022 (E)	192
Kulmuradov D.	
Evaluation of the technical condition of the engine using the analysis of the composition of gases used in internal combustion engines	197
Kiryigitov Kh., Taylakov A.	
Production wastewater treatment technologies (On the example of Ultramarine pigment production enterprise).	203
Abdullayev R.	
Improving the quality of gining on products.	208
Abdullayev R.	
Problems and solutions to the quality of the gining process in Uzbekistan.	212
Yusupov D., Avazov B.	
Influence of various mechanical impurities in transformer oils on electric and magnetic fields	216
Kharamonov M.	
Prospects for improving product quality in textile industry enterprises based on quality policy systems	223
Kharamonov M., Kosimov A.	
Problems and solutions to the quality of the gining process in Uzbekistan.	230
Mamahonov A., Abdusattarov B.	
Development of simple experimental methods for determining the coefficient of sliding and rolling friction.	237

Aliyev E., Mamahonov A.	
Development of a new rotary feeder design and based flow parameters for a seed feeder device	249
Ibrokhimova D., Akhmedov K., Mirzaumidov A.	
Theoretical analysis of the separation of fine dirt from cotton.	260
Razikov R., Abdazimov Sh., Saidov D., Amirov M.	
Causes of floods and floods and their railway and economy influence on construction.	266
Djurayev A., Nizomov T.	
Analysis of dependence on the parameters of the angles and loadings of the conveyor shaft and the drum set with a curved pile after cleaning cotton from small impurities	272
ADVANCED PEDAGOGICAL TECHNOLOGIES IN EDUCATION	
Jabbarov S.	
Introduction interdisciplinary nature to higher education institutions.	276
Tuychibaev H.	
Analysis of use of sorting algorithms in data processing.	280
Kuziev A.	
Methodology for the development of a low cargo network.	289
Niyozova O., Turayev Kh., Jumayeva Z.	
Analysis of atmospheric air of Surkhondaryo region using physico-chemical methods.	298
Isokova A.	
Analysis of methods and algorithms of creation of multimedia electronic textbooks.	307
ECONOMICAL SCIENCES	
Rashidov R., Mirjalolova M.	
Regulations of the regional development of small business.	315
Israilov R.	
Mechanism for assessment of factors affecting the development of small business subjects.	325
Yuldasheva N.	
Prospects of transition to green economy.	334
Malikova G.	
Analysis of defects and solutions in investment activity in commercial banks.	346