



Research of morphology and optical properties of complex-forming the ionit and their complex compounds of certain metals

Eshkurbonov Furkat Bozorovich ¹, Rakhmonkulov Jasur Eshmuminovich ^{2*},
Ulugov Bazar Dzhumaevich ²

¹ Termez State University, Tashkent, UZBEKISTAN

² Termez Branch of Tashkent State Technical University named after Islam Karimov, Tashkent, UZBEKISTAN

*Corresponding author: rakhmonkulovjasur@gmail.com

Abstract

The structure and properties of the obtained cross-linked ion exchangers were studied by scanning electron microscopy and IR spectroscopy. The results of the EDAX device showed that in ion exchangers, the elemental composition is represented mainly by carbon, oxygen, nitrogen, sulfur, chlorine, and sodium. Absorption in the 1400–1405 cm⁻¹ region, caused by the stretching vibrations of C = S and C = O groups, confirms the presence of thiol and oxo groups in the structure of the ion exchangers. The results of studying morphology and optical properties of complexing ion-exchangers based on diglycidyl thiocarbamide (diglycidyl thiocarbamide, the reaction products of epichlorohydrin and thiourea) and cobalt complex are presented. It has been found that with an increase in the concentration of the cobalt complex in the ion exchangers, the morphology changes, that is, the structural elements change on the surface of the samples. It has been shown that at high Co²⁺ contents, the structural elements of the compositions are refined.

Keywords: complexation, microstructure, composition, SEM, IR spectroscopy, ability, EDAX device results, curves

Eshkurbonov FB, Rakhmonkulov JE, Ulugov BD (2020) Research of morphology and optical properties of complex-forming the ionit and their complex compounds of certain metals. Eurasia J Biosci 14: 6045-6050.

© 2020 Eshkurbonov et al.

This is an open-access article distributed under the terms of the Creative Commons Attribution License.

INTRODUCTION

A new level of development of aviation, hydrometallurgy, and astronautics is associated with the creation of fundamentally new materials, in the first row of which are polymer composite materials (PCM). These materials provide high specific strength, operational reliability, and durability (Kattaev N.T., and Ramazonov A.Kh., 2016), which is very important not only for use in aerospace engineering but also in other industries (hydrometallurgy, energy, demineralized and purified waters used in various industries national economy). To assess the stability of the operating characteristics and changes in the microstructure of the PCM under force, it is necessary to carry out physical and chemical studies using methods of IR spectroscopy and scanning electron microscopy, micro- and nanoindentation at various structural levels (Litvinov V.B. et al., 2011). The world production of synthetic ion exchangers has doubled over the past decade. The countries of the independent states (CIS) rank second in the world in terms of the number of ion exchangers produced, and 65% of these ion exchangers are used for the enrichment and separation of metals in the

hydrometallurgical industry, in purified and purified water used in various sectors of the economy (Eshkurbonov F.B., 2013). The produced ion exchangers are also used in the treatment of thermal power plants, nuclear power plants and industrial waste 15%, in chemical technology 9%, in the food and pharmaceutical industries 6% and other industries¹. Ion exchangers make it possible to effectively separate metals by selective sorption from individual and mixed alloys in various media (Kablov E.N., 2012). For this reason, a large number of studies are focused on the synthesis of new ions, the study of their physicochemical properties, and the development of technologies for their use in sorption processes. Since the Republic of Uzbekistan gained independence (Ulugov B.D. et al., 2020, a, b, c), the organization of a high level of scientific research on the development of new types of products in the chemical industry and the provision of the local market with a wide range of chemical reagents that can replace imported ones. In this regard, the ions synthesized based on local raw

Received: February 2020

Accepted: April 2020

Printed: December 2020

materials containing donor atoms of sulfur, nitrogen, oxygen, and phosphorus effectively sorb from the alloys ions of noble metals, thermally and chemically stable, mechanically stable, and complex high-tech ions (Dzhalilov A.T. et al., 2013). The synthesis of new types of ion exchangers, determination of the sorption properties of metal ions, and the development of a technology for the extraction of noble metal ions from alloys are important in the world today (B.D. Ulugov, 2020). When studying the formation of complexing ion exchangers, it is difficult to form a complex of ions with ions of different metals in a solution containing donor atoms of sulfur, nitrogen, oxygen, and phosphorus, based on local raw materials. and the development of separation methods is an urgent problem (Deev I.S. et al., 2011).

Degree of Knowledge of the Problem

On the study of ionic synthesis and their technology. Pashkov, I. Losev, E. Trostyanskaya, A. Tevlina, A. Davankov, V. Laufer, F. Shostak, I.F. Samborsky, E. Ergodzhin, K. Saldadze, I. Khmel'nitskaya, S.A. Marandzhev, E. Lustgarten, V. Kopylova, A.A. Weisheidt, A.A. Vasiliev, N. Kuznetsova, R. Kusi, G. Martinez, I.S. Ahamed, S. Aoki, F.A. Long, M.A. Askarov, A. Jalilov, X.T. Sharipov, U. Musaev, S. Zainutdinov, Z.A. Tajikhojaev, X.X. Turaev, and others conducted research. The main scientific directions for the development of ion-exchange synthesis technology are based on the synthesis of ion-exchangers based on thiourea, urea thiosemicarbazide, thiophosphates, melamine, gossypol resin, formalin, epichlorohydrin, and polyacrylonitrile fibers, which can effectively separate metal ions from solutions (Eshkurbanov F.B., and Dzhalilov A.T., 2014; Turaev B.E. et al. 2020). Besides, scientific research has been carried out on the synthesis of new complexing ion exchangers by introducing monomers of new functional groups into existing ion exchangers (Turaev Kh. Kh. et al., 2013). However, the main direction in the synthesis of effective ion exchangers for precious metals is the synthesis of ion-exchangers based on monomers containing nitrogen, sulfur, oxygen, and phosphorus, using local substitutes for raw materials, their composition, structure, properties, extraction technologies, and various materials. aimed at studying the effective separation of metal ions (Khurram Sh. et al., 2020).

MATERIALS AND METHODS

Various physicochemical methods are used to study the structure and chemistry of the surface as well as to study the theoretical and applied issues of sorption on complexing ion exchangers (Eshkurbanov F.B., 2013). The analysis of the physicochemical characteristics of the ion exchangers DHT + GIPAN and DMT + GIPAN (Dzhalilov A.T., and Eshkurbanov F.B., 2014) is characterized by data using IRS and SEM.

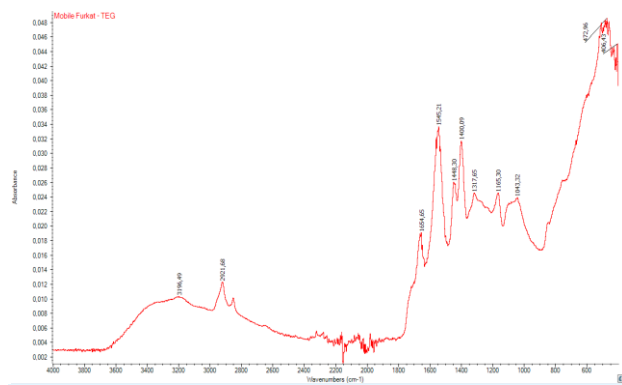


Fig. 1. IR spectrum of the DHT – GIPAN ion exchanger

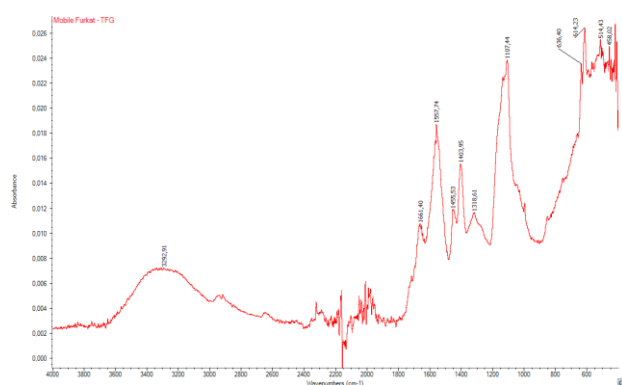


Fig. 2. IR spectrum of the ion exchanger DMT – GIPAN

RESULTS AND DISCUSSION

The IRS method allows one to obtain direct information on the change during sorption of the state of the surface groups of ion exchangers and chemical bonds in sorbed molecules; therefore, it is widely used to study the mechanism of sorption. IR spectra of complexing ion exchangers are shown in **Figs. 1** and **2**.

As seen in **Figs. 1** and **2**, the IR spectra of the ion exchangers DHT + HIPAN and DMT + HIPAN are very similar, which is probably due to the similar chemical structure. Their spectra lack the characteristic frequencies of epoxy groups (510-640, 1100-1170 and 3100-3300 cm^{-1}) and bands of N-H bending vibrations (1540-1665 cm^{-1}) and C-N stretching vibrations (1020-1200 cm^{-1}) appear bonds of amino groups, which indicates the chemical interaction of DHT and DMT with HIPAN. Absorption in the range 1400–1405 cm^{-1} , caused by the stretching vibrations of C = S and C = O groups, confirms the presence of thiol and oxo groups in the structure of ion exchangers (Turaev Kh. et al., 2013). The surface morphology of the ion exchangers was carried out by SEM to see the distinguishable changes in their structures. The EDAX device shows the elemental composition of the samples. SEM photographs of the ion exchangers are shown in **Fig. 3**, **4**, **5** and **6**. **Fig. 3** shows a photograph of the microstructure of the surface of the ion exchangers at a

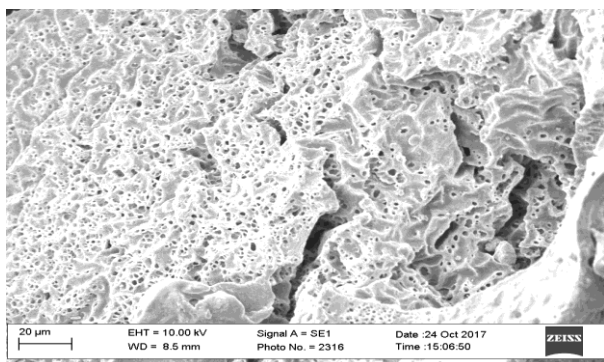


Fig. 3. The microstructure of DHT + HIPAN

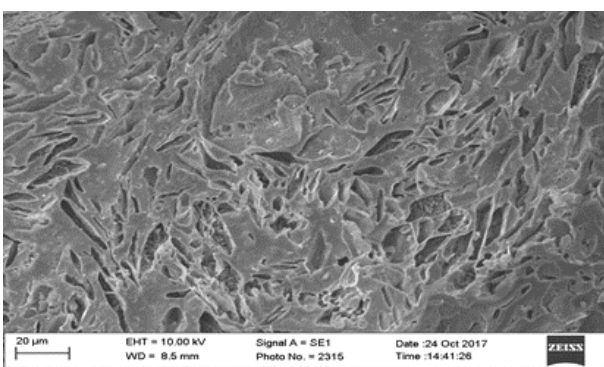


Fig. 4. The microstructure of DMT + HIPAN

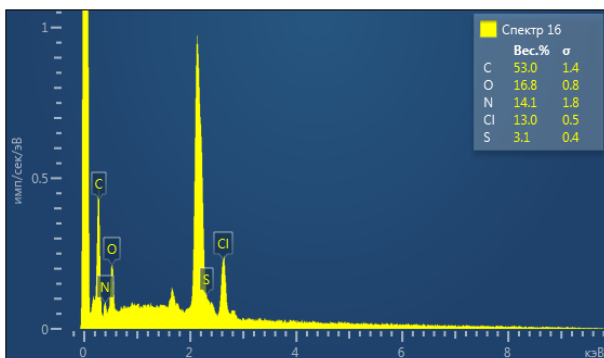


Fig. 5. Chemical elemental composition of DHT + HIPAN

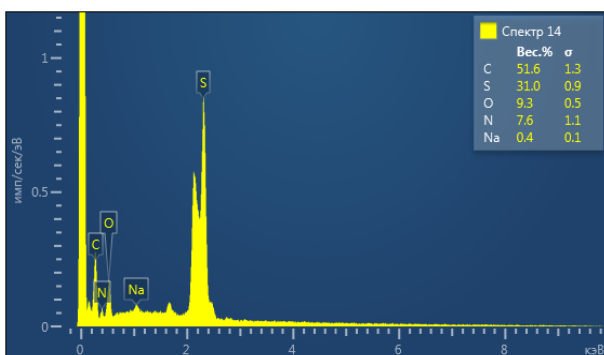
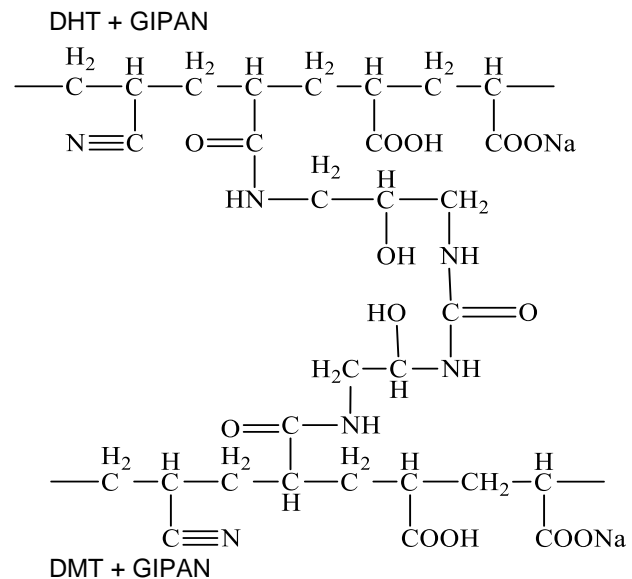
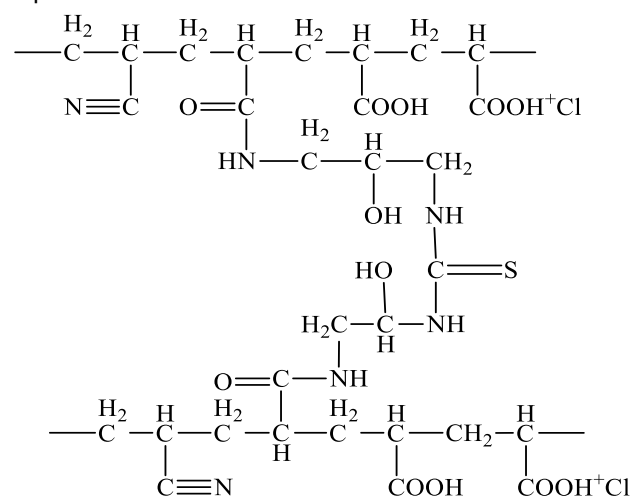


Fig. 6. Chemical elemental composition of DMT + HIPAN

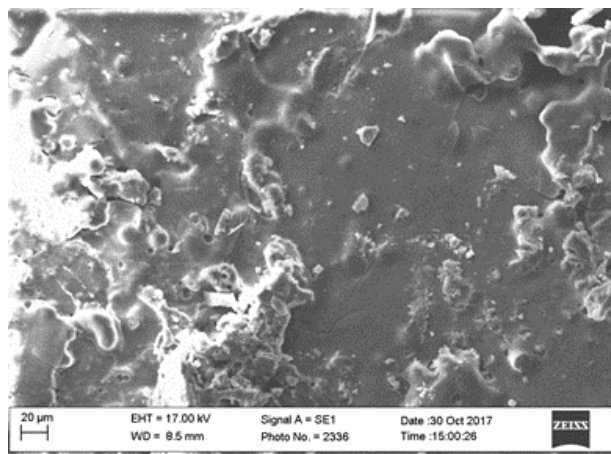
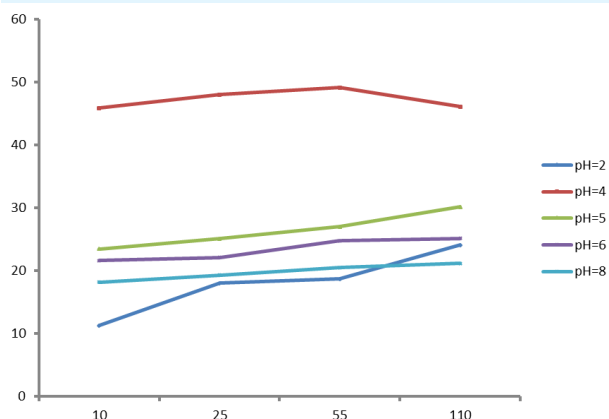
magnification of 500. The image shows that the surface has a heterogeneous structure, the ion exchanger has a helium structure, in which spherical, slit-like pores are traced and there are larger and smaller particles. The

results of the EDAX device showed that in ion exchangers, the elemental composition is represented mainly by carbon, oxygen, nitrogen, sulfur, chlorine, and sodium. Their content in weight percent: DHT + HIPAN C - 53.0%, O - 16.8%, N - 14.1%, Cl - 13.0%, S - 3.1%. DMT + HIPAN C - 51.6%, S - 31.0%, O - 9.3%, N - 7.6%, Na - 0.4%.

Based on chemical and spectral analyses, the structure of the synthesized polymers can be represented as follows:



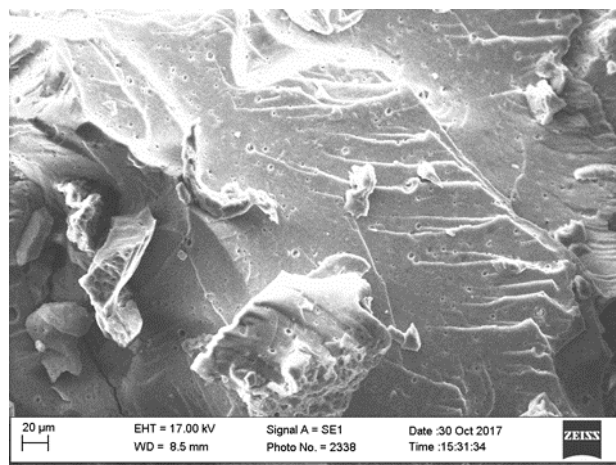
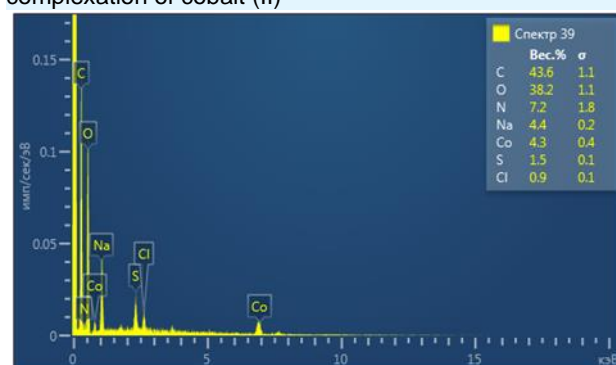
When DHT and DMT interact with HIPAN, sequential nucleophilic addition of amino groups to epoxy groups occurs. With an increase in the degree of conversion, the rate of the polycondensation reaction significantly decreases due to a decrease in the mobility and availability of functional groups in the process of formation of products of the spatial structure. As a result, macro reticular polyfunctional ion exchangers DHT + GIPAN and DMT + GIPAN were obtained. The process of complexation of cobalt ions depending on pH and time is shown in **Table 1**. The curves of dependence in the

Table 1. Influence of pH and time on the process of complexation of cobalt (II) ions with ion exchanger DHT + HIPAN**Fig. 7.** The microstructure of DHT + HIPAN after complexation of cobalt (II)

process of complexation of cobalt on pH, as can be seen from the figure, for all pH values are of the same type.

In the acidic range of pH 2, the degree of complexation was insignificantly high for cobalt ions, so for 10 min of the process, it was 11.3%, at 25 - 55 min 18.7%, and with an increase in time to 110 min, it slightly increased to 24.08%.

The greatest value of the cobalt complexation process was observed at pH 4. However, the complexation process was not very great; within 10 minutes the process is only 45.91%, then with an increase in time to 50 min, the largest cobalt complexation process is observed, reaches 49.12%. Further, as the time increased to 110 min, the cobalt complexation process decreased to 46.10%. A complex equilibrium of cobalt complexation was achieved within 55 min. Further, with an increase in pH to 5, a decrease in the process of cobalt complexation is observed, and within 10 minutes it is 23.5%, then at 55 minutes - 27.0%, and at 110 minutes it is 30.14%. With an increase in pH 6 and 8, the complexation process decreases.

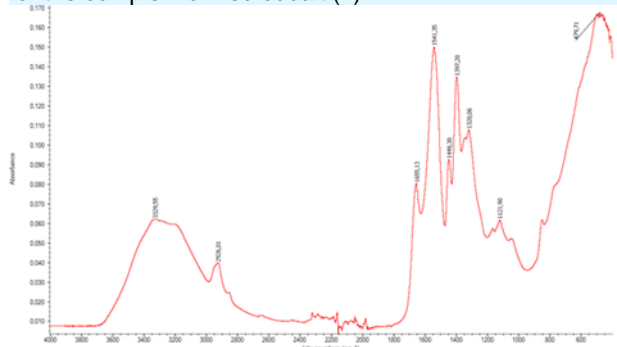
**Fig. 8.** The microstructure of DHT + HIPAN after complexation of cobalt (II)**Fig. 9.** Chemical elemental composition of samples obtained at a rate of 1.5 g of the ion exchanger and a time of 120 min at different concentrations of cobalt (II), 0.05 g/l

When using pH 6 for 10 minutes, the complexation of cobalt is 21.6%, and when the process is increased to 110 minutes it is 25.14%, a slight increase is observed in the process of complexation with increasing time. In an alkaline medium at pH 8, a decrease in the value of complexation is observed, for 10 minutes of the process 18.2%, and at 110 minutes - 21.7%. This is because cobalt ions weakly interact with the carboxyl groups of HIPAN in an alkaline medium. **Fig. 7-9.**

Shows electron micrographs of the sample surface. As can be seen from the SEM images, the surface (at x250 magnification) consists of large and small particles, between which there are pores.

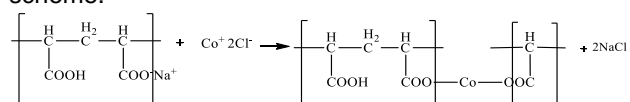
Light particles of cobalt predominate since they are noticeably large and densely complexed in the pores of the DHT + HIPAN ion exchanger. EDAX elemental results show cobalt contents of 4.3 and 0.2 wt. %. To clarify the mechanism of absorption of cobalt (II) ions by DHT + HIPAN at various concentrations of cobalt (II), the IR spectra of the samples were recorded (**Table 2**).

The spectra of all samples show intense absorption bands in the region of 3329, 2926 cm^{-1} of hydroxyl groups forming hydrogen bonds. The absorption bands of carboxyl groups corresponding to the stretching vibrations of C = O and C = S are present in the region

Table 2. IR spectrum of the ion exchanger DHT + HIPAN for the complex formed cobalt (II)

of 1655, 1541, 1448 cm^{-1} . This is because when the proton of the carboxyl group is replaced by cobalt ions, the COO^- group remains, and absorption bands at 1655, 1448, 1397 cm^{-1} and 1320 cm^{-1} , attributed to the asymmetric and symmetric stretching vibrations of carboxylate ions, increase in the IR spectra of cobalt ion exchangers.

At the same time, the absorption bands in the range of 1320, 1121 cm^{-1} , also associated with the COON group, become weaker. Analysis of IR spectra shows that the absorption bands at 479 cm^{-1} confirm the formation of the ν Co-O bond in the complexes. Based on the literature data, as well as on the methods of SEM and IR spectroscopic analysis, it is possible to state the expected chemistry of the process of interaction of complex-forming ion exchangers with heavy metal ions Co^{2+} . Polymeric substances interacting with metals form complex compounds according to the following scheme.



The equation scheme describes the reaction of the interaction of carboxyl COOH and hydroxyl OH groups of DHT + HIPAN with metals to form a bidentate complex or chelate.

CONCLUSIONS

Based on the study of the synthesized complexing ion exchangers, the fundamental possibility of creating competitive and environmentally friendly sorption technologies using new ion exchangers for hydrometallurgy processes is shown. New polyfunctional ion exchangers with high exchange capacity and multifunctional groups based on DHT, DMT, and GIPANs have been obtained. Using a special high-resolution technique, electron microscopic studies of the phase structure and surface of ion-exchangers based on DHT and DMT were carried out in the study of the optical properties of ion exchangers by IR spectroscopy. Thus, as a result of the studies carried out, the dependence of the process of complexation of metal ions on the concentration of salts was revealed, and when using the ion exchanger DHT + HIPAN, the process of complexation increases. It is shown that the greatest value of metal complexation is manifested when using low and medium concentrations of solutions of metal salts. So, when using the norm of the ion exchanger of 2 g at a concentration of 0.005 g / l, the process of complexation of metal ions Co^{2+} with the ion exchanger DHT + GIPAN was 91.5; 90.0; 99.0; 89.2%. Analysis of the IR spectra of the samples obtained by the interaction of DHT + HIPAN with metal ions shows their differences. These facts indicate a different nature of the interaction of cobalt (II) salts with DHT + HIPAN at the same rates of ion exchanger, time, and concentration of metals. IR spectra of the studied samples confirm the formation of the Me-O bond in the ion exchanger complexes. It was revealed that the binding of DHT + HIPAN with metals occurs due to the interaction of carboxyl COOH^- and hydroxyl OH -groups. Based on the data of SEM and IR spectroscopic analysis, the proposed chemistry of the process of interaction of DHT + HIPAN with Co^{2+} ions is presented.

ACKNOWLEDGEMENTS

The authors are thankful to the authorities of Termez State University and Termez Branch of Tashkent State Technical University named after Islam Karimov.

REFERENCES

- Deev I.S. et al. (2011, No.1). Investigation of the structural features and stress-strain state of carbon fibers with cross-sectional shapes. *Materials Science*, 23-28.
- Dzhalilov A.T. et al. (2013, 1). Study of the physicochemical properties of the synthesized complex-forming anions. *Uzbek chemical journal*, 10-12.
- Dzhalilov A.T., and Eshkurbanov F.B. (2014). Investigation of the rate of vanadium sorption by anion exchangers based on thiourea and epichlorohydrin. *Reports of the Academy of Sciences of the Republic of Uzbekistan*, 60-63.
- Eshkurbanov F.B. (2013, No.6). Synthesis and study of ampholyte based on hydrolyzed polyacrylonitrile. *Uzbek chemical journal*, 13-16.

- Eshkurbanov F.B., and Dzhililov A.T. (2014, No.3). Study of the sorption properties of the obtained ion exchanger based on hydrolyzed polyacrylonitrile. *Universum: Chemistry and biology: electron. scientific. journal*. Retrieved from <http://7universum.com/ru/nature/archive/item/1068>
- Eshkurbonov F.B. (2013, 5). Obtaining ion-exchangers based on the reaction of the interaction of thiourea, epichlorohydrin, and various amines. *Uzbek chemical journal*, 27-30.
- Gul V.E. (1971). Structure and strength of polymers. Chemistry (p. 334). Moskow: Monograph.
- Kablov E.N. (2012). Strategic directions for the development of materials and technologies for their processing for the period up to 2030. *Aviation materials and technologies*, No.5, 7-17.
- Kattaev N.T., and Ramazonov A.Kh. (2016). Synthesis and physicochemical properties of the new anionic. *Electronic scientific journal*, No.7 (28). Retrieved from <http://7universum.com/ru/tech/archive/item/3399>
- Kazitsyna L.A., and Kupletskaya N.B. (1971). Application of UV, IR, and NMR spectroscopy in organic chemistry. Higher school.
- Khurram Sh. et al. (2020). A Theoretical and Experimental Framework for the Formation of Mixed Anodic Films on Combinatorial Aluminium-Cerium Alloys, *Electrochimica Acta*, 2020, 137173, ISSN 0013-4686, (<http://www.sciencedirect.com/science/article/pii/S0013468620315668>), <https://doi.org/10.1016/j.electacta.2020.137173>
- Kirilov V.N. et al. (2012). Climatic resistance and damageability of polymer composite materials, problems, and solutions. *Aviation materials and technologies*, No.5, 412-423.
- Kobets L.P., and Deev I.S. (1997, V.57). Carbon fibers: structure and mechanical properties. *J. Composites Science and Technology*, 1571-1580.
- Litvinov V.B. et al. (2011, No.3). Structural and mechanical properties of high-strength carbon fibers. *Composites and Nanostructures*, 36-50.
- Murashov V.V. (2012). Determination of physical and mechanical characteristics and composition of polymer composite materials by acoustic methods. *Aviation materials and technologies*, 465-475.
- Narisava I. (1987). Strength of polymer materials. Chemistry (p. 400). Moscow: Monograph. www.ejobios.org
- Turaev B.E. et al. (2020). Optoelectronic devices based on nanocrystalline semiconductor (CDTE) AFN-films. *Monografia Pokonferencyjna, Research, Development # 33 # 2, 33 (2)*, 28-30.
- Ulugov B.D. (2020, a). The efficiency of use of Autodesk inventor engineering programs and pedagogical information technologies in the field of "Resistance of materials" in the process of teaching students of technical universities. *ACADEMICIAN: An International Multidisciplinary Research Journal*, 10(5), 130-143. <https://doi.org/10.5958/2249-7137.2020.00253.0>
- Ulugov B.D. et al. (2020, b). The problem of measuring large currents with the help of current sensors. *EPRA International Journal of Multidisciplinary Research (IJMR) - Peer Reviewed Journal*, 6(10), 312–318. <https://doi.org/10.36713/epra2013>
- Ulugov, B.D. et al. (2020, c). Comparative analysis of modern current converters. *EPRA International Journal of Multidisciplinary Research (IJMR) - Peer Reviewed Journal* 6, no. 10 (2020): 319–25. <https://doi.org/10.36713/epra2013>