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SYNTHESIS OF IONITE BASED ON EPOXY RESINS AND POLYAMINES USING NEW INITIATING SYSTEMS FOR METAL SORPTION

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Abstract. Purification of drinking water, isolation of rare metals from industrial wastewater, and water treatment for a wide variety of applications are constant tasks requiring various solutions. Population growth, the development of new territories, agriculture, factories and manufacturing enterprises, all of them need clean water. Ionite is an ion–exchange resin, one of the ways to solve the described problems. Ionite is able to sort various ions from solutions, thereby purifying them or releasing valuable ions during sorption. The urgent task is to produce ionites more economically with minimal energy and material costs. The process of ionite synthesis consists in polymerization and copolymerization of various monomers, while introducing various initiators and changing the process conditions, it is possible to achieve a product with high characteristics.

The ionite was based on well-known monomers such as epoxy resins and aliphatic polyamines. The reagents used were (AGE) allylglycidyl ether, (ED-20) epoxy resin, and (PEI) polyethyleneimine. A product was synthesized from diglycidyl ether of dioxydiphenylpropane, allylglycidyl ether, and polyethylenimine using initiating systems based on potassium persulfate ($K_2S_2O_8$) and azobisisobutyronitrile (AIBN). The synthesis process and method of introducing the initiator into the reaction mass were selected. The reaction was carried out in an organic solvent medium (DMF) - dimethylformamide. These initiators have not been described in known sources in combination with specific reagents during the synthesis of the ion exchanger. Further, the properties of a weakly basic anion exchanger were investigated using various methods, gravimetric, acid-base titration, infrared spectroscopy, atomic emission spectral analysis. The study of sorption capacity was carried out on the ions of metavanadate (VO^{3-}). According to the content of vanadium - (V), in the solution before and after sorption, the sorption capacity of the ionite - (CE) in mg/g was calculated.

The resulting ionite can solve the problems of water purification and industrial wastewater treatment. It can be used for the selective extraction of non-ferrous and rare metals contained in industrial wastewater.

Keywords: ionite, ion exchange resin, sorption, initiators, epoxy resins, aliphatic polyamines, allylglycidyl ether, polyethylenimine, potassium persulfate, azobisisobutyronitrile.

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1. Introduction

Simple methods for producing anion exchangers based on copolymers of glycidyl methacrylate and polyamines have been studied, and their potential for extracting metal ions from aqueous solutions has been demonstrated [1, 9, 10, 22]. A comparative analysis of synthetic polyamine resins with commercial counterparts has shown their high efficiency in sorption [2]. New epoxy sorbents with amino groups for the removal of azo dyes from aqueous media [3] have been developed, as well as initiating systems for controlled radical polymerization using diazo compounds [4]. The diffusion characteristics of azo initiators and their radicals [5] have been studied, as well as the structure and application of poly(ethylene glycol)-based macroinitiators for the synthesis of cross-linked polymer structures [6]. Special attention was paid to the reproducibility of free-radical copolymerization [7], as well as to the kinetics and mechanism of the polymerization of acrylamide and methyl methacrylate in the presence of various initiators, including potassium persulfate and benzoyl derivatives [8, 14, 16]. It was found that the structural and physical-chemical properties of the resulting polymers depend on the temperature, monomer concentration, and the nature of the initiating system [13, 17, 18]. The obtained data can be used to develop thermostable and chemically stable ion exchangers with high sorption capacity for metal sorption [11, 23, 24, 25].

The aim of this study was to synthesize an ion exchanger with new initiators under new conditions and to investigate the sorption properties of the resulting product towards metal ions.

The novelty of this study lies in the use of AIBN initiators and potassium persulfate together with known starting reagents for the synthesis of an ion exchanger. The literature cited describes the use of the components separately with other reagents. Conducting a joint synthesis and studying the properties of the resulting product is an urgent task.

2. Experimental part

The initial reagents are allylglycidyl ether (AGE) with a molar mass of 114.14 g/mol, polyethyleneimine (PEI) with a molar mass of 43.04 g/mol, and epoxy resin (ED-20) with a molar mass of 60.1 g/mol. The ratio of the average molar mass of the oligomers is 1:1:1 (mol). The synthesis was carried out using equipment such as a Loip 4T-105A thermostat, as well as ISOLAB 615,10,100, and IKA eurostar 20 digital overhead mixers. AGE was introduced into a three-necked flask with a reflux condenser and heated to 80°C. ED-20 was dissolved in dimethylformamide (DMF). Next, the initiator was added at a ratio of 0.1% (mass) by weight of the reaction mass.

Ionite «A»: the initiator $K_2S_2O_8$ – potassium persulfate was introduced as a 2% aqueous solution.

Ionite "B": the initiator azobisisobutyronitrile (AIBN) was introduced as a 2% solution in DMF.

Next, PEI was added through a dropping funnel as an emulsion of a 50% aqueous PEI solution in DMF. The reaction mass was stirred until it formed a gel, making the liquid thick and viscous. Curing was carried out in furnaces at a temperature of 100 °C.

The ionite was previously washed, prepared, and converted to the OH form according to standard methods. The static exchange capacity was determined using the standard method of acid-base titration according to GOST 20255.1-89. Ionites. Methods for Determining the Static Exchange Capacity. The method of determining the moisture content of the ionite was carried out in accordance with GOST 10898.1-84, taking into account the adaptations to the specific features of the sample under study.

The sorption properties were tested on metavanadate anions (VO_3^-), and the calculation was based on the vanadium content. The starting reagent is ammonium metavanadate, NH_4VO_3. To study the dependence of the sorption capacity (1) on the concentration of the initial solution, five solutions with concentrations of 25, 50, 100, 150, and 200 mg/L were prepared over a period of 7 days. To study the dependence of the sorption capacity on the pH of the initial solution for 7 days, five solutions with a concentration of 100 mg/l were prepared, with pH values: 2, 3, 4, 5, 6, 7, acidifier – nitric acid HNO_3 0.1%, alkalizing agent - ammonia hydrate NH_4OH 0.01%. To study the dependence of the sorption capacity on the contact time of the ionite with the solution, five solutions with a concentration of 100 mg/L and a pH of 2 were prepared. The contact time was 0.5, 1, 2, 4, and 24 hours. $\mathrm{qe} = \frac{(C_0 - C)V}{g}$ (1), $R = 100 - \left(\frac{100C}{C_0}\right)$ (2), where: qe – sorption capacity (mg/g); R – degree of extraction (%); C_0 – initial concentration of metal ions in the solution (mg/L); C – equilibrium concentration of metal ions in the solution (mg/L); V – volume of the solution (L); g – weight of the ion exchanger (g).

To study the infrared spectrum, the ionite samples were crushed and mixed with potassium bromide, then dried at 40–60 °C and cooled in an desiccator.

3. Results and their discussion

At the intermediate stage of ionite «A» synthesis, a light yellow, rather fluid gel was obtained. The finished product had a soft, rubbery texture and was insoluble in acids, alkalis, and water. After drying in an oven at 40–60 °C, the sample took the form of dark yellow-brown transparent granules. At the intermediate stage of ionite «B» synthesis, a jelly-like gel with a soft, elastic, rubber-like structure was obtained. The final product was an elastic, soft, and greasy material that was resistant to water, acids, and alkalis. After drying at 40–60 °C, the ionite looked like light brown transparent granules. The characteristics of ionites are listed in Table 1.

Table 1 - Comparison of the properties of ionites obtained using potassium persulfate and AIBN as an initiator.

	Ionite «A»	Ionite «B»
The initiator used	potassium persulfate	azobisisobutyronitrile
Molar ratio	1:1:1 - AGE: PEI: ED-20	1:1:1 - AGE: PEI: ED-20
Product yield	26.2 %	43.4 %
Curing time	70 hours	57.5 hours
Static exchange capacity	1.98 meq/g	2.54 meq/g
Ionite humidity	43.7 %.	27.1 %.
Appearance	Hard, yellowish-brown, darker, transparent, brittle	Hard, yellowish-brown, lighter, transparent, elastic

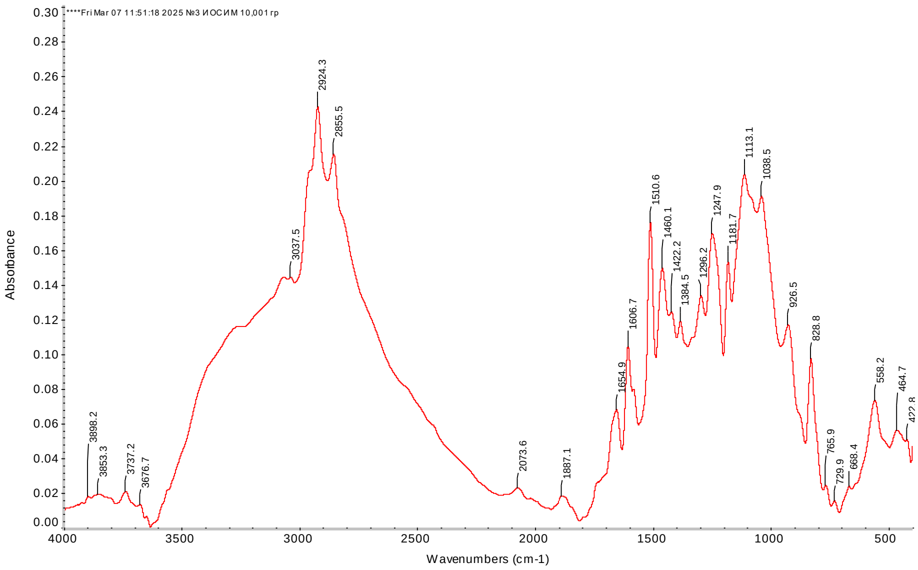

Figure 1 - IR spectrum of the «A» ionite sample

Figure 1 shows the IR spectrum of ionite «A». Figure 2 shows the IR spectrum of ionite «B». The IR spectra indicate the formation of an epoxy-amine cross-linked structure in both cases. Ion exchanger «A» is characterized by bands at 3898, 3853, 3678, 3668, and 3341 cm^{-1}, which belong to the O-H and N-H groups, as well as signals at 2937, 2924, and 2850 cm^{-1}, which correspond to the aliphatic - CH_2 fragments. The bands at 1655 and 1601 cm^{-1} are associated with the deformational vibrations of the amino groups and the aromatic framework, while the signals at 1248, 1113, and 1088 cm^{-1} indicate the CN and C-O-C bonds, i.e., the opening of the epoxy ring and the formation of a polymer network. A band around 926 cm^{-1} may indicate the presence of residual epoxy groups.

The spectrum of ion-exchange resin «B» is similar, but slightly different in the main bands. It shows bands at 3349 and 3078 cm^{-1} for the -OH, -NH-, and -NH2- groups, bands at 2925 and 2843 cm^{-1} for the -CH- fragments, and signals at 1651 and 1601 cm^{-1}. The bands at 1247 and 1112 cm^{-1} also confirm the formation of C-N and C-O-C bonds, while the band at 926 cm^{-1} is weak, which may indicate the completion of the reaction.

The difference between the spectra is no longer related to the set of functional groups, but to the way the polymer network is formed.

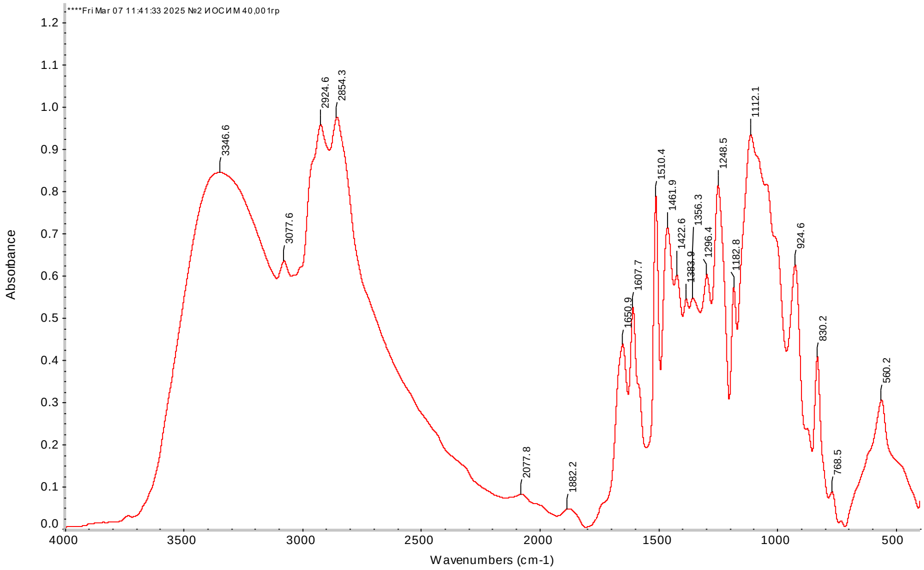

Figure 2 - IR spectrum of the «B» ionite sample

Table 2 shows the results of sorption. It has been shown that ionite «A» has higher and more stable sorption capacity values than ionite «B». In an acidic environment (pH 2.3), the maximum sorption location were observed, especially for ionite «B», but as the pH increased, its efficiency decreased significantly, while ionite «A» maintained more stable characteristics. An increase in contact time led to a regular increase in sorption capacity and degree of extraction for ionite «A», while the changes were less pronounced for ionite «B», which generally indicates a higher efficiency and stability of ionite «A» under the studied conditions. Figures 3 - 5 show the graphs of the sorption capacity as a function of concentration, pH, and time, respectively. Figure 6 shows the structural unit of the obtained ion exchange resin, where R is a branched oligomeric unit of PEI, epoxy oligomers, and oligomers of allylglycidyl ether that form a mesh structure.

Table 2 - Results of sorption of ionites «A» and «B»

Sorption depending on concentration
The ionite weight is 0.05 g; T=20ºC; static sorption for 7 days in 10 ml of the initial solution.

Initial solution (mg/L)	26.25	50.98	98.53	149	201.98
pH of the initial solution	6.6	6.6	6.6	6.6	6.6
Solution after sorption «A» (mg/l)	1	15.75	72.45	125.55	168.25
Solution after sorption «B» (mg/l)	1.65	33.92	79.1	127.5	175.78
Sorption capacity «A» (mg/g)	5.04	7.02	5.15	4.59	6.6
Sorption capacity «B» (mg/g)	4.92	3.37	3.84	4.19	5.11
Extraction rate «A» (%)	96.19	69.1	26.47	15.74	16.7
Extraction rate «B» (%)	93.71	33.46	19.72	14.43	12.97

Sorption depending on pH
The ionite weight is 0.05 g; T=20ºC; static sorption for 7 days in 10 ml of the initial solution.

Initial solution (mg/L)	99.85	104.68	101.92	102.38	103.24	99.77
pH of the initial solution	2.3	3.3	4	5.6	6.6	7.8
Solution after sorption «A» (mg/l)	37.46	57.31	71.14	73.08	72.97	64.56
Solution after sorption «B» (mg/l)	18.35	87.42	89.42	87.68	85.96	85.85
Sorption capacity «A» (mg/g)	12.4	9.36	6.12	5.77	5.92	6.97
Sorption capacity «B» (mg/g)	16.04	3.45	2.46	2.94	3.37	2.71
Extraction rate «A» (%)	62.48	45.25	30.21	28.62	29.32	35.29
Extraction rate «B» (%)	81.62	16.49	12.26	14.36	16.74	13.95

Sorption depending of time
The ionite weight is 0.05 g; T=20ºC; static sorption in 10 ml of the initial solution.

Initial solution (mg/L)	99.52	99.52	99.52	99.52	99.52
pH of the initial solution	2.3	2.3	2.3	2.3	2.3
Contact time (hours)	0.5	1	2	4	24
Solution after sorption «A» (mg/l)	80.01	61.16	43.56	35.9	22.7
Solution after sorption «B» (mg/l)	79.7	85.96	92.32	86.79	73
Sorption capacity «A» (mg/g)	3.9	7.54	11.08	12.67	15.06
Sorption capacity «B» (mg/g)	3.95	2.67	1.41	2.5	5.24
Extraction rate «A» (%)	19.6	38.55	56.23	63.93	77.19
Extraction rate «B» (%)	19.92	13.63	7.23	12.79	26.65

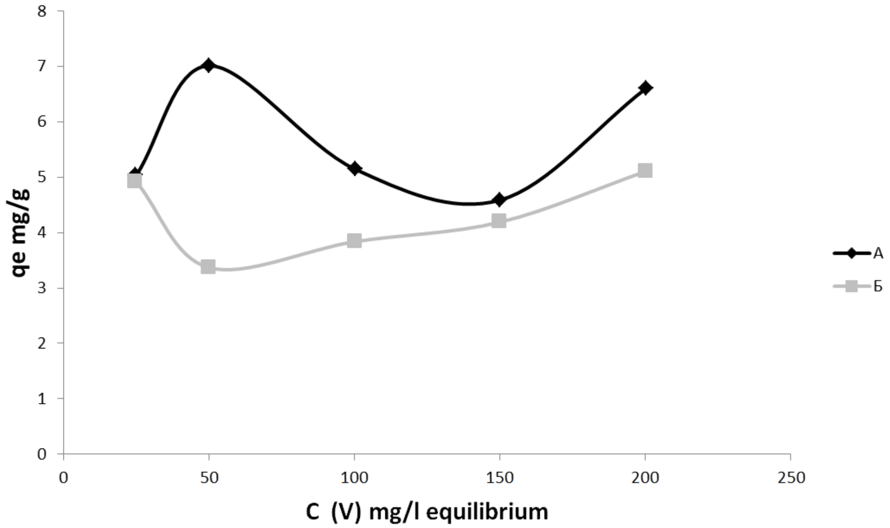

Figure 3 - Graph of the sorption capacity dependence on the concentration of the initial solution

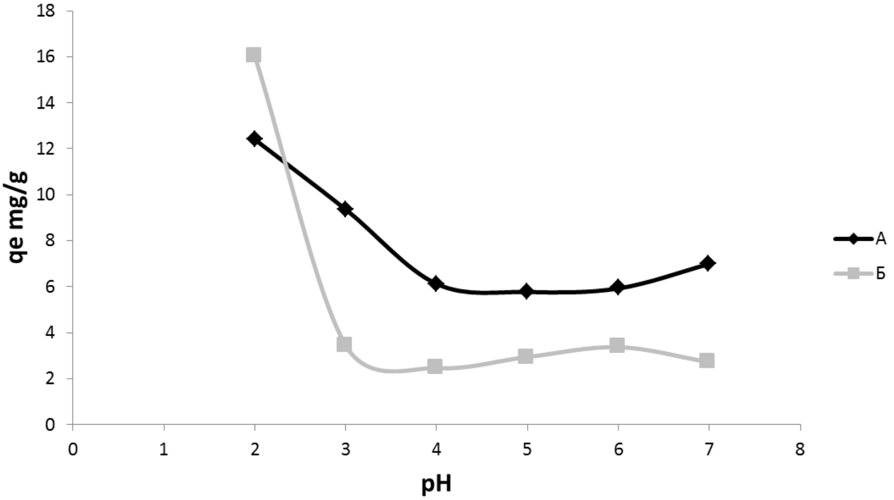

Figure 4 - Graph of the dependence of the sorption capacity on the pH of the initial solution

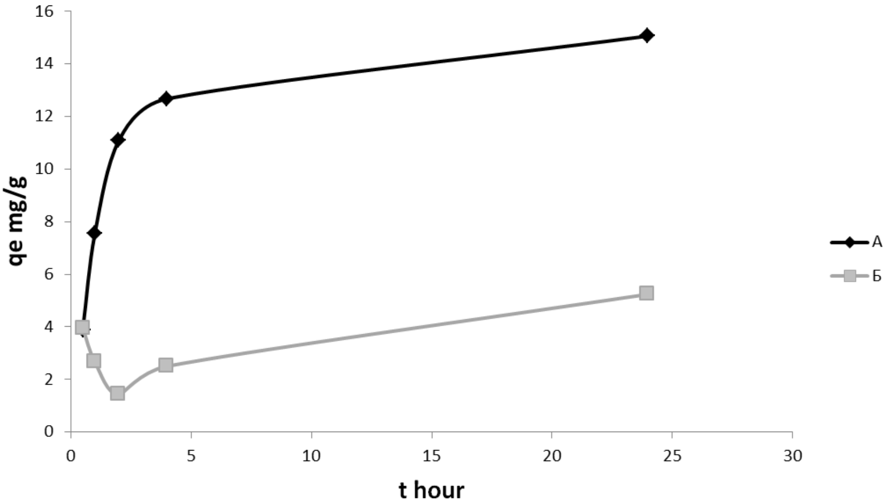

Figure 5 - Graph of the sorption capacity dependence of the sorption time

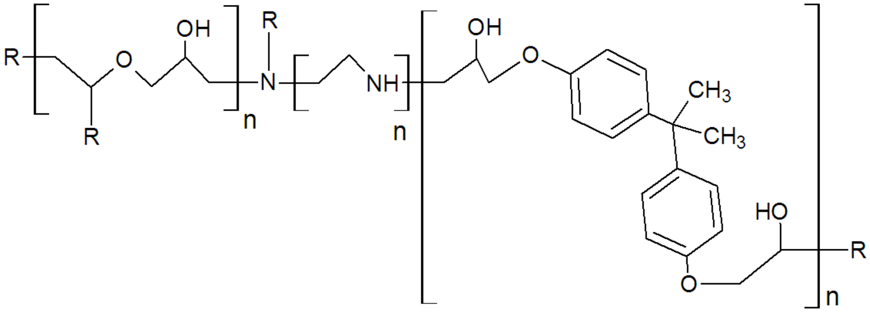

Figure 6 - Structural unit of the obtained ionite

4. Conclusion

It was possible to synthesize ionites using new initiators for this composition: potassium persulfate and AIBN. The conducted studies showed that the type of polymerization initiator significantly affects the characteristics of the obtained materials. The resulting ionites fully reflect the specific features of the synthesis conditions used and demonstrate pronounced differences in their main operational parameters.

Ionite "A" exhibited higher stability and sorption capacity, despite its higher moisture content and lower yield. The curing time was longer, which may indicate a slower polymerization process and the formation of a more developed porous structure with increased hydrophilicity. The experimental data showed that this material has stable sorption properties when interacting with metal ions. The static exchange capacity of ionite "A" was 1.98 meq/g, and the sorption capacity was 15.06 mg/g at pH 2.3 over a long sorption time.

Ionite B with a shorter curing time and higher yield had a higher sorption capacity at low pH. The static exchange capacity of ionite B was 2.54 meq/g, and the sorption capacity was 16.04 mg/g at pH 2.3.

The results of IR spectroscopy confirmed the formation of a dense and uniform polymer matrix. The obtained results confirm the potential of the synthesized ion exchangers for use in sorption processes and further research in the field of ion exchange materials.

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Conflict of interest: The authors declare that there is no conflict of interest that requires disclosure in this article.

МЕТАЛДАРДЫ СОРБЦИЯЛАУ ҮШІН ЖАҢА БАСТАМАШЫЛ ЖҮЙЕЛЕРДІ ҚОЛДАНА ОТЫРЫП, ЭПОКСИДТІ ШАЙЫРЛАР МЕН ПОЛИАМИНДЕР НЕГІЗІНДЕГІ ИОНИТ СИНТЕЗІ

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Түйіндеме. Ауыз суды тазарту, өндірістік ағынды сулардан сирек металдарды бөліп алу, суды әр түрлі қолдану салаларына дайындау әр түрлі шешімдерді қажет ететін тұрақты міндеттер болып табылады. Халықтың өсуі, жердің жаңа аумақтарын игеру, ауыл шаруашылығы, зауыттар мен өндірістік кәсіпорындар, олардың барлығы таза суға мұқтаж. Ионит-ион алмасу шайыры, сипатталған мәселелерді шешудің бір жолы. Ионит ерітінділерден әртүрлі иондарды сұрыптай алады, осылайша оларды тазартады немесе сорбция процесінде құнды иондарды шығарады. Ең аз энергетикалық және материалдық шығындармен иониттерді неғұрлым экономикалық тиімді өндіру өзекті міндет болып табылады. Иониттерді синтездеу процесі әртүрлі мономерлерді полимерлеу және сополимерлеу болып табылады, ал әртүрлі бастамашыларды енгізу және процесс жағдайларын өзгерту арқылы жоғары өнімділікке қол жеткізуге болады. Иониттің негізі эпоксидті шайырлар мен алифатты полиаминдер сияқты белгілі мономерлер болды. Қолданылатын реагенттер: (АГЭ) - аллилглицид эфирі, (ЭД-20) - эпоксидті шайыр, (ПЭИ) – полиэтиленимин. Диоксидифенилпропан диглицидил эфирінен, аллилглицедил эфирінен және полиэтилениминнен калий персульфаты ($K_2S_2O_8$) және азобисизобутиронитрил - (АИБН) негізіндегі инициациялық жүйелерді қолдану арқылы өнім синтезделді. Синтез процесі және бастамашыны реакция массасына енгізу әдісі таңдалды. Реакция органикалық еріткіш (ДМФА) – диметилформамид ортасында жүргізілді. Бұл бастамашылар белгілі көздерде ионит синтезіндегі нақты реактивтермен бірге сипатталмаған. Әрі қарай әр түрлі әдістерді, гравиметриялық, қышқылдық-негіздік титрлеуді, инфрақызыл спектроскопияны, атомдық-эмиссиялық спектрлік талдауды қолдана отырып, әлсіз негізді аниониттің қасиеттері зерттелді. Сорбциялық қабілетін зерттеу метаванадат иондары (VO_3^-) бойынша жүргізілді. Ванадийдің құрамы бойынша – (V), сорбцияға дейінгі және кейінгі ерітіндіде иониттің сорбциялық сыйымдылығы - (СЕ) мг/г деп саналды. Алынған ионит су мен өндірістік ағынды суларды тазарту мәселелерін шеше алады. Өнеркәсіптік ағынды суларда кездесетін түсті және сирек металдарды селективті оқшаулау үшін қолдануға болады.

Түйінді сөздер: ионит, ион алмасу шайыры, сорбция, бастамашылар, эпоксидті шайырлар, алифатты полиаминдер, аллилглицедил эфирі, полиэтиленимин, калий персульфаты, азобисизобутиронитрил.

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СИНТЕЗ ИОНИТА НА ОСНОВЕ ЭПОКСИДНЫХ СМОЛ И ПОЛИАМИНОВ С ПРИМЕНЕНИЕМ НОВЫХ ИНИЦИИРУЮЩИХ СИСТЕМ ДЛЯ СОРБЦИИ МЕТАЛЛОВ

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Резюме. Очистка питьевой воды, выделение редких металлов из промышленных стоков, подготовка воды для самых различных областей применения являются постоянными задачами, требующие различных решений. Рост населения, освоение новых территорий земли, сельское хозяйство, заводы и производственные предприятия, все они нуждаются в чистой воде. Ионит – ионообменная смола, один из способов для решения описанных задач. Ионит способен сортировать различные ионы из растворов, тем очищая их или выделять ценные ионы в процессе сорбции. Актуальной задачей является более экономически выгодное производство ионитов с минимальными энергетическими и материальными затратами. Процесс синтеза ионитов заключается в полимеризации и сополимеризации различных мономеров, при этом вводя различные инициаторы, и меняя условия процесса можно добиться продукта с высокими характеристиками. Основой для ионита послужили известные мономеры, такие как эпоксидные смолы и алифатические полиамины. Используемые реагенты: (АГЭ) - аллилглицидиловый эфир, (ЭД-20) - эпоксидная смола, (ПЭИ) – полиэтиленимин. Был синтезирован продукт из диглицидилового эфира диоксидифенилпропана, аллилглицедилового эфира и полиэтиленимина с применением инициирующих систем на основе персульфата калия ($K_2S_2O_8$) и азобисизобутиронитрила - (АИБН). Был подобран процесс синтеза и способ введения инициатора в реакционную массу. Реакция велась в среде органического растворителя (ДМФА) – диметилформамид. Данные инициаторы не были описаны в известных источниках в сочетании с конкретными реагентами при синтезе ионита. Далее были исследованы свойства слабоосновного анионита с применением различных методов, гравиметрического, кислотно-основного титрования, инфракрасной спектроскопии, атомно-эмиссионного спектрального анализа. Исследование сорбционной способности проводилось по ионам метаванадата (VO_3^-). По содержанию ванадия - (V), в растворе до и после сорбции, считалась сорбционная емкость ионита – (CE) в мг/г. Полученный ионит может решить проблемы очистки воды и промышленных стоков. Может целесообразно применятся для селективного выделения цветных и редких металлов, содержащихся в промышленных стоках.

Ключевые слова: ионит, ионообменная смола, сорбция, инициаторы, эпоксидные смолы, алифатические полиамины, аллилглицедиловый эфир, полиэтиленимин, персульфат калия, азобисизобутиронитрил.

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