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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS AND STUDY OF SULFOCATIONITES BASED PHENOL-FORMALDEHYDE OLIGOMERS MODIFIED WITH AMIDE COMPOUNDS

Speciality: **2304.01 – Macromolecular Chemistry**

Field of science: Chemistry

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GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and the degree of research development. In the era, a primary objective of macromolecular chemistry is development of novel macromolecular compounds (MC) with tailored new compositions and specific performance indicatots. These materials are designed to replace existing analogues across various fields of industry. Currently, one of the most rapidly developing directions in science and technology is the synthesis, research and application of ion exchangers derived from macromolecular compounds[1].

In recent years, the production volume of ionites (sulfocationites) based on synthetic polymers has increased significantly worldwide. The main part of the ionites obtained is widely used in the processes of softening and purification of industrial water, the separation of non-ferrous and precious metals from ores in the hydrometallurgy, and the remediation of wastewater through the removal of toxic and heavy metals[2].

High molecular weight compounds (HMC) are used in various fields of science and technology, imposing certain requirements on the ion exchangers obtained on the basis of these compounds. These requirements indicate that the synthesis of compounds with new properties is promising. Consequently, the preparation of ion exchange sulfocationites, the study of their physicochemical properties and their subsequent application in various fields is considered one of the current scientific directions. Since ion exchange sulfocationites belong to polyfunctional compounds with numerous functional groups, their scope of application is exceptionally broad.

Literature studies show that the properties of ion exchangers depend on their composition, synthesis conditions, physicochemical, physicomechanical, sorption and other indicators. When using ion-

[1] Grachek, V.I. Synthesis and research of properties of new nitrogen-sulfur-containing fibrous ionites / V.I. Grachek, A.A. Shunkevich, O.I. Isakovich [et all.] // Journal of applied chemistry, – 2013. №11, – p. 1757-1762 .

[2] Imanbekov, K.I., Ergozhin, E.E., Kendirzhanov, E.R. Vyniloxyethylamine and epoxyphenolaldehyde based ionites // – Saint-Petersburg: Applied chemistry, – 2007. №8, – p. 1394-1396.

exchange sulfocationites in various industries, particular attention should be paid to their insolubility in water and organic solvents, high sorption properties capacity toward various ions, chemical and thermal stability, the possibility of reuse after regeneration, as well as environmental and economic feasibility. In many cases, it is difficult for ion exchangers to fully combine this set of proporties. The synthesis, research, and application of new ion exchangers that satisfy aforementioned performance characteristics remain critical, practical and theoretical objectives in scientific research.

Ion exchangers (sulfocationites) derived from MB, including thermoreactive oligomers and cooligomers are classified as insoluble polyelectrolytes, they consist of macromolecules with a network structure, distinguished by chemical, mechanical, and thermal stability at various pH values for a long time in the environment, and exist in the form of granules, thin layers (membranes), etc. Many well known ion exchangers are produced on the basis of appropriate transformations of MB, among which phenol-formaldehyde oligomer (PFO) holds particular importance.

Due to the low amount of free monomers in the modified products of FFO, which are produced on an industrial scale, have wide raw material base, and its non-environmental hazard, and its functional capabilities, it has attracted interest to use them in the production of ion exchangers (sulfocationites). The use of sulfocationites based on modified phenol-formaldehyde oligomer (MPFO) for various purposes is relevant because it is both economically and environmentally favorable.

It is known that PFO conversion products have limited swelling capacity, resistance to thermal and aggressive environments, and physical and mechanical properties. Initial studies have revealed that the sulfonation products of these compounds also have ion exchange capabilities. However, the synthesis and study of MPFO-based sulfocationites with organic compounds containing amide groups has not been conducted.

The possibilities of synthesizing ion-exchange sulfocationites for the first time were investigated by using MPFO as a raw material in combination with amide group compounds.

The research was carried out in accordance with the scientific research plan of the "Technology of Organic Substances and High-Molecular Compounds" department of the Azerbaijan State Oil and Industry University, within the framework of "Synthesizing industrially significant chemical substances and materials, the development and implementation of diverse technological processes".

Object and subject of the study. MPFO with amide group compounds was taken as the object of the study. Modified oligomers were sulfonated, the optimal variant of the process was determined, the structure, physicochemical properties, thermal stability of the synthesis products were studied.

The subject of the study encompasses the cation-exchange properties of MPFO sulfo derivatives modified with amide group compounds alongside the formulation of recommendations for practical application.

Research aims and objectives. The aim of the dissertation is to synthesize new sulfocationites possessing ion-exchange properties by sulfonating thermoreactive PFO modified with amid containing compounds (acetamide (AA), benzamide (BA), carbamide (KA), oxamide (OA) and terephthaldiamide (TPDA)) through polymer analogous transformation reactions, followed by a comprehensive study of their properties and potential industrial applications.

To achieve this goal, the following objectives were established:

- Development of a methodology for sulfonation of MPFO with organic compounds containing one or two amide groups (AA, BA, KA, OA and TPDA);
- Optimization of the synthesis process through mathematical modeling of experiments;
- Elucidation of the chemical structure of the obtained sulfo-derivatives and the underlying sulfonation mechanisms using advanced chemical and physical analytical techniques to validate the research hypotheses;
- Characterization of the physicochemical and thermal properties (using DTA and TGA methods) of the sulfonation products derived from MPFO and amide containing organic compounds;

– sulpho derivatives (bulk volume, true density, solubility in various solvents, swelling rate, specific volume, mechanical strength, static and dynamic exchange capacity, ion exchange rate, etc.) and analysis of the results;
– Investigation of the application possibilities of cation exchange compounds in softening water hardness, determination of patterns, comparison with known industrially significant sulfocationite (KU -1) and unmodified PFO-based sulfocationite;
– Proposing a scheme for industrial-scale production of the sulfonation process by making some additions to the existing technological scheme;
– Quality determination of new sulfocationites using fuzzy logic.

Research methods. Sulfocationites based on MPFO with amide group compounds were synthesized in laboratory conditions. Their physical and chemical properties and performance indicators of the transformation products were studied using appropriate chemical analysis methods. Additionally, mathematical modeling was used in process optimization, IR spectroscopy was used to investigate the probable structure of sulfo derivatives (using "Protege-460" spectrometer of the Nicolet company and the Lumos IR-Fourier microscope of the Bruker company), DTA and TGA were used in the study of thermal properties (in the "Jupiter STA 449F_3" apparatus from NETZSCH company), Laser diffraction was used to determinate particle size (in the Mastersizer-3000 Laser device), and X-ray diffraction was used in structural studies (XRD TD 3500).

Main provisions submitted for defense

– Study of the sulfonation process of thermoreactive (resol-type) PFO modified with amide group organic compounds (AA, BA, OA, KA and TPDA), and characrerization of the the structure and properties of the resulting products;
– nitrogen- containing MPFO, their effectiveness as cationites, and comparative analysis with established analogues;
– Recommendations for the application of new sulfocationites and reduction of water hardness in technical waters ;
– Proposing a conceptual technological scheme for the industrial-scale production of sulfonated products.

Scientific novelty of the research. For the first time by sulfonating thermoreactive (resol-type) PFO modified with amide group organic compounds (AA, BA, OA, KA and TPDA), sulfocationites with cation exchange properties were synthesized and used in the softening of hard water. The patentability of these findings serves as a primary indicator of the scientific novelty of the work.

- The nitrogen-containing PFO was optimized by mathematical modeling of experiments, the optimal process parameters were determined as following oligomer to acid molar ratio of 1:3, temperature 135^0C and reaction time 2-3 hours.
- The structure of MPFO sulfo derivatives (sulfocationites) modified with amid containing organic compounds was studied by IR spectroscopy, confirming the probable reaction mechanism;
- It was found that the incorporation of nitrogen-containing modifiers into the structure of macromolecules enhances cation-exchange properties. This effect was attributed to the creation of polar centers by nitrogen-containing fragments, which facilitate cation attraction. Furthermore, he high content of methylol groups (~10-12%) in thermoreactive unmodified PFO and their participation in sulfonation process were confirmed.
- Granulometric analysis was performed using Laser diffraction to determine the particle size and volume fractions of the sulfonation products of MPFO. The diameters corresponding to 10, 50, and 90% of the cumulative volume were determined. In each case, it was found that the particle sizes of the samples modified with amide group compounds were significantly higher than those of their unmodified analogues for the same volume fractions.
- It was found that the thermal stability of sulfonation products of MPFO with organic compounds containing amide groups was high. The residual mass at ~900^0C in the oligomer sample modified with TPDA was 16%, and in the oligomer sample modified with BA it was 15%;
- The possibility of using sulfonation products of some PFO modified with organic compounds containing amide groups in aggressive environments has also been determined;

– It was determined that MPFO-based sulfocationites with organic compounds containing one or two amide groups have the ability to reduce the total hardness of water from 8.5 and 10.0 mg-eq/l to 0.3-0.5 mg-eq/l, and their application was considered highly appropriate.

Theoretical and practical significance of the study. The main indicators of MPFO-based sulfocationites modified with organic compounds with amide groups were studied. These indicators were compared with the indicators of the unmodified oligomers, as well as with the main indicators of the industrially important KU-1 brand ion exchanger. This study revealed that sulfocationites characterized by a low content of free phenol and formaldehyde are environmentally safe. Sulfocationites synthesized from such compositions can be used in water softening processes. This is both economically and environmentally favorable. The exchange capacity of such ionites is approximately 1.5 times higher. They are more durable in chemical and mechanical terms.

The proposed ion exchangers are suitable for use in technical water treatment, metallurgy, organic synthesis, related fields.

It has been proposed to obtain MPFO-based sulfocationite on an industrial scale by making certain additions to the existing technological scheme.

Approbation and application of the dissertation work. The results of scientific research on the topic of the dissertation were reported at International (9) and Republican (1) Scientific Conferences and published in relevant materials:

VI International Conference on European Science and Technology (Munich, Germany-2013); Materials of the XIII International Scientific Conference "Actual Problems of Chemistry" for doctoral students, masters and young researchers dedicated to the 96th anniversary of the birth of the National Leader H. Aliyev (Sumgayit-2019); International Conference on Actual Problems of Chemical Engineering, dedicated to the 100th anniversary of the Azerbaijan State Oil and Industry University (Baku-2020); International Conference on "Reconstruction and recovery in post-conflict situations-RRPCS" (Baku-2021); “IV International Scientific

Conference of Students and Young Researchers dedicated to the 100th anniversary of the birth of the National Leader Heydar Aliyev" (Baku-2023); "Scientific Conference of Doctoral Students and Young Researchers dedicated to the 100th Anniversary of the birth of the National Leader Heydar Aliyev" (Baku-2023); "Heydar Aliyev and the Nature of Azerbaijan" International Conference Dedicated to the 100th Anniversary of the Birth of the National Leader of the Azerbaijani People Heydar Aliyev, Institute of Molecular Biology and Biotechnologies (Baku-2023); International Scientific Conference "Modern Problems of Macromolecular Compounds Technology" (Baku-2024); All-Russian scientific conference with international with the participation of "Modern problems organic chemistry" (Novosibirsk -2025); XII International conferences "Polymer material lowered fuel" (Balashikha - 2025) .

Publications relevant to the dissertation. The results of the scientific research conducted within the scope of the dissertation work were reflected in 26 published works.These include 13 articles (3 Web of Science), 10 theses published in conference proceedings (1 republican and 9 international), 2 are textbooks appropriate to the specialty. Also, the candidate has secured 1 test certificate and 1 patent appropriate to the topic. Most of the articles have been published in foreign peer-reviewed journals, such as "Actual Problems of Humanities and Natural Sciences", "Scientific Publications of Postgraduate and Doctoral Students", "International Scientific Journal Theoretical and Applied Science", "Plastic Masses", etc.

Scope and structure of the dissertation. The dissertation consists of an introduction, four chapters, a conclusion, a list of references, and two appendices.

The total volume of the work comprises 159 pages, including 29 figures, 26 tables and 169 bibliografic references. The total characters count 180981 (including introduction 14795, chapter I -63331, chapter II -20213, chapter III -46273, chapter IV-34328, conclusions- 2041 characters).

MAIN CONTENT OF THE DISSERTATION WORK

The introduction provides an overview of the relevance of the research defines the goals and objectives,highlights scientific innovations, practical significance of the findings,and presents an approbation of the conference materials that reflect the main results of the dissertation.

The first chapter investigates current trends in the synthesis, characterization, and application of PFO, especially oligomers modified with organic compounds of various classifications. Literature references reflecting their ion-exchange properties, along with other areas of application are provided. In addition, pier-reviewed research related to ion-exchangers obtained on the basis of various reactive polymers is analyzed. Finally, the choice of the dissertation topic was justified through analytical analyses.

The second chapter describes to a description of the equipment, reagents, and research methods used in the implementation of the work, including the sulfonation process of MPFO using nitrogenous compounds, as well as methods for studying the ion exchange properties of sulfocationites.

In the third chapter examines the factors affecting the performance of ion exchangers (sulfocationites) based on MPFO with organic compounds containing an amide group (AA, BA, OA, KA and TPDA), depending on their composition, the mechanism of the ion exchange process, the determination of the optimal parameters of the sulfonation process by mathematical modeling, the results of spectral (IR, DTA and TGA, Mastersizer, X-ray phase) analyses of sulfocationites are presented and relevant analyses are conducted.

The fourth chapter presents the results of evaluation of cation exchangers based on sulfonation products of MPFO in reducing water hardness. The optimal sulfocationite was selected based on multiple evaluation criteria using the fuzzy TOPSIS method.

The final part of the dissertation presents the scientific conclusions drawn from the research and provides a list of cited literature.

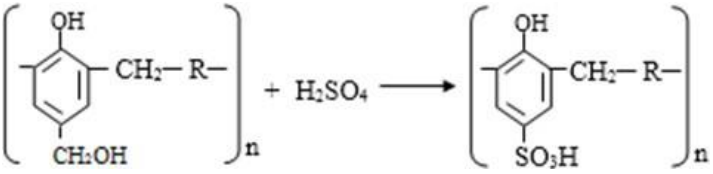
1. Sulfonation of thermoreactive PFO modified with organic compounds containing amide groups

The sulfonation process of PFO functionalized with amide containing organic compounds was carried out at 135^0C for 2-3 hours according to the following procedure.

MPFO (40 g) and 98% sulfuric acid (120 g) are added to 250 ml volume laboratory reactor and equipped with a condenser and heated to 135^0C. After the oligomer is completely melted, it is cooled to room temperature, transferred to a vessel, where a calculated volume (30 ml) of a 37% solution of aqueous formaldehyde (formalin) is added to the reaction mass. The vessel is solidified in an oil bath at a temperature of 110^0C for two hours. After cooling, the resulting sulfocationite is washed with water until the effluent reaches a neutral pH (pH=7). Then, its particle size is reduced to 1-2 mm.

In the research, MPFO with amide group containing organic compounds was used as the main raw material for the preparation of sulfocationites for the first time. The properties of sulfocationites synthesized via the modification process with various amide compounds (AA, BA, OA, TPDA and KA) exhibit partial variations. When the degree of copolycondensation increases, the static and dynamic conversion capacities of the obtained oligomer-based sulfocationites also change. Oligomers modified with amide group organic compounds were sulfolated by analogous conversion reactions, as a result of which a sulfogroup was introduced into each benzene ring.

The synthesized sulfocationites can be generally shown as follows:



Here, R – represents an organic compound containing an amide group.

MPFO-based sulfocationites with amide group compounds are dark brown or black compounds that are insoluble in water and organic solvents.

2. Determination of optimal parameters of the sulfonation process using mathematical modeling

Developing a mathematical model of the reaction to optimize the process of obtaining sulfonic cations is one of the important and urgent research issues.

In order to optimize the sulfonation process, experimental studies were conducted on various input parameters, a database was created based on the results obtained, and functional dependencies between mathematical modeling methods and process parameters and optimal regime indicators were determined.

During the study, two of the three input parameters were kept constant, and the dependence of the sulfocationite yield on the third parameter was analyzed. To more clearly identify the regularities, the sulfonation process was carried out under the same acid consumption conditions, and graphs of the dependence of the yield on the variable parameter were constructed while keeping the two temperature parameters constant. The results are shown in Figure 2.1.

As illustrated in the figure, for the first and second curves, (mass of 40 g, duration of 2 and 3 hours), the yield of sulfocationite increased by 2-4% as the temperature rise from 120^0C to 150^0C (from 72.16% to 73.22% and from 70.08% to 74.17%, respectively) were observed.

The same trends were observed for the process taking 60 g and lasting 2 hours. Thus, in the third curve, the yield varies in the range of 74.07-76.03%, and with an increase in the amount of components, it takes a slightly higher value. The fourth curve demonstrates that at a mass of 50 g and a duration of 2.5 hours, the yield does not noticeably depend on temperature, but forms at the same point: 78.08-78.09%.

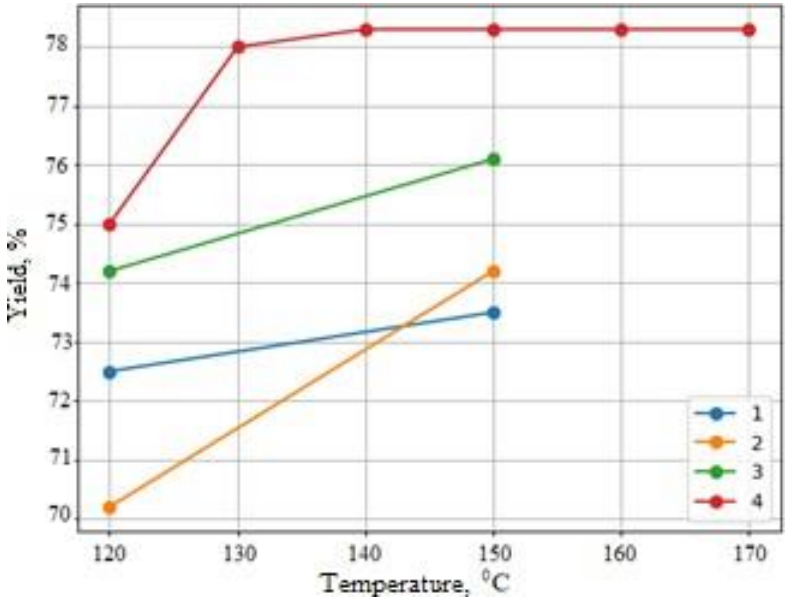


Figure 2.1. Temperature dependence of the sulfonic acid yield:

1. The oligomer mass is 40 g, the process duration is 2 hours.
2. The oligomer mass is 40 g, the process duration is 3 hours.
3. The oligomer mass is 60 g, the process duration is 2 hours.
4. The oligomer mass is 50 g, the process duration is 2.5 hours.

KA was studied using the multi-factorial experimental design method. The objective of the study was to characterize the interaction between the factors affecting the process through a mathematical polynomial dependence and to determine the optimal technological regimes based on the resulting model. Based on the experimental results, the primary input and output parameters of the process were determined and their mutual influence was analyzed.

The factors affecting the main output parameter in the study – the yield of MPFO-based sulfocationite with KA are presented in Table 2.2:

X_1 – mass of the oligomer relative to a fixed amount of acid, g;

X_2 – reaction temperature, ^{0}C;

X_3 – reaction time, hours.

Table 2.2

Baseline level and range of change of factors

Names	Natural scale of factors		
	X_1	X_2	X_3
Basic level	50	135	2.5
Variation step	10	15	0.5
Lower level	40	120	2
Upper level	60	150	3

Based on the proposed mathematical model, the sulfonation process was optimized. The optimal values of the technological parameters of the process were determined and the optimal indicators corresponding to the maximum yield were determined as follows:

X_1=32 g., X_2=135^{0}C, X_3=2.5 hours

Experiments conducted under optimal conditions demonstrate the accuracy of the results obtained. Thus, the optimal parameters of the sulfonation process were studied using the multifactorial design of experiments method and it was found that the maximum yield is 78.13% when the amount of oligomer is 32 g, the process temperature is 135^{0}C, and the reaction time is 2.5 hours (Table 2.3).

Table 2.3

Planning matrix and results of experiments

m	W	v	m	W	v	mW	mv	Wv	mWv						
x1	x2	x3	x1	x2	x3	x1x2	x1x3	x1x4	x1x4	$x^2=2d^2$	$x3^2=x2d^2$	$x3^2=x3d^2$	$x3^2=x3d^2$	y1	y1
300	150	3	1	1	1	1	1	1	1	0,2697	0,2697	0,2697	0,2697	75,34	75,3
150	150	3	-1	1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,17	74,5
120	150	3	1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,17	74,9
60	120	3	-1	-1	-1	-1	-1	1	1	0,2697	0,2697	0,2697	0,2697	74,56	74,9
100	150	3	-1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,56	74,5
60	150	3	1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,21	74,5
60	60	4	1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,21	74,4
50	50	5	1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,23	74,3
50	50	5	-1	-1	-1	-1	-1	-1	-1	0,2697	0,2697	0,2697	0,2697	74,07	74,0
40	70	6	10	0	0	0	0	0	0	0,2697	0,2697	0,2697	0,2697	74,59	72,0
70	65	15	105	1	1	1	-1	1	-1	0,2697	0,2697	0,2697	0,2697	72,70	72,3
31,334	**135**	**2.5**	-1	-1	-1,215	0	0	1,2154	0	0,7486972	-0,7303	1,4771743	-0,7303	**78,13**	**77,9**
35	125	2.5	135	0	-1,215	0	0	0	0	0,7486972	0,7486972	-0,7303	-0,7303	78,13	77,9
10	12,5	1.5	12,5	0	0	0	0	0	0	-79,77	-0,7303	-0,7303	-0,7303	78,73	78,8
10	21556	10	10	0	0	0	0	0	0	-79,77	-0,7303	-0,7303	-0,7303	78,89	78,8
6	31	15	5	0	0	0	0	0	0	-79,77	-0,7303	-0,7303	-0,7303	78,83	78,8

3. Study of the probable structure of MPFO-based sulfocationites modified with amide compounds using spectral analysis

The structural characteristics of sulfonation products of modified oligomers were studied by infrared (IR) spectroscopy (Fig. 3.1-3.2). IR-spectral analysis of the samples was carried out on a Nicolet "Protege-460" spectrometer, as well as on a LUMOS IR-Fourier microscope (BRUKER). The spectra were recorded within the wavelength range of 600-4000 cm^{-1}.

The IR spectral properties of MPFO-based sulfocationites synthesized in the presence of alternative modifiers were examined. The analysis results showed that although slight shifts were observed in certain absorption bands, the characteristic bands of the functional groups were generally similar and structurally analogous across the samples.

The observation of absorption bands at 1609-1687 cm^{-1} belonging to $–SO_3$ groups in the IR spectrum of unmodified and MPFO-based sulfocationites indicates that sulfocationites are formed due to the formation of chemical bonds as a result of the interaction of oligomers with solid sulfuric acid.

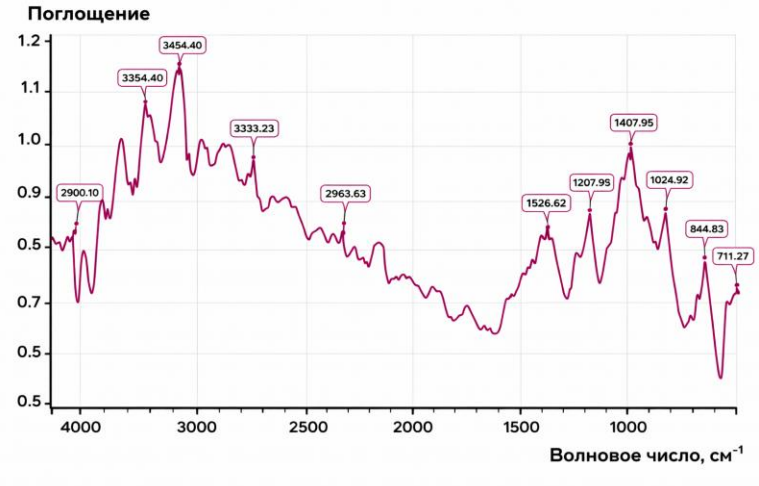


Figure 3.1. IR spectrum of unmodified PFO-based sulfocationite

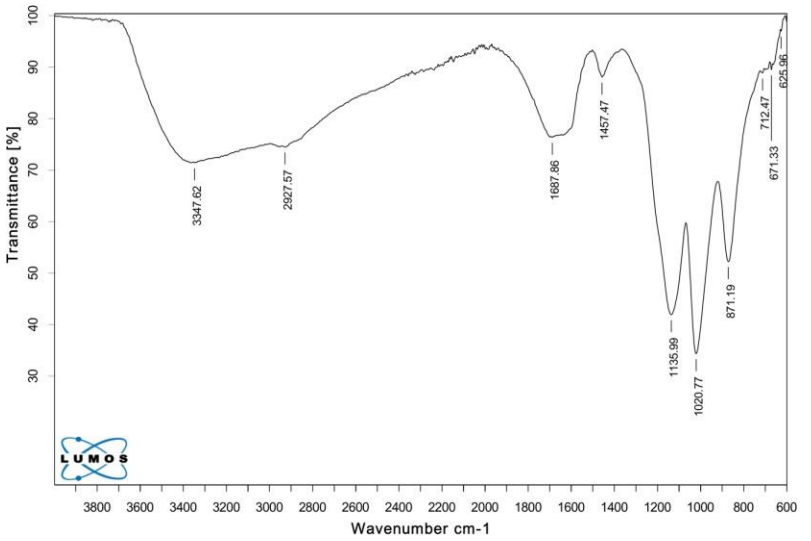


Figure 3.2. IR spectrum of KA-modified PFO-based sulfocationite

IR spectra of AA, BA, OA, KA and TPDA modified PFO-based sulfocationites revealed the presence of $–SO_3$ groups in the absorption bands at 1617.04-1639.20; 1653.06; 1687.86; 1609.13-1655.08 and 1655.08 cm^{-1}, respectively. These results indicate that methylol groups readily react with sulfuric acid, leading to the addition of sulfo groups (HSO_3) to the benzene ring.

In the IR spectrum of both oligomer and sulfocationite, the presence of a benzene ring is confirmed by valence vibrations in the 1600-1450 cm^{-1} bands and deformation vibrations in the 1610-1590 cm^{-1} bands. ≡CH and $=CH_2$ groups are observed in the 765-705 cm^{-1} and 2840-2940 cm^{-1} absorption bands. The presence of the HSO_3^- group is determined by valence vibrations in the 1300-1570 cm^{-1} bands and deformation vibrations in the 1070-1036 cm^{-1} bands.

Thus, all functional groups consistent with the proposed mechanism of sulfonation of oligomers were observed in the IR spectrum, confirming the synthesis of the target compounds.

The destruction process of ion exchangers depends on the structure of the macromolecule used, the nature and form of the

ionogenic group, and occurs at different temperatures. This process is mainly carried out in the presence of polar or ionogenic groups within the polymer matrix.

Thermogravimetric study of samples was carried out on a "Jupiter STA 449F_3" apparatus (NETZSCH), in the temperature range of 100-900^{0}C, The temperature increase rate was 20 K/min in an inert (nitrogen) environment and the correlation between the thermal properties of the samples and their composition was studied (Fig. 3.3-3.4). The results were compared with the corresponding indicators of unmodified PFO. Some similar results were obtained when alternatives modifiers were used.

Unmodified or MPFO-based ion exchangers are used in various processes, including those requiring thermal stability when operated at low and high temperatures. Thermogravimetric analysis has shown that thermal decomposition of MPFO-based sulfocationites with unmodified and those containing amide-group compounds occurs stepwise both increasing temperature.

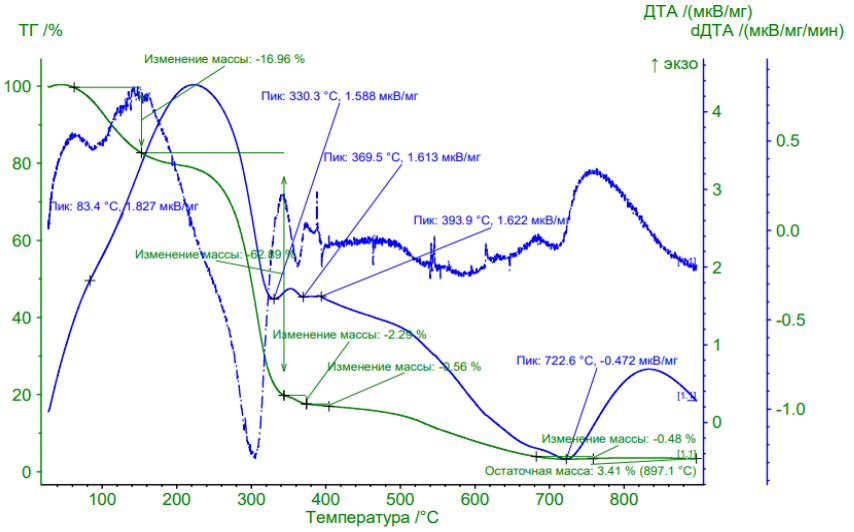


Figure 3.3. Thermal degradation of unmodified PFO-based sulfocationite

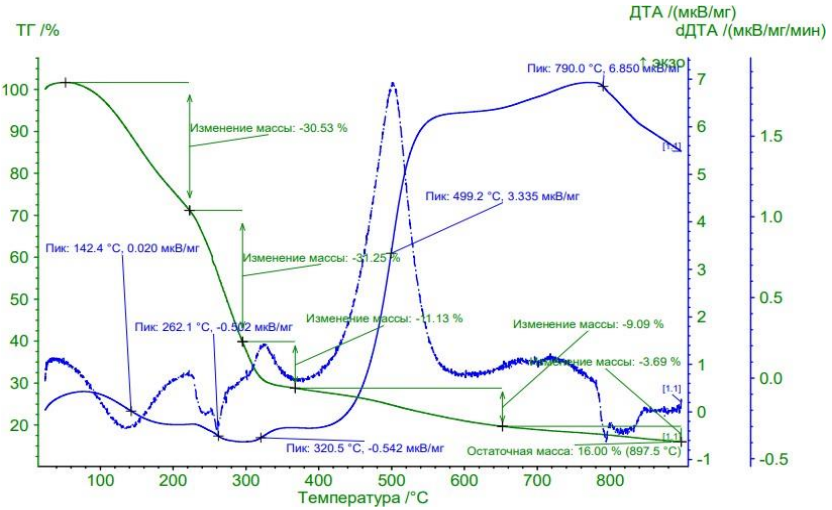


Figure 3.4. Thermal degradation of PFO-based sulfocationite modified with TFDA

In the range of 200-300^{0}C, the separation of mainly moisture and volatile components is observed, while the temperature range of 300-400^0 C is characterized as the main degradation stage, during which a significant mass loss occurs in the samples.

At temperatures exceeding 400^{0}C, modified samples containing nitrogen exhibit higher thermal stability, which is explained by the strength of the C–N bonds within their structure. The results of the analysis show that sulfocationites modified with amide group compounds have higher thermal stability, and the best indicators are observed in samples modified with BA and TPDA.

Table 3.1 presents key parameters of sulfocationites of the thermal destruction of both unmodified PFO and MPFO-based sulfocationites of various compositions, specifically their half-life duration and half-disintegration temperature.

As shown in the table, the half-lives and half-life durations and half-disintegration of MPFO-based sulfocationites vary depending on the specific amide groups. Overall, the results obtained show that the type and structural features of the modifiers directly affect the thermal stability of sulfocationites, and the presence of structural units leads to an increase in the thermal stability of sulfocationites.

Table 3.1

Sulfocationites with different compositions half-life and temperature

No.	Name of sulfocationites	Half-disintegration duration, min.	Half-disintegration temperature, ^{0}C
1	Unmodified PFO-based sulfocationite	8.0	210
2	AA modified PFO-based sulfocationite	11.0	240
3	BA modified PFO-based sulfocationite	11.0	260
4	OA modified PFO-based sulfocationite	10.2	225
5	KA modified PFO-based sulfocationite	10.4	220
6	TPDA modified PFO-based sulfocationite	12.1	280

The particle size determination was carried out using the next-generation laser analyzer Mastersizer 3000. Table 3.2 presents the particle size distribution parameters (D_{10}, D_{50}, D_{90}) of the investigated sulfocationite samples, along with their distribution characteristics.

Table 3.2

Particle size distribution of sulfocationites with compositions

No.	Sulfocationites	D_{10} μm	D_{50} μm	D_{90} μm	Of volume, %	Span
1	Unmodified PFO-based sulfocationite	121	184	270	18.5	0.808
2	AA modified PFO-based sulfocationite	155	276	417	16.5	0.949
3	BA modified PFO-based sulfocationite	97.5	161	240	17	0.887
4	OA modified PFO-based sulfocationite	367	528	734	21.5	0.697
5	KA modified PFO-based sulfocationite	385	602	914	11.5	0.879
6	TFDA modified FFO-based sulfocationite	178	313	557	12.5	1,208

The results presented in the table indicate that the particle size and their distribution characteristics in modified oligomer-based sulfocationite samples significantly depend on the nature of the amide

group used. Specifically, some modifiers lead to an increase in particle size (OA, KA), whereas others result in the formation of finer particles with a relatively homogeneous distribution (BA). Based on the values of the span parameter, the sample modified with TFDA is characterized by a broader particle size distribution (heterogeneous structure), while the OA-modified sample exhibits a narrower distribution range (relatively homogeneous system). Thus, comparative analysis of the granulometric parameters demonstrates that particle size and distribution characteristics directly influence the application properties of sulfocationites.

Structural characterization of the synthesized compounds, including the assessment of their crystalline and amorphous phases was conducted using X-ray phase analysis. Measurements were carried out at room temperature on TD-3500 X-ray diffractometer.

The results of X-ray phase analysis of unmodified samples and sulfonation products of TPDA, BA, AA modified PFO are given in Figures 3.5-3.6.

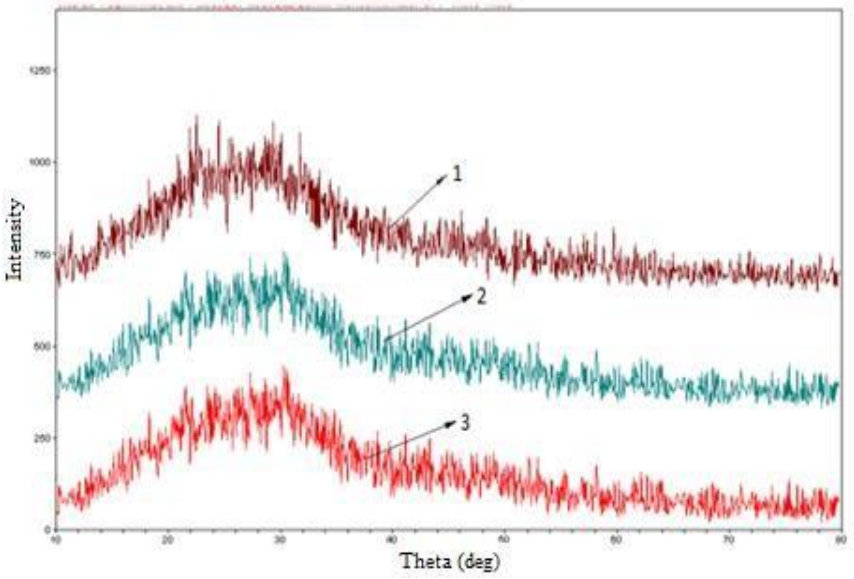


Figure 3.5. X-ray diffraction patterns of sulfonation products of BA (1), AA-modified (2) and unmodified (3) PFO

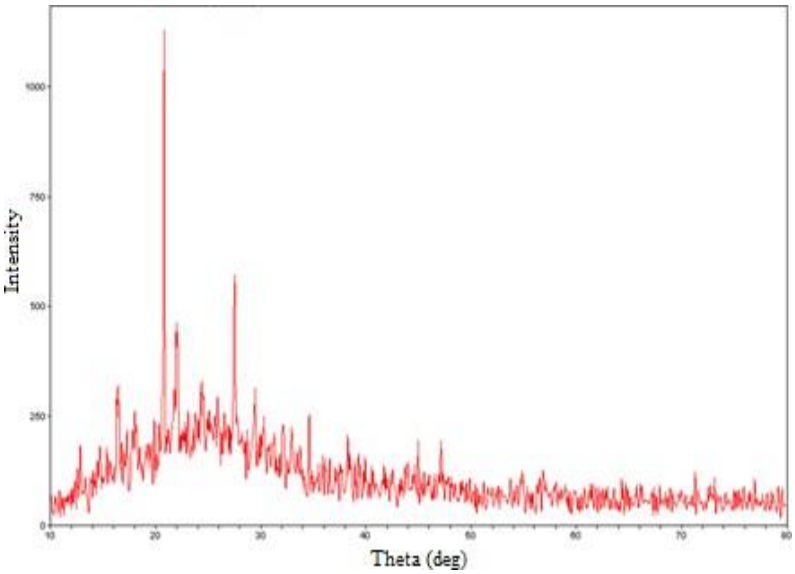


Figure 3.6. X-ray diffraction pattern of a sample of the sulfonation product synthesized from TPDA modified PFO

As illustrated in the figures, no sharp peaks are observed in the X-ray diffraction patterns of the unmodified samples and the sulfonation products of MPFO with BA and AA. This absence can be explained by the fact that the structure of these compounds is amorphous in nature. There are no structured parts, and no lattice formation is visible. This is expected for a sample that does not contain nitrogen. Since the formation of ionized fragments is statistically unlikely.

Investigation of the mechanism of the process suggested that alternative outcomes were possible as such states could have occurred in the other two nitrogen-containing samples. However, as demonstrated, there is no significant difference, the structures are amorphous in nature. A noticeable difference is observed only in the TPDA modified PFO-based sulfocationite samples.

Table 3.3 presents the main parameters of unmodified and MPFO-based sulfocationites. These parameters were also compared with the parameters of the industrially produced KU-1 cation exchanger for comparison purposes.

Table 3.3

Main indicators of sulfocationites

No.	Sulfocationites	Functional group	Particle size, mm	Spill volume, g/ml	Swelling rate, %	Specific volume, ml/g	Static change capacity, mg-eq/g	True density, kg/m^3	Dynamic change capacity, mg-eq/g
1	KU-1 cationite	–SO_3 –OH	0.4-2	0.74	0.19	3.2	1.35	1096.4	0.56
2	PFO-based sulfocationite	–SO_3 –OH	0.8-2	0.71	0.18	3.0	2.10	1082.2	0.82
3	AA-modified PFO-based sulfocationite	–SO_3 –OH >NH >CO	1-2	0.60	0.16	8.0	2.84	1188.6	0.9 8
4	BA-modified PFO-based sulfocationite	–SO_3 –OH >NH >CO	1-2	0.62	0.14	8.4	2.96	1189.4	0.98
5	OA-modified PFO-based sulfocationite	–SO_3 –OH >NH >CO	1-2	0.52	0.15	8.4	3.6	1192	0.96
6	TPDA-modified PFO-based sulfocationite	–SO_3 –OH >NH >CO	1-2	0.52	0.17	8.6	3.65	1194.6	0.98
7	KA-modified PFO-based sulfocationite	–SO_3 –OH >NH >CO	1-2	0.58	0.15	8.0	3.24	1186.8	0.96

The results show that the functionality of MPFO with organic compounds containing amide groups differs compared to unmodified PFO.

Although no significant differences in particle size were observed, the bulk volume was found to be smaller in the nitrogen-containing oligomer samples. This difference was more pronounced in oligomesr modified with nitrogen compounds containing two amide groups. The sulfonation process involves the introduction of new, highly polar functional groups, leading to the formation of irregular hydrogen bonds within the macromolecule and the formation of pore structure.

The slight decrease in swelling degree in nitrogen-containing sulfocationite samples is explained by the increase in functionality and density.

Some differences were observed in the specific volume indices of the samples. The specific volume of sulfonation products of functionalized oligomers was higher than that of unmodified sulfocationite. It is explained by the increased polarity of macromolecules resulting from the introduction of amide groups, the enhanced degree of methylation and sulfonation, as well as the subsequent influence of water dipoles.

One of the important indicators of sulfonic acid cations is their static exchange capacity. The results show that samples modified with organic compounds containing amide groups have higher indicators compared to their analogues. This is explained by the positive effect of the large number of amide groups on the ion exchange capacity.

Dynamic exchange capacity is one of the main indicators characterizing the maximum amount of ions absorbed per unit mass under filtration conditions through an ion exchange layer. Research results show that the sulfonation products of modified oligomers demonstrate an advantage of about 20% over their analogues, which is also observed in the actual density indicators.

In general, the following final conclusions can be drawn from the above analyses:

– After sulfonation of MPFO with organic compounds containing one or two amide groups, it exhibits high cation exchange

properties compared to similar transformation products of the unmodified oligomer;

– The presence of amide groups in oligomers have a positive effect on the corresponding transformations, increasing the reactivity of sulfonation products of oligomers and the polarity of macromolecules, thereby enhancing the attraction of positively charged particles in the system. It is observed that this effect is further enhanced with the increase in amide groups;
– The results obtained confirm the hypothesis that oligomers modified with amide group compounds will be effective as ion exchangers (cation exchangers);
– Based on the results of the conducted scientific research, it can be noted that the physicochemical indicators of the sulfocationites obtained are more effective than the existing KU-1 sulfocationite, and its use is recommended for various purposes.

To reveal the possibilities of using sulfocationites in aggressive environments, the resistance of selected samples (based on AA and BA modified PFO) to HCl and HNO_3, as well as 5% NaOH solutions, was studied, and the results are given in Table 3.4. The studies were conducted at a temperature of 25^0C and for 2 days.

Table 3.4

MPFO-based sulfocationites based on acetamide and benzamide to aggressive environments (temp.-25^0C, time-2 days)

No.	Compound	Environment	Static change capacity, mg-eq/g		The difference in change, %	Mass loss, %
			in the water	in the environment		
1	AA modified PFO based sulfocationite	HCl	2.84	2.69	94.7	2.7
2		HNO_3	2.84	2.53	89.1	4.2
3		NaOH	2.84	2.28	80.2	6.1
4	BA modified PFO based sulfocationite	HCl	2.96	2.76	93.2	2.6
5		HNO_3	2.96	2.57	87.0	3.9
6		NaOH	2.96	2.37	79.9	5.7

The results of the conducted studies show that AA and BA based MPFO-based sulfocationites exhibit considerable chemical resistance to aggressive environments. Both samples demonstrate higher

resistance in acidic environments (5% HCl and HNO_3 solutions) retaining the bulk of their ion exchange capacity. In alkaline environments (in 5% NaOH solution) However, a certain degree of loss and a decrease in static exchange capacity are observed, which can be explained by partial destruction of the structure. Comparative analysis reveals that the BA modified PFO-based sulfocationite exhibits higher stable results in aggressive environments. In general, the results obtained indicate that the use of sulfocationites in aggressive environments can be considered promising as ion exchange materials.

4. MPFO based sulfocationites with amide group compounds in water softening

To reduce the hardness of technical water, MPFO-based sulfocationite samples modified with amide group compounds were used. Water hardness is determined by the trilonometric method. A dark blue chromium indicator was used to determine the total hardness, and muroxide ($C_8H_8N_6O_6$ – ammonium purpurate was used to determine the hardness caused by calcium ions. The experimental setup for the water hardness is shown in Figure 4.1.

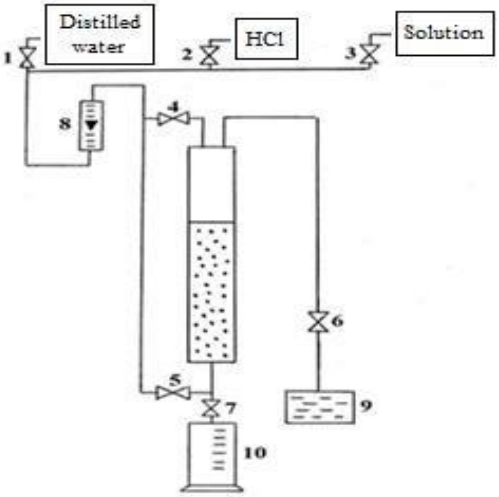


Figure 4.1. Water hardness determining device

1-7 – drawers, 8 – consumption measuring, 9 – washing water for special bowl,10 – softened water for size container

Samples of MPFO-based sulfocationite with an amide group compound were tested. For this purpose, two different hard water samples with different total hardness (8.5 and 10 mg-eq/l) were used, and a 0.01N Trilon-B solution was used during titration. The water was filtered through the device and analyzed , and the results are given in Table 4.1.

Table 4.1

Average hardness value of unmodified and softened water using KA modified PFO-based sulfocationite

Hard water samples	Unmodified PFO-based sulfocationite		KA modified PFO-based sulfocationite	
	Total hardness	Ca^{2+} hardness	Total hardness	Ca^{2+} hardness
No. 1 Total hardness=8.5; Ca^{2+}=4	4.76	1.53	0.47	0.15
No. 2 Total hardness=10; Ca^{2+}=4	0.25	0.13	0.06	0.03

In the next stage of the research, the dynamic ion exchange capacity (the amount of ions that can be captured per unit volume of sulfocationite) was calculated in the process of softening various model coarse water samples with KA modified PFO-based sulfocationite.

Similar experiments were conducted with other amide group compounds in the presence of MPFO-based sulfocationites, and the results are reflected in Table 4.2.

Based on the positive results of the conducted studies, the possibility of softening technical water in the presence of MPFO-based sulfocationites with amide group compounds has been revealed and is recommended for use forpractical application. The synthesized sulfocationite samples were tested by AQUAPRO LLC with the participation of employees of the “Energy Production Technologies” and “Technology of Organic Substances and High Molecular Compounds” departments of ASOIU and confirmed with the relevant test report.

Table 4.2

Cation exchange performance of MPFO-based sulfocationites with amide group compounds

No.	Sulfocationites	Full dynamic ion-exchange capacity, mg-eq/m^3	Static exchange capacity, mmol/g	Residual water hardness, mkg-eq/dm^3
1	PFO-based sulfocationite	175.1	2.10	16
2	AA modified PFO based sulfocationite	225.1	2.84	21
3	BA modified PFO based sulfocationite	229.4	2.96	21
4	OA modified PFO based sulfocationite	235.3	3.60	22
5	TPDA modified PFO based sulfocationite	242.5	3.65	22
6	KA modified PFO based sulfocationite	241.3	3.24	20

Thus, softened water has a positive effect on solving many problems in everyday life, industry and agriculture. The use of softened water in heating systems prevents the formation of scale, which leads to approximately 10% energy savings and a reduction in overall heating costs.

4. Determining the quality of new sulfocationites using fuzzy logic

In recent times, in addition to classical logic approaches in the analysis of technological processes, there has been growing scientific interest in methods based on imprecise and fuzzy data.

The TOPSIS method is one of the widely used methods in multi-criteria decision making and is distinguished by its simplicity of application. This method enables to determine the alternative that is closest to the positive ideal solution and the furthest from the negative ideal solution.

Fuzzy logic theory allows for more effective results when analyzing uncertain and imprecise data. This approach yields more

accurate and reliable results in the assessment of the physicochemical stability of sulfocationites.

In the study, the quality of MPFO modified with amide containing organic compounds was evaluated using a fuzzy logic. For comparative analysis PFO-based sulfocationites were also assessed. The evaluation was based on the indicators presented in Table 5.1.

Table 5.1

Main indicators of MPFO-based sulfocationites with amide containing organic compounds

No.	Sulfocationites	Static change capacity, mg-eq/g	Dynamic change capacity, mg-eq/g	Swelling rate, %	Specific volume, ml/g	True density, kg/m^3
1	PFO-based sulfocationite	2.10	0.82	0.18	3.0	1082.2
2	AA modified PFO based sulfocationite	2.84	0.9 8	0.16	8.0	1188.6
3	BA modified PFO based sulfocationite	2.96	0.98	0.14	8.4	1189.4
4	OA modified PFO based sulfocationite	3.6	0.96	0.15	8.4	1192
5	TFDA modified PFO based sulfocationite	3.65	0.98	0.17	8.6	1194.6
6	KA modified PFO based sulfocationite	3.24	0.96	0.15	8.0	1186.8

In fuzzy TOPSIS, a fuzzy decision matrix is initially constructed. Fuzzy numbers are used to represent the ranking of alternatives for each criterion. The fuzzy variables are expressed by triangular fuzzy numbers A=(l, m, u). The objective of the study is to identify the optimal sulfocationite from the available alternatives.

Initially, the decision-making problem and evaluation criteria are defined. Weights (ω)are assigned to each criterion according to their relative importance. Then, using fuzzy values, each alternative is evaluated for each criterion.

Table 5.2

Criteria weights and initial prices of alternatives

Weights (ω)		ω_1			ω_2			ω_3			ω_4			ω_5		
		0.1	0.4	0.7	0.15	0.3	1	0.08	0.15	1	0.05	0.1	0.6	0.01	0.05	1
Criteria / Alternatives (Sulfocationites)		C1			C2			C3			C4			C5		
		Static change capacity, mg-eq/g			Dynamic change capacity, mg-eq/g			Swelling rate, %			Special volume, ml/g			True density, kg/m ^{3}		
		l	m	you	l	m	you	l	m	you	l	m	you	l	m	you
A1	PFO-based sulfocationite	1	2.1	3.5	0.6	0.82	4	0.02	0.18	1	1	3	5	960	1082	1200
A2	AA modified PFO-based sulfocationite	1	2.84	4	0.5	0.98	3	0.05	0.16	2	6	8	10	1000	1189	1200
A3	BA modified PFO-based sulfocationite	1.5	2.96	5	0.2	0.98	2	0.08	0.14	2	5	8.4	11	950	1189	1250
A4	OA modified PFO-based sulfocationite	2	3.6	6	0.6	0.96	3	0.06	0.15	2	3	8.4	10	920	1192	1300
A5	TPDA modified PFO-based sulfocationite	1	3.65	4	0.1	0.98	3	0.01	0.17	1	2	8.6	12	800	1195	1350
A6	KA modified PFO-based sulfocationite	2	3.24	5	0.5	0.96	2	0.07	0.15	2	5	8	10	850	1187	1270

The number of alternatives (A) in the fuzzy decision matrix is 6 and the number of criteria (C) is 5. The alternatives are A1–PFO-based sulfocationite, A2–AA-modified PFO based sulfocationite, A3–BA-modified PFO based sulfocationite, A4–OK-modified PFO based sulfocationite, A5–TPDA-modified PFO based sulfocationite and A6–KA-modified PFO based sulfocationite. The criteria are selected as C1–static exchange capacity, C2–dynamic exchange capacity, C3–swelling ratio, C4–specific volume and C5–true density (Table 5.2).

The solution to the problem was implemented in stages and the following result was obtained (Table 5.3).

Table 5.3

Rank of alternatives

Result			
Ai	**Rank**	**Alternatives**	
0.193766	6	**A1**	PFO based sulfocationite
0.525486	3	**A2**	AA modified PFO based sulfocationite
0.497	4	**A3**	BA modified PFO based sulfocationite
0.746828	1	**A4**	OA modified PFO based sulfocationite
0.571232	2	**A5**	TFDA modified PFO based sulfocationite
0.47763	5	**A6**	KA modified PFO-based sulfocationite

Thus, based on the ranking of sulfocationites, the A4-alternative (OA modified PFO-based sulfocationite) has the highest index of 0.746828 compared to the others.

CONCLUSIONS

The main scientific results obtained in the dissertation are as follows;

1. For the first time, the sulfonation process of thermoreactive PFO modified with organic compounds with amide groups - AA, BA, OA, KA, TPDA - was carried out. Optimal conditions were determined by mathematical modeling: oligomer to acid - 1:3 molar ratio, temperature of 135^0C, reaction time of 2-3 hours [16].
2. The structure of MPFO-based sulfocationites modified organic compounds containing an amide group has been studied. The reaction mechanism by the addition of sulfo groups ($–SO_3H$) to the benzene ring has been confirmed [5, 6, 8, 13, 15].
3. The cation exchange properties of the synthesized sulfocationites were studied and compared with unmodified oligomer-based and KU-1 sulfocationites. It was found that the main indicators of nitrogen-containing sulfocationites were higher [5, 6, 8, 13, 15].
4. According to the results of Laser Diffraction analysis, the most effective sample in terms of adsorption was the oxamide-based MPFO sulfocationite. This sulfocationite is distinguished by a larger surface area and a high adsorption kinetics.
5. High thermal stability of sulfonation products of MPFO with organic compounds containing amide groups was determined. The residual mass at ~900^0C in the oligomer sample modified with TFDA was 16%, and in the oligomer sample modified with BA was 15% [17].
6. Studies of sulfonation products of MPFO with organic compounds containing amide groups have shown t their stability for use in aggressive environments [3].
7. Tests of MPFO-based sulfocationites as cation exchangers have been conducted in the softening of hard water yielding positive results [7, 14, 19].
8. The degree of proximity to the ideal and non-ideal solutions was evaluated using the fuzzy TOPSIS method, it was determined that

the A4-alternative (OA modified PFO-based sulfocationite) had the highest index of – 0.746828.

The main content of the dissertation work is reflected in the following scientific works:

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