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Synthesis and Investigation of Amino-carboxy-sulfonate Amphoteric Ionite Obtained by Methods of Transformation and Modification of Polymers

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Abstract A comprehensive approach has been developed to solve the problem of using a secondary product of fat-and-oil production and secondary polymer materials as resource-saving raw materials in the creation of new functional ion-exchange materials. Alternative technological solutions for the trasformation and modification of the developed ion–exchange materials are proposed in order to obtain amino–carboxy-sulfonate ionite sorbent by introducing additional strong acid sulfogroups or by introducing spent (in water treatment processes) cationite into the matrix as a functional component. The modified amphoteric ionite, which has a functional component in its composition – spent «Ky-2-8» cationite, was studied by scanning electron microscopy (SEM) in order to study the structure of the nanostructure and microstructure of the surface and inner region of ion-exchange materials. Amphoteric ionite, which has a functional component in its composition, has predominantly (≤ 26%) the best exchange capacity values for 0.1 n. NaOH solution is 5.2–5.4 mg-eq/g in comparison with an unmodified ion-exchange material.

Keywords Ionite, Sulfonation, Modification, Scanning electron microscope, «Ky-2-8», Ion exchange matrices, Polyethylenepolyamine, Furfural, Oleum, Olein-palmitic fraction of cotton soapstock

1. Introduction

A rational approach to the treatment of secondary products and industrial waste, as well as consumer products, is to maximize their processing into other products with increased added value.

The analysis of scientific and technical literature on waste from the fat and oil industry and secondary polymers, namely secondary polyethylene terephthalate, predetermined the development of a new technological direction for the integrated use of secondary raw materials of cottonseed oil, namely olein-palmitic fraction/mixture and chemically processed secondary polyethylene terephthalate as the most acceptable secondary products for use in the production of ion exchange materials and inhibitory compositions.

The choice of these secondary products is justified by the fact that a huge amount of plastic waste, including in the form of recycled polyethylene terephthalate (PET containers), partially lost their properties, but retained their technological properties, make it possible to recycle them by chemical modification for reuse as feedstock to obtain new types of chemical products – ion exchange materials and inhibitory compositions.

Considering that PET containers decompose in natural conditions for centuries, during this time solid waste will accumulate in huge quantities, polluting and clogging the planet.

Another class of unrealized polymers and processing technology for which has not been developed are secondary products of mesh (crosslinked) polymers, namely spent synthetic ionites. Used synthetic ionites have been unclaimed to date and have not been considered as secondary raw materials. However, such properties of spent ionites as a sufficiently high residual sorption capacity, the identity of the chemical composition of newly developed ion exchange materials indicate the prospects of their use as a functional component in the production of various types of ionites and sorbents.

In the production of cottonseed oil and fatty acids, depending on the technological scheme and methods of separating the main products, many secondary products and waste are formed, the processing of which plays an important role. In our research, the olein-palmitic fraction/mixture of cotton soapstock will be investigated and used.

The aim of the research is to provide alternative technological solutions for the modification of ion-exchange materials in order to obtain an amino-carboxy-sulfonate

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polymer by methods of transformation of strongly acidic sulfogroups and modification by introducing regenerated spent strongly acidic cationite «Ky-2-8» into the matrix as a functional component.

The research is aimed at:

Firstly, the secondary use of polymer waste and secondary products of fat-and-oil production in order to obtain new types of ion-exchange materials, which is important from an environmental point of view and from the perspective of rational use of raw materials.

Secondly, it is aimed at a comprehensive solution of technological issues aimed at the development and creation of polymer – polymer ion-exchange materials for multifunctional purposes with high functional and operational characteristics of application.

Comprehensive studies of the regularities of the synthesis of ion–exchange matrices in the presence of spent ionites as polymer-dispersed nanomaterials have been carried out, the features of their structure formation and the analysis of the interrelationships of the structure and properties of ion-exchange materials have been revealed. For the first time, the technological foundations for the production of filled ion-exchange materials for multi-purpose purposes are formulated. Since the use of spent ionites depends on the dispersion, the required degree of dispersion was determined taking into account the direction of ionite use.

When synthesizing ion-exchange matrices, the complex structure of ion-exchange materials should be taken into account, including gel sections in which the polymer itself and spent ionites represent separate phases. On this basis, the gaps in the gel regions are classified as micro- and mesopores, where functional groups of spent ionites are also localized on the walls of these pores.

The increasing consumption of ionites by various industries requires the development and improvement of methods for obtaining alternative ion exchange materials through previously unknown or little-known technological solutions for the creation of promising new generation ionites.

The structures of nanostructures and microstructures of ion-exchange materials have been studied, as well as their physico-mechanical and technological properties have been studied. Currently, electron microscopy is the main direct method of studying the structure of nanostructures and microstructure of ion-exchange materials. Its main advantages over other microscopy methods are: direct instantaneous image formation, a wide range of easily variable magnifications, a large depth of focus at high resolution, the possibility of diffraction examination (and, consequently, obtaining a variety of information about the internal structure of an ion-exchange material and its state: structure, ordering, porosity, etc.), the possibility of microrentgenospectral (elemental) analysis, etc.

The research has undertaken a theoretical study of the mechanism of the chemical reaction of an «АНФ» by polycondensation of styrene, furfural in the presence of polyethylenepolyamine (PEPA) and its subsequent sulfonation [1-4].

Alternative technological solutions for the creation of ion-exchange materials are proposed, which are considered in two directions: first, the introduction of additional strongly acidic sulfogroups ($–SO_3H$) to amphoteric ionites, which already have weakly acidic carboxyl groups (–COOH) and weakly basic tertiary and secondary amino groups ($–N^+R_2H$ and $–N^+RH_2$) in their structure [5] in order to obtain amino-carboxy-sulfate amphoteric ionite with improved exchange capacity by cationite; secondly, modification of the developed ion-exchange materials by introducing regenerated spent synthetic ion-exchange materials of any type into the matrix, depending on their purpose and application.

Technological solutions for the synthesis of amino-carboxy -sulfonate ion exchange material obtained by methods of transformation of sulfo groups and modification of polymers by introducing a functional component up to 20 wt.% make it possible to obtain new ion-exchange materials with amphoteric properties and improved exchange capacity values for cationic groups.

In connection with the above, we have previously obtained a number of ion-exchange materials and studied their basic physico-chemical and sorption properties [6-9].

2. Main Body

2.1. Explanation of Methods

Amino-carboxy-sulfonate amphoteric ionite AR&ISM-12 was obtained by the transformation method. At the first stage, polycondensation of the olein-palmitic fraction/mixture of cotton soapstock [10] with a secondary product of polymer products (SPPP), PEPA and furfural produced amphoteric ionite containing carboxylic, secondary and tertiary amino groups. Amphoteric ionites with sufficiently good properties were obtained at a mass ratio of olein palmitic fraction of cotton soapstock (OPF CS) to SPPP, PEPA and furfural 0.5:0.5:1.0:1.0 and a temperature of 90-110℃, conventionally designated AR&ISM-10 with exchange capacities EC, mg-equiv/g of 0.1 n. NaOH solution - 4.0; 0.1 n. HCl solution – 4.9.

Similarly, the polymer matrix AR&ISM-11 was obtained by chemical interaction of OPF CS with SPPP and furfural 0.5:0.5:1.0, that is, on the basis of a three-component system acceptable for functionalization by sulfonic acid groups.

At the second stage, sulfonation of a three-component polymer matrix AR&SM-11 pre-swollen in a sulfonating agent was carried out. To carry out sulfonation of AR&ISM-11, 70-98% sulfuric acid, oleum with different SO_3 content were used. The mass ratio of the sulfonating agent to ionite was 5:1; the sulfonation time was 4-8 hours, the temperature was 70-110℃. The resulting amino-carboxy-sulfonate amphoteric ionite AR&ISM-12 was purified from unreacted starting materials by sequential washing with 5% solutions of $HC1 \rightarrow H_2O$ distilled water, $NaOH \rightarrow H_2O$ distilled water to a neutral reaction of washing waters and polymer granules were dried to an air-dry state.

At the third stage, the modification of the developed ion exchange materials AR&ISM-10 was carried out by introducing the purified strongly acidic cationite «Ky-2-8» (cationite used in water treatment processes) into the matrix as a functional component in order to obtain modified amphoteric ionite AR&ISM-13.

Amphoteric ionites with sufficiently good properties were obtained at a mass ratio of OPF CS: «Ky-2-8» to SPPP, PEPA and furfural 0.5:0.5:0.5:1.0:1.0 and a temperature of 80-90°C, conventionally designated AR&ISM-13 with an exchange capacity of 0.1 n. NaOH solution (cationic groups), which have indicators 5.2-5.4 mg-eq/g, of which for carboxyl groups (SEC_{COOH}) 3.1-3.2 mg-eq/g; for sulfogroups (SEC_{SO3H}) 2.1-2.2 mg-eq/g.

2.2. Results and Discussion

Considering that during sulfonation, the reaction rate decreases as a result of dilution of sulfuric acid with water formed by the reaction, and the substitution reaction eventually stops, an excess of sulfuric acid or oleum was used in studies to shift the sulfonation equilibrium to the right side.

Sulfonation of the cured polymer matrix (resin) AR& ISM-11 with sulfuric acid (d=1.84 at 5 times the weight excess) for 10 hours produced ionite with an exchange capacity of 0.1 n. NaOH solution is 0.5-0.7 mg-equiv/g higher than the exchange capacity of AR&ISM-10. The next stage of the experiments should be carried out using 5% oleum as a sulfonating agent. The resulting amino-carboxy-sulfonate amphoteric ionite, conventionally designated as – AR&ISM-12, has an exchange capacity of 0.1 n. NaOH solution – 5.1-5.2 mg-eq/g. The data characterizing the dependence of the exchange capacity on the sulfonation conditions at a weight ratio of 5% oleum: AR&ISM-11 – 5:1 is shown in Figure 1.

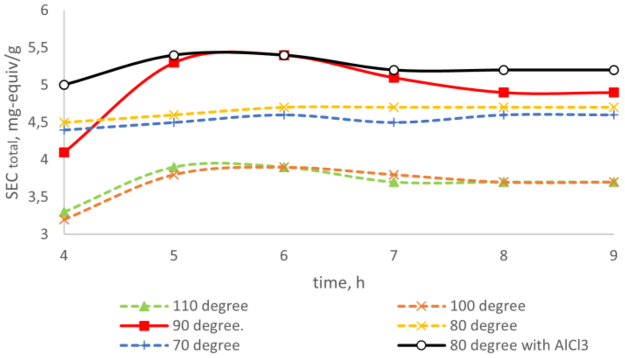


Figure 1. Dependence of the exchange capacity on the temperature and time of sulfonation

The sulfonation processes were investigated and it was found that an increase in the duration of sulfonation of ionite with 5% oleum from 4 to 8 hours at 100-110°C leads to a change in the fractional composition and mechanical strength of ionite. Sulfonation at such temperatures leads to a decrease in the mechanical strength of the ionite and to a decrease in the total exchange capacity of ionites by cationic groups.

It was revealed that the optimal processes of sulfonation of ionites with 5% oleum are: a) the duration of sulfonation is 5-6 hours at a temperature of 90°C; b) the duration of sulfonation is 4 hours at a temperature of 80°C with the presence of a catalyst in the reaction mixture – $AlCl_3$ ($\leq$ 0.5%) to achieve the appropriate conversion. As can be seen from the graph data, the introduction of anhydrous $AlCl_3$ into the reaction mixture as a catalyst and due to the corresponding conversion, the value of the exchange capacity is slightly improved.

The exchange capacity of the considered amphoteric ionite during sulfonation with an excess of oleum (5:1) in the presence of $AlCl_3$ for 4 hours at 80°C is shown in Figure 2. The introduction of $\geq$ 0.5% $AlCl_3$ into the reaction mixture slightly reduces the value of the exchange capacity, including the exchange capacity for both sulfogroups (SEC_{SO3H}) so is the total exchange capacity (SEC_{total}).

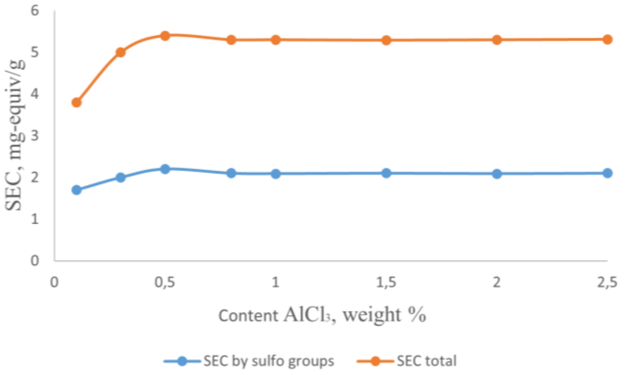
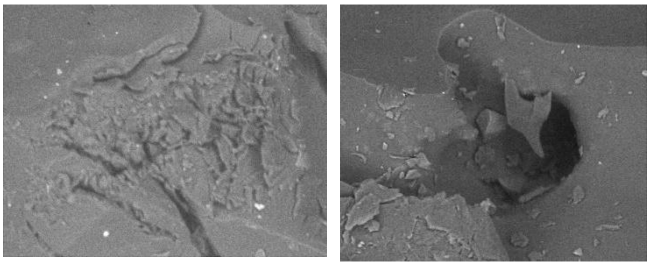


Figure 2. Dependence of the exchange capacity on the amount of catalyst during sulfonation for 4 hours at 80°C

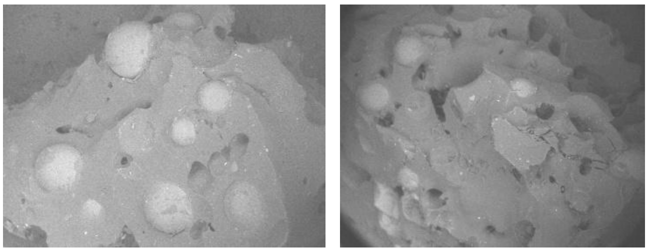
A study of the physico-chemical properties and exchange capacity of modified AR&ISM-13 ionite showed that the introduction of regenerated «Ky-2-8» cationite into the AR&ISM-10 matrix makes it possible to purposefully increase the EC by 0.1 n. NaOH solution (cationic groups), which have indicators of 5.2-5.4 mg-equiv/g, of these, by carboxyl groups (SEC_{COOH}) 3.1-3.2 mg-equiv/g; by sulfogroups (SEC_{SO3H}) 2.1-2.2 mg-equiv/g; while maintaining the performance characteristics of ionite.

Modified amphoteric ionite AR&ISM-13 has a high exchange weight (0.5-0.6 g/cm^3), low swelling in water and organic solvents, the volume increase during swelling in water does not exceed 3-4% of the volume of dry ionite. The low swelling is a consequence of the initially developed pore surface of the inner part and the amphoteric ionite itself under study, while sorption and exchange reactions of the ionogenic groups of the ionite itself and «Ky-2-8» cationite located in the ionite structure are activated.

The humidity of the modified ionite under study is in the range of 10-15%, and the loss of ionite capacity in H-form after heating in air at 160°C for 24 hours is 8-10%. The modified amphoteric ionite AR&ISM-13, which contains a functional component – «Ky-2-8» cationite used in water treatment processes, was studied by scanning electron microscopy (SEM), which is the main direct method for studying the structure of the nanostructure and microstructure of the surface and inner region of ion-exchange materials [11-12].



a) Ionite AR&ISM-10 (x230) b) Ionite AR&ISM-13 (x230)



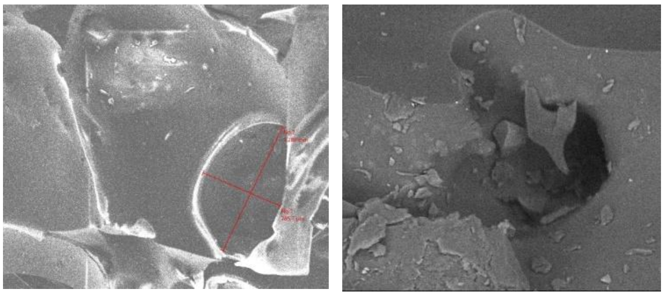
c) Ionite AR&ISM-13 (x35)

Figure 3. Electron microscopic image of the ionite structure (magnification x230)

Scanning of the surface and inner region of the modified amphoteric ionite was carried out using a scanning electron microscope (SEM) of the JEOL JSM-IT200LA brand (Japan). SEM mapping of the studied amphoteric ionites in reflected electric current rays showed that the internal structure of [13] particles (irregularly shaped granules) are almost the same, no crystalline inclusions were detected. The difference in granules is manifested in the porosity of the unmodified ionite (Figure 3a) and in the regular change in the structure of the modified amphoteric ionite AR&ISM-13, which has a functional component – «Ky-2-8» (spent), which has a changed shape of granules having particles with chips, cracks on the surface with a certain dispersion (Figure 3b-3c).

The use of SEM in the study of various phenomena related to the destruction of ionites, especially the study of the process of the appearance and development of cavities inside individual ionite granules is very informative [14].

The following types of destroyed granules of the investigated amphoteric ionite were studied (Figure 4): a) thin-walled shells with a hole connecting the inner cavity of the granules with the external environment; b) thick-walled shells, where the process of formation of internal cavities has just begun and which in appearance differ little from ordinary ionite granules.



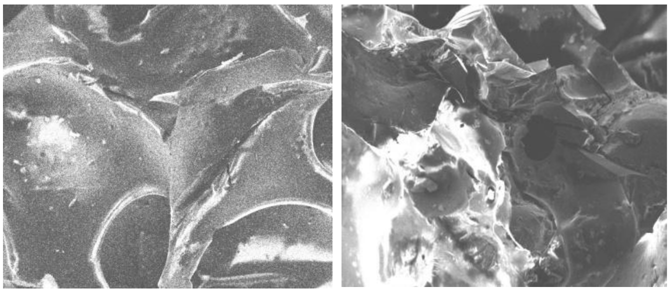
a) b)

Figure 4. Electron microscopic image of the ionite surface structure (x230)

Equally effective is the use of the SEM method to study changes in the structure of ionite granules by their sequential transfer from one salt form to another.

SEM studies of a regular change in the structure of modified amphoteric ionite AR&ISM-13, which has a functional component – «Ky-2-8» (spent), have shown that the transfer of ionite from one form to another does not cause destructive structural changes (Figure 5b), unlike the ion-exchange material AR&ISM-10. So, in the pictures you can see that in AR&IM-10, longitudinal and transverse slits can be observed, as well as a change in the reflectivity of the internal cavities of ionite granules (Figure 5a).

In this regard, based on optical studies by the SEM method for modified amphoteric ionite AR&IS M13, high mechanical strength in regeneration processes can be predicted.



a) Ionite AR&ISM-10 b) Ionite AR&ISM-13

Figure 5. Mapping of synthesized ionites after repeated regeneration (x230)

Polymer-polymer ion-exchange materials (PPIM) were obtained by dispersing a small amount in a polymer matrix (≤ 20 wt.%) of spent ionite (natural nanocomposite), particles that are less than 1.5 mm in size. The creation of PPIM has become one of the most effective ways to develop new types of ion-exchange materials.

Within the framework of the formation of ionite particles, the role of spent ionites on morphology and functional behavior in the system of ion-exchange materials has been experimentally confirmed for the first time.

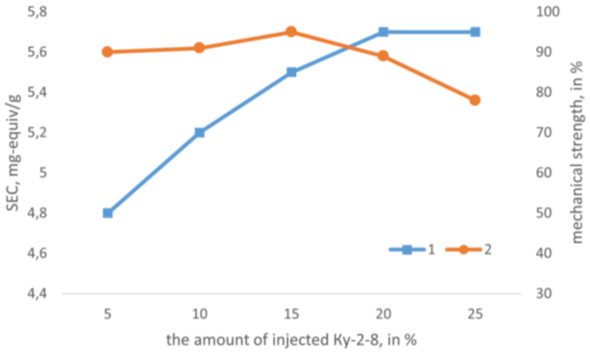


Figure 6. Dependences of EC (1) and mechanical strength (2) of AR&ISM13 on the amount of the introduced modifying reagent

The effect of the amount of the introduced functional component – «Ky-2-8» (spent) on the exchange capacity of AR&ISM-13 ionite (Figure 6) was studied, so the introduction of a functional component up to 20 wt.%. The value of the exchange capacity for cationic groups increases, but affects the mechanical strength of the ionite.

The production of ion-exchange materials with additional porous and microstructural characteristics allowed the development of new efficient ion-exchange materials. The production of polymer-polymer ion-exchange materials allows the use of spent ionites as active functional fillers.

The use of spent ionite particles with a size of ≥ 1.5 mm in the ion exchange matrix contributes to the formation of concentration stresses at the phase interface. This is due to the uneven distribution of spent ionite particles in the ionite structure and the formation of defective zones at the point of contact of spent ionite particles in the ion exchange material structure.

The introduction of particles with a size of ≤ 1.5 mm with an assumed uniform distribution in the gel structure of the ion-exchange material formed is characterized by a decrease in internal stresses and the presence of a minimum number of defective zones.

Summarizing the results of the study, it can be concluded that the content of spent ionite in the range of 16-18% of the total mass of the ion-exchange material allows, due to the synergistic effect, to achieve satisfactory indicators of exchange capacity and mechanical strength.

Along with the study of the mechanical strength of ionites during regeneration, the thermal stability of amphoteric ionites AR&ISM-10 and AR&ISM-13 was investigated by thermogravimetric method.

The thermal stability of AR&IS-10 and AR&IS M13 ionites was studied on a DTG-60 device manufactured by Shimadzu (Japan). The 5 mg sample was heated from 20 to 800℃ at a heating rate of 10℃/min. The analysis was carried out in an atmosphere of inert gas – argon, at a feed rate of 80 ml/min. Powdered aluminum oxide II (Al_2O_3) was chosen as the reference for comparison. Comparing the mathematical data of thermogravimetric analysis of curves (Figure 7) of ion exchange materials AR&ISM-10 and AR&ISM-13 (Table 1), it can be seen that the temperature of the onset of thermal degradation in AR&ISM-10 and AR&ISM-13 is almost the same, but already with a mass loss of 15% you can see The difference is that the temperature of ionite destruction with a mass loss of 50% differs by about 40-50 degrees.

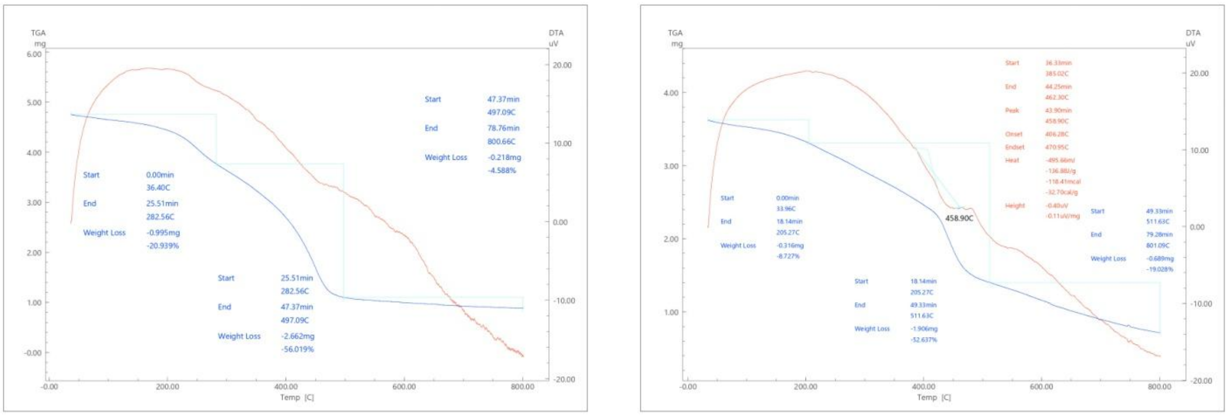
Complete destruction of ion-exchange materials is observed with a mass loss of 65%, at temperatures of 450℃ and 555℃. It follows from the data that amphoteric ionite AR&SM-13 is more thermally stable.

3. Conclusions

- By chemical interaction of the olein-palmitic fraction /mixture of cotton soapstock with SPPP and furfural 0.5:0.5:1.0, that is, on the basis of a three-component system, a polymer matrix acceptable for functionalization by sulfonic acid groups was obtained. The resulting amino -carboxy-sulfonate amphoteric ionite has an exchange capacity of 0.1 n. NaOH solution – 5.1-5.2 mg-eq/g.

Table 1. Results of thermogravimetric analysis of ionite mass loss from temperature

Mass loss (%) from the temperature (℃)														
Ion exchange material AR&ISM-10														
m,%	2	5	10	15	20	25	30	35	40	45	50	55	60	65
T, ℃	76	166	225	250	274	306	336	363	385	404	419	431	441	450
Ion exchange material AR&ISM-13														
m,%	2	5	10	15	20	25	30	35	40	45	50	55	60	65
T, ℃	78	169	227	262	304	347	385	416	432	442	451	463	489	555



a) Ionite AR&ISM-10

b) Ionite AR&ISM-13

Figure 7. TGA and DTA of ion exchange materials

- Modification of previously developed ion-exchange materials obtained by polycondensation of olein-palmitic fraction/ mixture of cotton soapstock with SPPP, PEPA and furfural was carried out by introducing into the matrix the regenerated strongly acidic cationite «Ky-2-8» (cationite used in water treatment processes) as a functional component in order to obtain a modified amphoteric ionite with an exchange capacity of 0.1 N. NaOH solution – 5.2-5.4 mg-eq/g.
- The developed ionite compositions using spent ionites make it possible to reduce the cost of production and obtain ion-exchange materials with the required operational properties (good mechanical strength, thermal stability and exchange capacity).

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