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Granular copolymer synthesis of acrylonitrile and hexahydro 1.3.5-triacryliltriazine and its physicochemical properties

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Abstract: A new anion-exchange granular sorbent was obtained by cross-copolymerization of 1,3,5-triacrylyltriazine with acrylonitrile and its chemical modification with hydroxylamine. In this reaction, hydroxylamine showed a catalytic effect, bringing the static exchange capacity (SEC) of the anion exchanger to 4.7 mg-eq/g.

The influence of the concentration of hydroxylamine, temperature and reaction time on the static exchange capacity (SEC) of the anionite was investigated. Carrying out the amination reaction at 95 ° C for 5 h and increasing the hydroxylamine concentration in the reaction mixture to 10 mass% turned out to be the most optimal conditions for modification.

The potentiometric titration curves showed that the resulting anion exchange had low basic groups. The ionization constant of the functional groups of the anionite (pK = 7.1), found in the graphic solutions of the Henderson-Hesselbach equation, confirms the low basicity of the anionite.

The sorption capacity of the anion exchanger for metal ions from aqueous solutions containing a mixture of Ag+, Pd2+, Pt2+, Cu2+, Sc2+, Ba2+, Sr2+, Ni2+, Co2+, Cd2+, Cr3+ and etc. ions is 10-8 g/l and pH = 5 - 6. It has been established that the new anion exchanger selectively and quantitatively (100%) absorbs Ag+ and Pd2+ ions, which is promising as a selective sorbent for Ag+ and Pd2+ ions.

Keywords: copolymer, modification, anion exchanger, potentiometric titration, sorption.

I. Introduction

It is known that sorption materials are increasingly finding their application in various fields of industry, where it is required to improve the quality of raw materials and products, deep purification of technological solutions. It is important that sorbents used in industry to increase the yield to a high percentage are technologically and economically viable. Accordingly, the problem of synthesizing ion-exchange materials capable of selectively sorbing metal ions from complex technological solutions remains urgent.

The aim of this work is to synthesize new ion-exchange granular sorbents based on a copolymer of acrylonitrile and hexahydro 1,3,5 – triacrylyltriazine, to find the optimal modification parameters, and to study the physicochemical properties of the obtained anionites.

II. Experimental technique

The modification of the copolymer of the porous structure of AN and GTT with hydroxylamine was carried out in a 5% solution of dimethylformamide at 369-371K for 5 hours in a weakly acidic medium in order to partially swell the granules [1, pp.25-30].

The resulting anion exchanger was identified by IR spectroscopy, which confirms the absorption bands of ionogenic groups, in particular, in the regions of 2250, 1650 and 1250 sm $^{-1}$, corresponding to the vibrations of CO, NH, NH_2 bonds.

The acid-base properties of the sorbents were determined by potentiometric titration. To determine the pK functional groups of the ion exchange sorbent, ie to establish the relationship between the degree of their ionization and the pH values of the solution, we used the Henderson-Gesselbach equation [2, p. 110]:

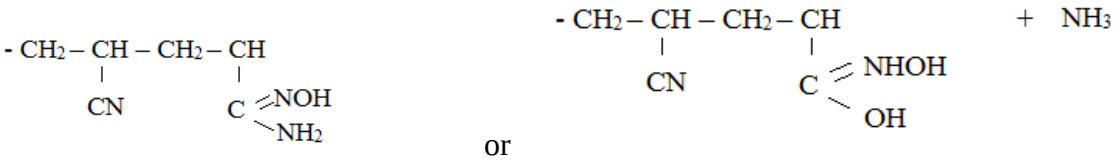
$$pH = pK \pm lg\frac{1-\alpha}{\alpha},$$

where the sign in front of the second term of the equation is positive for bases and negative for acid groups.

The sorption of metal ions was studied by a dynamic method. The initial concentration of elements was 10^{-8} g/l, and the pH of the solution was 5-6. For the quantitative characterization of sorption, the neutron activation method was used, for which the initial solution and the sample of the ion exchanger were irradiated after sorption. The value of the degree of sorption was calculated from the difference in the pulse area of the elements that correspond to certain energy values (in keV).

III. Results and its discussion

It is known that the interaction of cross-linked polyacrylonitrile and granules based on it with hydroxylamine will lead to the production of polyacrylamidoximes or polymeric hydroxamic acids. The modification process can be schematically represented as follows:



or

As a result of the modification reaction in the presence of a catalyst, the SEC of the anionite reaches 4.7 mg-eq/g.

It is known that the development of a new method of anion exchanger requires optimal conditions for the synthesis of his research, which is important both from a technological and economic point of view. To find the optimum synthesis conditions have been studied the effect of various factors (the concentration of hydroxylamine, temperature, reaction time) for static exchange capacity (SEC) ion exchangers, which is the basic measure of the qualitative and quantitative evaluation of the reaction product.

To establish the effect of hydroxylamine concentration on SEC, the modification was carried out in the concentration range from 5 to 16 mass percent of the aminating agent. Figure 1 (a) shows the dependence of the SEC of anion exchangers on the concentration of hydroxylamine.

The results of studying the effect of temperature on the SEC of modified copolymers are shown in fig.2. As can be seen from the data presented in fig.1(b), in the temperature range of 369 K, the SEC value of anionite sharply increases.

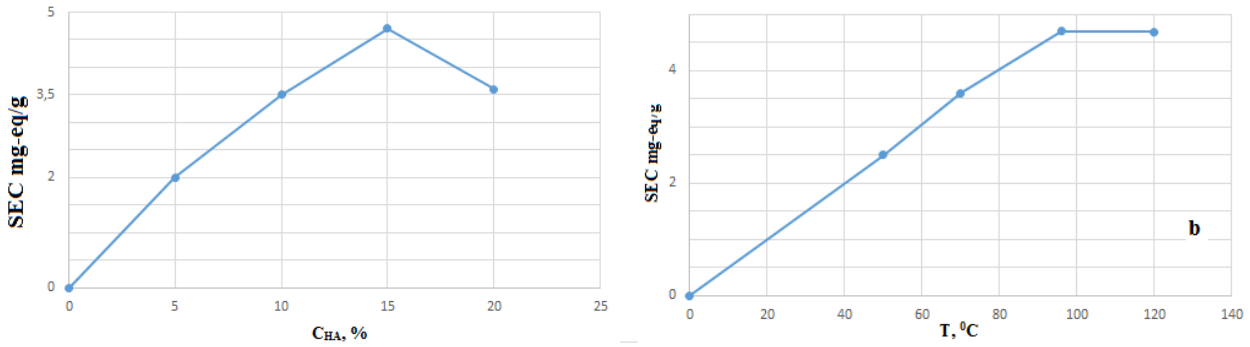


Figure 1. Dependence of the SEC of the anionite on the concentration of HA in the reaction mixture (a) and the process temperature (b)

The effect of the duration of the amination reaction was studied in the range from 2 to 8 hours, the results of which are shown in fig.2.

As can be seen from the data presented in Fig. 2, the dependence curve comes through a maximum with a reaction duration of 5 hours. An increase in the reaction time leads to a decrease in the SEC.

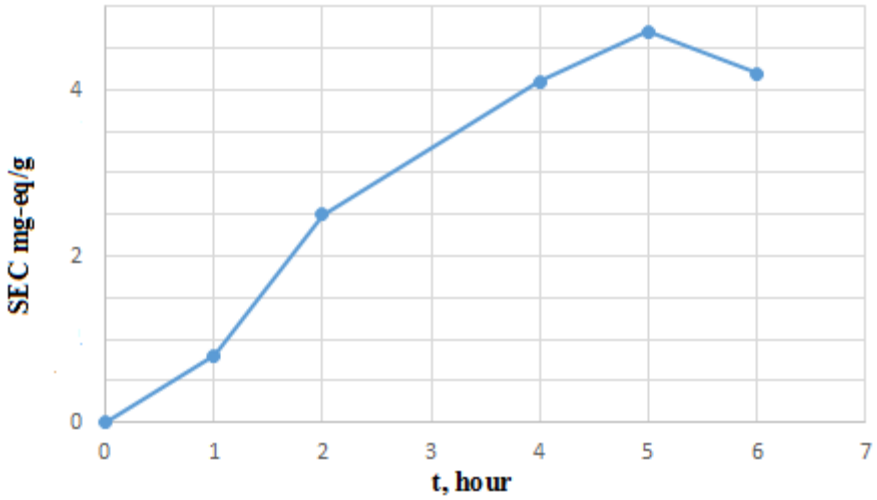


Figure 2. Dependence of the SEC of the anion exchanger on the duration of the amination reaction

As can be seen from the data presented in fig.3. The obtained anion exchanger has a weakly basic character, the pK of which is 7.1

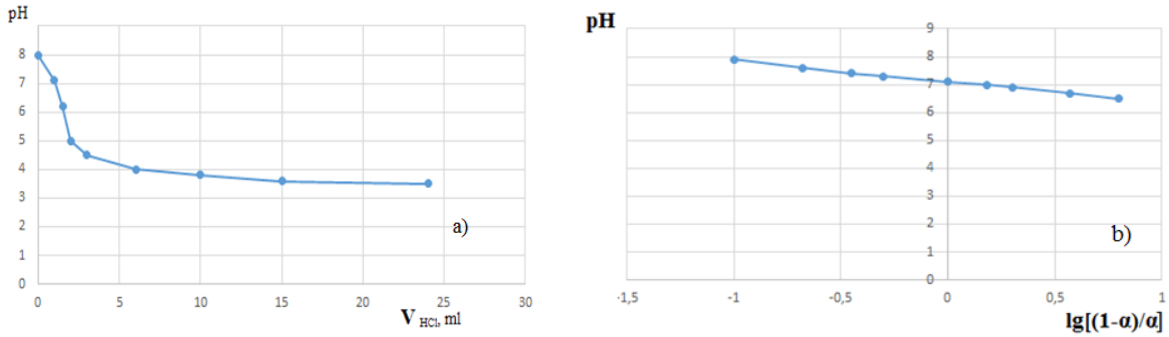


Figure 3. Curves of potentiometric titration of anionite: differential (a) and expressed in the coordinates of the Henderson-Hesselbach equation (b)

The presence of imine and amine groups containing a lone pair of electrons, as well as a spatial structure, imparts complexing properties to the anion exchanger [3]. To clarify the complexing

properties of the obtained anionite, the sorption of metal ions from aqueous solutions containing mixtures of various ions such as Ag^{+}, Pd^{2+}, Pt^{2+}, Cu^{2+}, Sc^{2+}, Ba^{2+}, Sr^{2+}, Ni^{2+}, Co^{2+}, Cd^{2+}, Cr^{3+} and others with an initial concentration of 10^{-8} g/l and pH = 5-6 was studied. It was found that the new anion exchanger selectively and quantitatively (100%) absorbs Ag^{+} and Pd^{2+} ions, which has prospects as a selective sorbent for Ag^{+} and Pd^{2+} ions.

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