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RADICAL COPOLYMERIZATION OF N-MORPHOLIN-2-CHLORO-ISOPROPYL ACRYLATE WITH ACRYLIC ACID AND KINETIC PARAMETERS OF SPONTANEOUS POLYMERIZATION.

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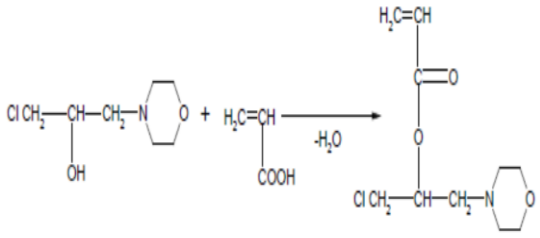
Abstract: The kinetics of the synthesis process is studied and the radical copolymerization of N-morpholine-2-chloro-isopropyl acrylate with acrylic acid in organic solvents is considered. It was found that in all cases the copolymers are enriched with N-morpholine-2-chloro-isopropyl acrylate (MHIPA) units. The use of MHIPA as a comonomer leads to an increase in the copolymerization rate and intrinsic viscosity of the studied copolymers. The urgency of the research problem in the indicated directions, the current state and solutions are shown.

Keywords: morpholine, diethylene glycol, kinetics, simulation, chemical process

Introduction

Polymers containing functionally active groups have a wide range of biomedical activity and are used as bactericides, fungicides, and pharmaceuticals. Most often, to obtain such polymers, monomers of the vinyl and acrylic series containing carbonyl and amino groups in the structure were used.

In order to synthesize potentially biologically active monomers, N-morpholine-3-chloro-2-propanol was esterified with acrylic acid and the reaction can be represented by the following scheme:



Acrylic acid, N-morpholine-2-chlorisopropyl acrylate, DMF, ethyl alcohol were distilled before use, and drying was carried out according to standard methods. The initiator, trinitrile of azobisisobutyric acid (TAC), was recrystallized from a solution in absolute ethanol and dried in a vacuum desiccator to constant weight.

Main part

The studies were carried out by the gravimetric method. This method is based on the separation of the polymer from the reaction medium by precipitating it into solvents that dissolve the monomer and do not dissolve the polymer. The polymer is isolated in the form of a precipitate, which is washed with a precipitant, and then dried to constant weight and weighed on an analytical balance. The rate of reaction is determined from the degree of conversion of the monomer at a given point in time. From the values of the obtained polymerization rates at different temperatures, the rate constants are determined, and from the temperature dependence of the polymerization rate constants in Arrhenius coordinates, the total activation energy of polymerization is estimated. The copolymerization reaction was

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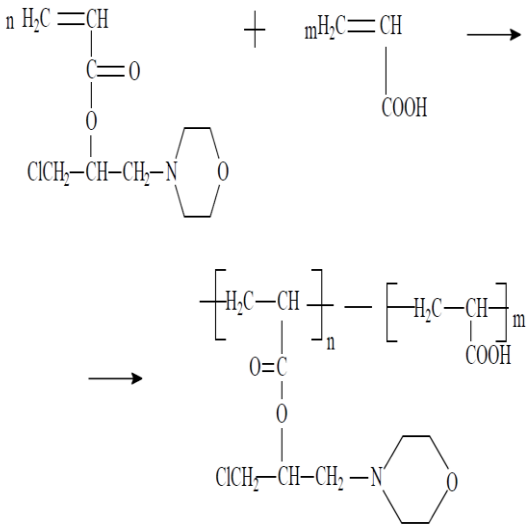
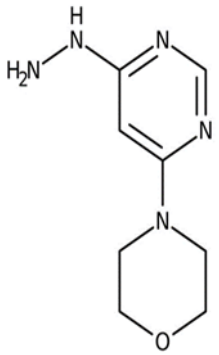
carried out at a temperature of 50-90 ° C in the presence of a radical initiator TAK in an organic solvent. The copolymerization reaction was carried out to a degree of conversion of 15-17%, which was determined upon reaching a syrupy mass. It was found that in different ampoules the copolymerization rate is different - the reaction rate increases with an increase in the content of N-morpholine-3-chloro-isopropyl acrylate in the comonomer mixture. After the end of copolymerization, the ampoules were cooled to room temperature and the stoppers were opened; the contents were slowly poured with stirring into a glass with a precipitant (benzene). Copolymer samples were lyophilized and weighed to constant weight on an analytical balance with an accuracy of ± 0.0002. The resulting copolymers are white powdery products that dissolve in water, ethanol, dimethylformamide. The synthesis of N-morpholine-2-chloro-isopropyl acrylate was carried out according to the method described in the work. The intrinsic viscosity of the copolymers was measured in an Ubelode-type viscometer in a 0.1 N aqueous solution of KCl at 30 ° C. The IR spectra of the synthesized compounds were recorded on a Sistem-200 FT-IR spectrophotometer.

The results of the copolymerization reaction of MHIPA with AA can be represented by the following general scheme:





To determine the chemical composition and structure of the obtained copolymerThe methods of elemental analysis and IR spectroscopy were applied. In the IR spectra of copolymers, absorption bands characteristic of a double bond are absent, which confirms the occurrence of the reaction at the vinyl groups of the starting monomers. In the absorption regions characteristic of the morpholine fragment, the spectra of the copolymers are identical to the spectra of the initial MCIPA. Stretching vibrations of C-O and C = O groups of acrylic acid are in the range of 1135 cm-1 and 1600-1700 cm-1. To determine the effect of the composition of the initial mixture of monomers on the composition of the resulting copolymers, the reaction was carried out at different molar ratios of the copolymers (Table 1)

Copolymerization of MHIPA with AA in a DMF solution (TAC = 6 10-3 mol / l, 90 ° C, 1.5 hours)

Composition of the initial mixture,%mol Nitrogen content,% Composition of the copolymer,% mol [η], dl

The composition of the initial	**Nitrogencontent,%**	**Copolymercomposition, mol%**	**[η], дл/г**

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mixture, mol%					
MHIPA	АК		MHIPA	АК	
35	50	2,36	38,23	65,23	0,1
62	30	3,654	65,3	40,23	0,43
71	20	4,251	72,56	20,23	0,558

In the IR spectra of the synthesized monomer, bands of stretching vibrations of C-N groups in the region of 1220 and 1020 cm-1 were found, the first of which is stronger, bending vibrations of C-H in the region of 1400 cm-1. On the low-frequency side of the band and (CH) in In the region of 2800-2700 cm-1, characteristic absorption bands of the morpholine ring are found, and the vibrations of –CH2 – groups have bands of asymmetric and symmetric stretching vibrations in the region of 2950-2920 cm-1, respectively. In the region of 1300-1100 cm-1, there are bands corresponding to torsional and fan vibrations of methylene groups and bending vibrations of methine groups. The pendulum vibrations of the methylene group are in the range of 785-715 cm-1, the absorption of about 705 cm-1 is due to deformation vibrations. Asymmetric stretching vibrations of the C-O-C groups are at 1150-1070 cm-1, the stretching vibration bands of the –C = C– bond are in the region of 1680-1620 cm-1. Also, the structure of the synthesized compound is confirmed by mass spectra. In this regard, further studies of the polymerization reaction of MHIPA were carried out in an environment of organic solvents. To establish the main regularities of the detected polymerization reaction, the influence of various factors was studied: the nature of the solvent, the initiator, the concentration of the monomer and the initiator. The radical polymerization of N-morpholine-3-chloroisopropyl acrylate was studied in the presence of an initiator dinitrilazobis-isobutyric acid (TAK) at 50-70 ° C in a solution of organic solvents.

Conclusion

Studies of the effect of the nature of the solvent on the kinetics of the polymerization process were carried out in acetone, ethanol, and dimethylfomamide.

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