

Investigation Of The Process Of Copolymerization Of Methacrylic Acid With N-Morpholine-3-Chlorous Isopropylacrylate

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ABSTRACT

The article shows studies of the reaction of radical copolymerization in the presence of the initiator azobisisobutyronitrile in dimethylformamide medium, linear copolymers of methacrylic acid with N-morpholine-3-chloroisopropylacrylate were synthesized in the temperature range of 60-70 °C. The influence of the concentration of the initiator on the copolymerization process was studied, and the influence of the ratio of the initial monomers was also established. The composition and structure of the synthesized copolymers were studied by FT-IR spectroscopy. Thermogravimetric studies were carried out and it was found that the curve of the binary copolymer has a wide endothermic peak in the region of 206°C, this is due to the formation of intermolecular anhydride bonds, which contribute to an increase in its thermal stability

Keywords:

Copolymer, N-morpholine-3-chloroisopropylacrylate, methacrylic acid, radical initiator, IR spectroscopy

At present, carboxylcontaining copolymers of synthetic origin are of considerable interest due to the constant expansion of their practical applications, for example, as thickeners, cutting fluids, pour point depressants, plasticizers for concrete mixtures, etc. It is well known that compounds containing a morpholine group in their composition have a wide range of different properties and can be used in pharmaceuticals, medicine, agriculture, oil and gas industry, etc. Literature sources show that the copolymerization of methacrylic acid (MAA)

with basic vinyl monomers, copolymers based on which are produced on an industrial scale and are widely used in various fields of the national economy [1-3].

The aim of the study is the copolymerization of methacrylic acid with N-morpholine-3-chloroisopropylacrylate (MHIPA), which was carried out in the presence of a radical initiator DAA in a solution of dimethylformamide (DMF) in the temperature range of 60-70 °C. The influence of various factors on the course of the reaction was studied (table).

Table

Copolymerization of MHIPA [M1] with MAA [M2] in dimethylformamide

Conditions for the copolymerization process			Exit, %	[η], дЛ/г
Content [M ₁]:[M ₂], weight. %	Quantity of DAA, mol.l ⁻¹	Temperature, °C		

30:70	$1 \cdot 10^{-3}$	60	77,0	0,47
30:70	$2,5 \cdot 10^{-3}$	60	85,0	0,65
30:70	$5 \cdot 10^{-3}$	60	92,2	0,82
30:70	$5 \cdot 10^{-3}$	70	91,7	0,70
30:70	$5 \cdot 10^{-3}$	75	89,4	0,53
50:50	$5 \cdot 10^{-3}$	60	94,5	0,90
70:30	$5 \cdot 10^{-3}$	60	96,8	0,98
30:70	$7 \cdot 10^{-3}$	60	95,0	0,80

The table data show that an increase in the content of MHIPA in the initial mixture of monomers from 30 to 70 weight % leads to an increase in the intrinsic viscosity from 0.82 to 0.98 dl/g, which is explained by the more reactive ability of MHIPA in the copolymerization reaction compared to MAA.

The study of the influence of the reaction temperature shows that its increase in the range of 60-75 °C leads to a decrease in the yield and value of the intrinsic viscosity of the

copolymer. From the results obtained, the optimal conditions for the synthesis of copolymers can be considered: temperature 60°C, initiator concentration $5 \cdot 10^{-3}$ mol/l, reaction time 4 hours.

The composition and structure of the synthesized copolymers were studied by elemental analysis and IR spectroscopy (Fig. 1). The approximate formula of copolymers can be represented as follows:

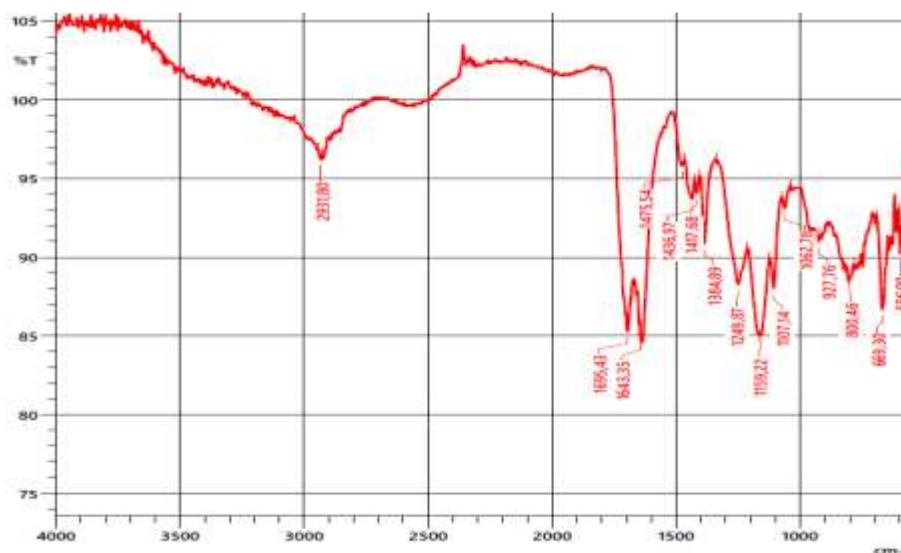
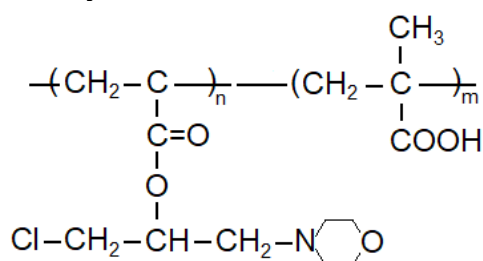


Fig.1. IR spectrum of MHIPA copolymer with MAA at a ratio of 30:70.

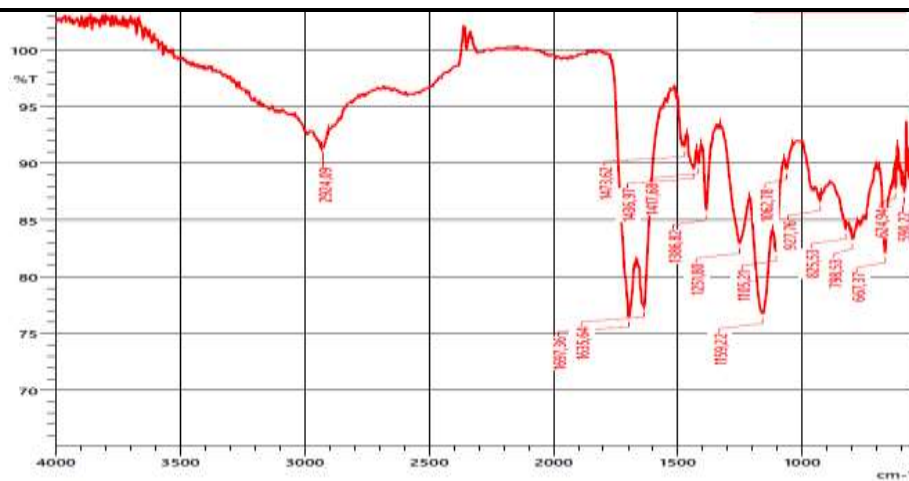


Fig.2. IR spectrum of MHIPA copolymer with MAA at a ratio of 50:50.

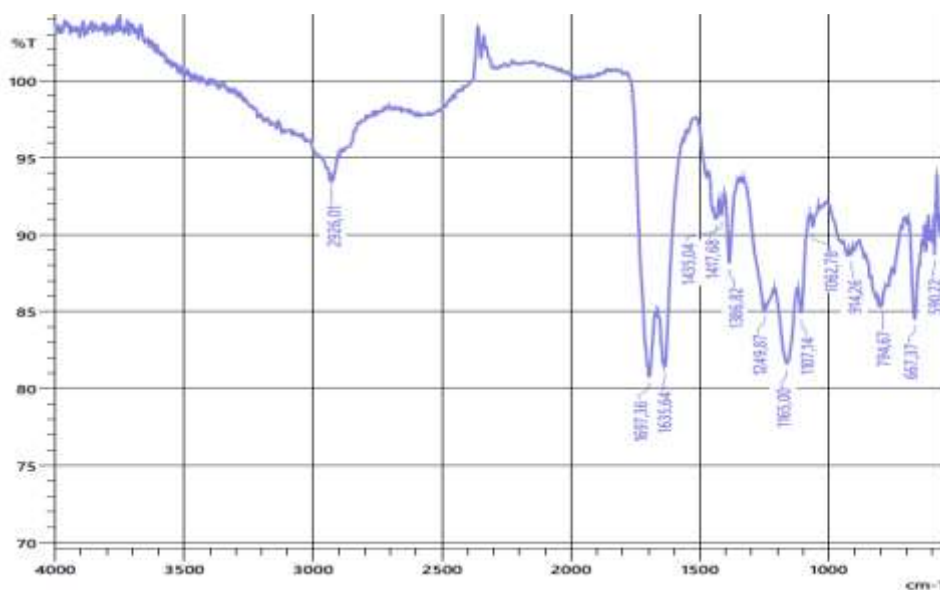


Fig.3. IR spectrum of MHIPA copolymer with MAA at a ratio of 70:30

The IR spectra of the synthesized copolymers show that there are no absorption bands characteristic of the double bond in the region of 1629 cm^{-1} , which confirms the reaction proceeding at the vinyl groups of the initial comonomers. The stretching vibrations of the C=O groups are in the regions of 1107 cm^{-1} and 1697 cm^{-1} ; in the regions of 1165 , 1249 cm^{-1} there are characteristic bands of C–O–C groups, and absorption bands of CH₃ and CH₂ groups at 1387 , 1417 , 1435 , 1436 .

Thus, the binary copolymerization of methacrylic acid with N-morpholine-3-chloroisopropylacrylate in the presence of the DAA initiator in a dimethylformamide solution at $60\text{--}70^\circ\text{C}$ was studied. The effect of the

monomer ratio, initiator concentration, and temperature on the copolymerization reaction has been studied. The structure and composition of the synthesized copolymers have been determined.

Based on this, the synthesis of new morpholine-containing copolymers based on methacrylic acid with N-morpholine-3-chloroisopropylacrylate is of theoretical and practical interest.

Further, thermogravimetric studies of the synthesized copolymers based on methacrylic acid with N-morpholine-3-chloroisopropylacrylate (MHIPA) were carried out (Fig. 4,5).

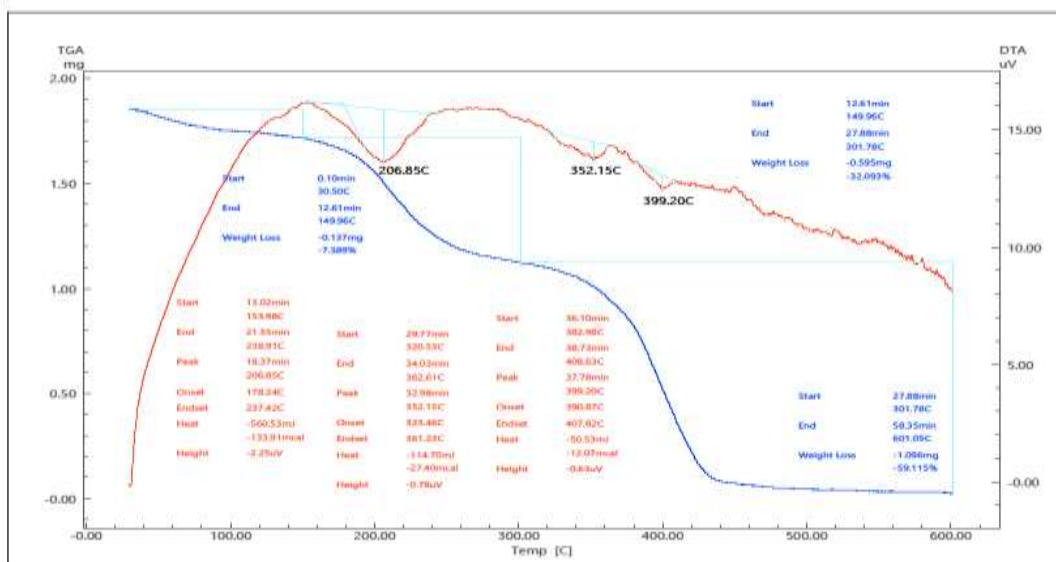


Fig.4. Thermogram of the synthesized MHIPIA copolymer with MAA at a ratio of 30:70.

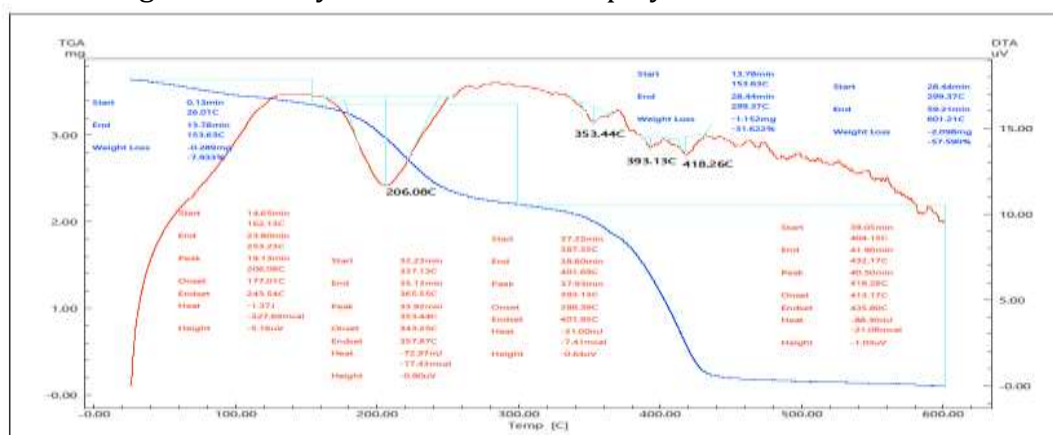


Fig.5. Thermogram of the synthesized MHIPIA copolymer with MAA at a ratio of 30:70

Thus, the curves of thermogravimetric analysis show that the curve of the binary copolymer has a wide endothermic peak in the region of 206 °C. Apparently, the broadening of the endothermic peak in the DTA curve of the copolymer is associated with the formation of intermolecular anhydride bonds, which contribute to an increase in its thermal stability.

Thus, the approximate structure and thermal stability of the synthesized copolymers have been determined.

References

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