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Chemical modification of functional copolymers

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Abstract: *The possibility of chemical modification of thermally-stable functional copolymers of 1-vinyl-1,2,4-triazole with vinyl acetate of various compositions synthesized under conditions of free-radical polymerisation in the presence of azobisisobutyronitrile was studied. Modification of the copolymers was carried out by alkaline hydrolysis; as a result, new copolymers containing vinyl triazole and vinyl alcohol units in the macromolecules were obtained. The structure, composition, physical and chemical properties of the copolymers were determined using elemental analysis, infrared spectroscopy and thermogravimetric analysis. With an increase in the number of vinyl alcohol units in the copolymer from 25 to 87 mol %, a nearly twofold reduction in the intrinsic viscosity of the copolymers was observed. The copolymers, which exhibit dielectric properties, are characterised by electrical conductivity of the order of 10^{-14}–10^{-15} S/cm. According to thermogravimetric analysis data, the copolymers are resistant to thermo-oxidative degradation up to 270–290 °C depending on the composition. The introduction of a vinyl alcohol fragment into the structure of copolymer macromolecules contributed to the improvement of their fibre- and film-forming properties – that is, fibres and transparent elastic films with good adhesion to various metal surfaces can be formed from solutions of modified copolymers based on 1-vinyl-1,2,4-triazole.*

Keywords: *copolymers, 1-vinyl-1,2,4-triazole, vinyl acetate, vinyl alcohol*

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Химическая модификация функциональных сополимеров

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Резюме: *Исследована возможность химической модификации функциональных термостойких сополимеров 1-винил-1,2,4-триазола с винилацетатом разного состава, синтезированных в условиях свободно-радикального инициирования в присутствии динитрила азобисизомасляной кислоты. Модификацию сополимеров осуществляли методом щелочного гидролиза, в результате получены новые сополимеры, содержащие в макромолекулах звенья винилтриазола и винилового спирта. Структуру, состав и физико-химические свойства сополимеров определяли с использованием методов элементного анализа, ИК-спектроскопии и термогравиметрического анализа. Установлено, что с увеличением количества звеньев винилового спирта в сополимере от 25 до 87 мол.% наблюдается уменьшение значений характеристической вязкости сополимеров практически в два раза. Сополимеры проявляют диэлектрические свойства, характеризуются удельной электрической проводимостью порядка 10^{-14}–10^{-15} См/см. По данным термогравиметрического анализа сополимеры обладают устойчивостью к термоокислительной деструкции до 270–290 °С в зависимости от состава. Введение фрагмента винилового спирта в структуру макромолекул сополимеров способствовало улучшению их волокно- и пленкообразующих свойств, а именно: из растворов модифицированных сополимеров на основе 1-винил-1,2,4-триазола успешно формируются волокна и прозрачные эластичные пленки с хорошей адгезией на различных металлических поверхностях.*

Ключевые слова: *сополимеры, 1-винил-1,2,4-триазол, винилацетат, виниловый спирт*

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INTRODUCTION

N-vinylazoles and vinyl acetate are promising monomers for the development of new polymer materials having special physical and mechanical parameters and a wide range of valuable properties [1–13]. The radical copolymerisation of 1-vinyl-1,2,4-triazole with vinyl acetate have previously been successfully used for synthesising new functional copolymers of various compositions with triazole and acetate fragments in macromolecules, which have demonstrated good solubility (including water solubility), high thermal stability (up to 300–325 °C), as well as dielectric and stabilising propertie [14–17].

The purpose of this study was to investigate the possibility of modifying the copolymers of 1-vinyl-1,2,4-triazole (VT) with vinyl acetate (VA) using alkaline hydrolysis, as well as to explore the physical and chemical properties of the newly obtained copolymers.

EXPERIMENTAL

Elemental analysis was performed using a Thermo Finnigan Flash EA 1112 analyser. The IR spectra were recorded on a Vertex 70 spectrometer (Bruker, Germany). The intrinsic viscosity was measured by a Ubbelohde viscometer. Thermal analysis was performed using a Q-1500 thermogravimetric analysis system (MOM, Hungary). Electrical conductivity was measured on a E6-13A teraohmmeter. VTs were synthesised according to the procedure proposed in [18]; vinyl acetate and azobisisobutyronitrile (AIBN) (Aldrich) were used without additional purification.

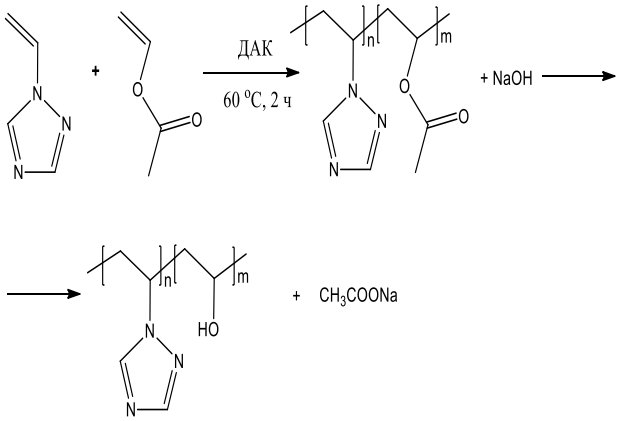
Synthesis of copolymers. The copolymerisation of VT with VA was carried out in sealed ampules in an argon atmosphere in ethyl acetate in the presence of AIBN (1% wt) at 60 °C for 2 h at different ratios of monomers. The copolymers were isolated by precipitation from DMF into acetone, washed with ethyl alcohol and dried in vacuum (40 °C, P_2O_5) until constant weight.

Modification of copolymers by alkaline hydrolysis. 0.35 g of crushed and dried VT copolymer with VA was placed in a round bottom flask equipped with a mechanical stirrer, a refrigerator with a calcium chloride tube and a contact thermometer. Next, a 2 % solution of NaOH in methanol (6 ml) was added from a dropping funnel. The reaction was carried out at 60 °C for 3 hours until the formed gel turned into a powder. Modified copolymers were then isolated by centrifugation, washed three times with ethanol and dried in vacuum (40 °C, P_2O_5) to constant weight. The degree of hydrolysis was determined by the method presented in the work edited by Kurenkov V.F.[1]

RESULTS AND DISCUSSION

The copolymerisation of VT with VA was carried out under conditions of free-radical initiation at different monomer ratios in the initial reagent mixture. As a result, copolymers of various compositions (73:27, 47:53, and 13:87 mol %) were obtained in the form of light-yellow powders that were soluble in water, DMSO and DMF. The resulting copolymers were then modified by alkaline hydrolysis in a NaOH – methanol medium at 60 °C for 3 h (see schematic reaction diagram and table below).

The obtained modified copolymers 1–3 comprise light-yellow powders soluble in DMSO, DMF, DMAA and in water (through swelling).



Schematic diagram of the synthesis and modification of 1-vinyl-1,2,4-triazole and vinyl acetate copolymers

Схема синтеза и модификации сополимеров 1-винил-1,2,4-триазола с винилацетатом

[1] Wessling RA, Gibbs DS, Obi BE, Beyer DE, Delassus PT, Howell BA. Vinylidene Chloride Polymers. In: *Encyclopedia of Polymer Science and Technology*. New York: John Wiley and Sons, 2002, vol. 4, p. 458–510. https://doi.org/10.1002/0471440264.pst391

Synthesis conditions and characteristics of the copolymers of 1-vinyl-1,2,4-triazole with vinyl acetate (VA) and vinyl alcohol (VOH)

Условия синтеза и характеристики сополимеров 1-винил-1,2,4-триазола (ВТ) с винилацетатом (ВА) и виниловым спиртом (ВС)

Sample	Composition of copolymer VT: VA:VOH, mol %		Yield, %	Degree of hydrolysis, %	Intrinsic viscosity, dL/g	
	original	modified			original	modified
1	73:27:0	73:2:25	74	92.7	2.3	1.66
2	47:53:0	47:0:53	73	100	0.82	1.14
3	13:87:0	13:0:87	67	100	0.35	0.81

The macromolecules of copolymers 2 and 3 consist of vinyl triazole and vinyl alcohol units in different ratios. In the IR spectra of the copolymers, characteristic absorption bands of the valence and deformation vibrations of the triazole ring are preserved: 1503–1506 (C=N), 1430–1435 (C-N), 1273–1275 (N-N), 1001–1004 (C-H), 660–663 (C-N); the absorption bands of the vinyl acetate fragment disappear: 1730–1734, 1227–1235 (C=O), 1370–1375, 1430–1435 (CH_3), and a wide absorption band emerges in the region of 3000–3340 cm^{-1}, due to the appearance of vinyl alcohol OH-groups in the macromolecules of copolymers.

With an increase in the number of vinyl alcohol units in the copolymer from 25 to 87 mol %, a nearly twofold reduction in the intrinsic viscosity of the copolymers was observed.

The copolymers, which exhibit dielectric properties, are characterised by electrical conductivity of the order of 10^{-14}–10^{-15} S/cm. According to thermogravimetric analysis data, the copolymers are resistant to thermo-oxidative degradation up to 270–290 °C depending on the composition. The introduction of a vinyl alcohol fragment into the structure of the copolymer macromolecules contributed to an improvement of their film-forming properties. It was observed that fibres and transparent elastic films formed -from solutions of modified copolymers demonstrate good adhesion properties on various surfaces.

CONCLUSION

Thus, by chemically modifying copolymers of 1-vinyl-1,2,4-triazole with vinyl acetate, new soluble copolymers were synthesised, whose macromolecules consisted of vinyl triazole and vinyl alcohol units. The copolymers have high thermal stability, exhibiting good insulating properties, as well as supporting film and fibre formation, and are highly promising for the development of new, practically-useful materials.

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Contribution

Galina F. Prozorova, Nadezhda P. Kuznetsova, Svetlana A. Korzhova carried out the experimental work, on the basis of the results summarized the material and wrote the manuscript. Galina F. Prozorova, Nadezhda P. Kuznetsova, Svetlana A. Korzhova have equal author's rights and bear equal responsibility for plagiarism.

Conflict interests

The authors declare no conflict of interests regarding the publication of this article.

The final manuscript has been read and approved by all the co-authors.

Критерии авторства

Прозорова Г.Ф., Кузнецова Н.П., Коржова С.А. выполнили экспериментальную работу, на основании полученных результатов провели обощение и написали рукопись. Прозорова Г.Ф., Кузнецова Н.П., Коржова С.А. имеют на статью равные авторские права и несут равную ответственность за плагиат.

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Все авторы прочитали и одобрили окончательный вариант рукописи.

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