






## PHYSICAL AND ANALYTICAL CHEMISTRY

### Article

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### Investigation of Physical and Chemical Properties of Thermosetting Copolymers of Polyethylene Glycol Maleate with Acrylic Acid During their “Cold” Curing

The paper presents information on the study of physical and chemical properties of solutions of polyethylene glycol maleate in acrylic acid and products of their “cold” curing. The weight-average molecular weight of the initial polyethylene glycol maleate was established using the GPC method. The value of the degree of unsaturation of both the initial unsaturated polyester and copolymers based on it was determined using the bromide-bromate method. The dynamic viscosity of the polymer-monomer mixture of polyester and acrylic acid was established using viscometry. The density of the stock solutions was analyzed using the pycnometric method and the density of the cured products was determined using the hydrostatic method. The total volume shrinkage of the copolymers was calculated from the obtained density values. The composition of the copolymers was analyzed/studied with the help of HPLC. The degree of swelling of the cured products was also calculated by gravimetric method, and their identification was carried out using IR- and <sup>1</sup>H NMR-spectroscopy. The surface topography of cured copolymers was investigated by SEM. The results obtained indicate to the possibility of controlling the properties of cured unsaturated polyester by varying the initial composition of polymer-monomer mixture depending on its purpose and obtaining on its basis the polymer matrix for the manufacture of bulk products with good physical and chemical properties.

**Keywords:** unsaturated polyester, “cold” curing, density, viscosity, total volumetric shrinkage, hydrophobicity, thermosetting polymers, bulk products

### Introduction

Nowadays, thermosetting polyester binders are widely used in various industrial fields due to their good performance properties, including stability and resistance to negative external influences [1–3]. In particular, in the automotive industry the thermosetting polyester binders are used for the production of high-strength composites and coatings, as well as in the production of adhesive compositions that provide strong and durable bonding of various materials [4]. In the aerospace industry, they are used to create lightweight and strong structural materials that must withstand extreme operating conditions such as high temperatures and strong mechanical loads [5, 6].

In addition, thermosetting polyester binders play an important role in the production of construction materials [7, 8]. They are used as polymer binders for bulk products such as concretes [9–11] and construction mixtures to improve their strength properties as well as increase their resistance to environmental influences [12, 13]. Polyester binders are also actively used for the production of modern high-quality insulation materials, which are used in the construction and electrical industries for protection against high temperatures and corrosion [14, 15].

One of the important directions in the development of thermosetting polyester binders is the creation of polymers with unique combinations of properties that can be adapted to specific technological processes. This is achieved by introducing various additives and fillers into the polymer structure, which make it possible to adjust its mechanical, thermal and chemical properties depending on the requirements of the final product. In particular, the addition of minerals, carbon fibers or other materials can significantly increase the strength and resistance of polymers to the negative effects of the external environment [16–18].

Thermosetting polyester binders themselves contain unsaturated bonds in their structure, which provide reactivity and polymerization ability [19]. As a result of the crosslinking reaction, a three-dimensional mesh structure is formed, which significantly increases the mechanical and thermal characteristics of the material and contributes to increasing their resistance to aggressive chemical media. The structure of unsaturated polyesters, consisting of repeating units, allows modifying the properties of polymers by changing the ratio of the monomers and using different curing methods. This opens up opportunities for the creation of the materials with specified characteristics, which is especially important in the context of modern requirements to materials.

The modification of thermosetting polyester materials for improving their properties is great importance. Modern developments in the field of thermosetting binders are aimed at creating polymers with improved resistance to ultraviolet radiation, increased mechanical strength and improved adhesion to various surfaces. This makes it possible to expand the field of application of the materials based on thermosetting polyester binders in such innovative industries as the production of electronic components, biomaterials, and nanocomposites [4, 7, 13, 14].

To date, the most studied are the curing reactions of unsaturated polyesters with styrene [20] and methyl methacrylate to obtain rigid polymeric materials with good performance properties [21]. However, there is no information in literature on the use of unsaturated carboxylic acids as a solvent-curing agent in the curing of polyester binders in order to obtain the filler products based on them.

In our previously published article, we investigated the properties of polyethylene(propylene)glycol-fumarate curing products to determine the possibility of using them as a polymer matrix of hermetic materials [22, 23]. This paper presents data on the study of physicochemical properties of both initial solutions of polyethylene glycol maleate (p-EGM) in acrylic acid (AA) and cured products based on them. The presented studies will allow us to evaluate the suitability of this binary system of p-EGM–AA as thermosetting polyester binders for the production of filler products with minimal inclusion of mineral additives.

### *Experimental*

The following reagents (“Sigma-Aldrich”) were used in this study:

- ethylene glycol and maleic anhydride (co-reagents for the synthesis of initial p-EGM), and zinc chloride (catalyst for the polycondensation reaction) in the first step;
- in the second step, acrylic acid was used as a solvent to prepare solutions of p-EGM in unsaturated carboxylic acid;
- “cold” curing of the above solutions was carried out in the presence of an initiating system consisting of benzene peroxide (BP, initiator) and dimethylaniline (DMA, activator). Dioxane was used as a solvent. All reagents used were purchased from “Sigma-Aldrich”. The purity of the reagents used was 99.95 %, therefore they were used without additional purification.

The initial unsaturated polyester — polyethylene glycol maleate — was obtained by polycondensation of ethylene glycol (dioatomic alcohol) and maleic anhydride (dicarbonic anhydride) with a small excess of the first co-reagent. Zinc chloride was used as a catalyst, which allowed the process to be carried out at a reduced temperature 150–160 °C. The reaction flow was monitored by determining the acid number until the value of 30–40 units was reached, as well as measuring the volume of water released as a result of polycondensation [20]. The molecular weight of the synthesized p-EGM was determined using GPC on a VISCOTEK 270 DUAL DETECTOR MALVERN. The studies were carried out using dust-free dioxane. The measured molecular weight was approximately 1232 Da ( $M_w$ ). The polyester yield was 98 %.

The degree of unsaturation of the synthesized p-EGM was determined by bromide-bromate method, which was 89 % [20].

Further, solutions of p-EGM in acrylic acid (AA) of different mass composition were prepared: ~60:40 mass.%, ~70:30 mass.%, ~80:20 mass.%, respectively. After preparation of the solutions, their dynamic viscosity at 293 K was determined. The studies were carried out on a vibrating viscometer SV-10. Additionally, the viscometer is equipped with a VT3 liquid thermostat, which allows to maintain the temperature set dur-

ing the experiment. The density of these solutions (ISO 1675:1985 (GOST 18329-2014)) was determined using a pycnometer.

Further, curing was carried out at 293 K in the analyzed solutions after introducing an optimized “cold” curing initiating system (1.0 % : 0.15 % of the polymer-monomer mixture mass) [23]. The obtained p-EGM-AA copolymers were washed with dioxane in order to get rid of the residues of unreacted mixture. The obtained mother liquors were examined by HPLC, from which the actual compositions of the synthesized copolymers were calculated. The yield of the copolymers was also determined gravimetrically [20].

Identification of the curing products was carried out using IR and NMR spectroscopy, in particular to determine the presence of functional groups. The degree of unsaturation of the analyzed copolymers was also determined by bromide-bromate method. For IR study, potassium bromide tablets were fabricated and analyzed on an FSM 1201 spectrometer. NMR analysis was carried out on a DX-90M instrument [21].

The total volumetric shrinkage of the copolymers was calculated on the basis of the obtained values of densities of solutions and cured products, respectively [23].

The surface topography of the cured polymers was studied by SEM on a MIRA 3 high-performance microscope (TESCAN) with a 5.0 kV high-voltage (HV) detector. The SE detector was used to acquire images at 57.6-57.7 kx magnification with composite contrast, which allows imaging with very detailed object structure at the nanometer level. The field of view was 6.00 nm and the image resolution was 1024×1024 pixels [22].

The hydrophobicity of cured products was established by gravimetric method. Studies of the degree of swelling were carried out by keeping a sample (~0.3 g) of the test samples in water ( $T = 20\text{ }^{\circ}\text{C}$ , pH 7) for 24 hours. The calculation was carried out according to the formula:

$$\alpha = \frac{m - m_0}{m_0} \cdot 100\%,$$

where  $m$  — the mass of the swollen sample, g;  $m_0$  — mass of dry sample, g.

### Results and Discussion

Bulk-type polymer products are becoming increasingly in demand as they are characterized by their versatility and good performance advantages, allowing them to be used in various fields of industries. They are actively used in various spheres, including construction industry, automotive and shipbuilding, electronic industry, consumer products and other industries.

One of the main reasons for their popularity is the possibility of manufacturing the polymer bulk materials on their basis. At the same time, the casting method allows to observe strict parameters when manufacturing such products of non-standard or complex geometry. One of the most promising compounds for the production of bulk goods are the unsaturated polyesters of various compositions, the curing of which in the presence of an initiating system of optimized composition allows to make the process of production of the final product economically profitable. For this purpose, we synthesized an initial unsaturated polyester — polyethylene glycol maleate — by polycondensation reaction. Figure 1 schematically represents the reaction of synthesis:

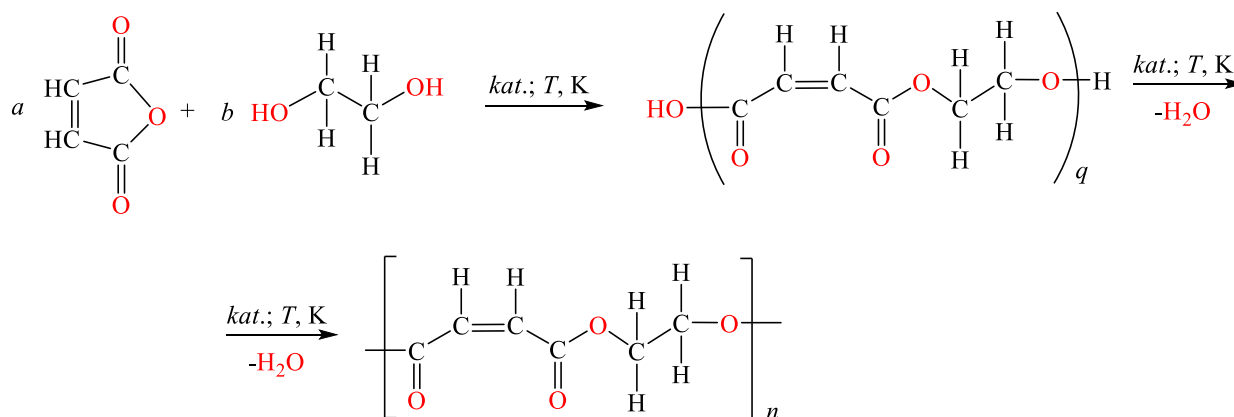


Figure 1. Polyethylene glycol maleate link formation

Solutions of p-EGM with AA of different concentrations were prepared and their density and viscosity were determined. The results obtained are presented in Table 1.

Table 1

**Physical and chemical properties of stock solutions of p-EGM ( $M_1$ ) with AA ( $M_2$ ),  $T = 293\text{K}$** 

Composition of prepared solution, mass.%		Dynamic viscosity value ( $\eta$ ), mPa·s	Solution density value ( $\rho$ ), g/cm <sup>3</sup>
$M_1$	$M_2$		
58.48	41.52	294.3±0.1	1.2069±0.060
70.22	29.78	576.5±0.2	1.2202±0.061
79.51	20.49	736.8±0.3	1.2412±0.061

Analyzing the data in Table 1, a good correlation between the content of unsaturated polyester in solution and the values of their dynamic viscosity and density is observed. In particular, at the minimum content of p-EGM (~60 mass.%) in the solution, the minimum values of its dynamic viscosity (294.3 mPa·s) and density (1.2069 g/cm<sup>3</sup>) are observed. On the contrary, the maximum content of unsaturated polyester (~80 mass.%) corresponds to the maximum value of dynamic viscosity (736.8 mPa·s) and density (1.2412 g/cm<sup>3</sup>), which is a consequence of high viscosity characteristics of p-EGM. As a result, with increasing p-EGM content in the solution, an increase in such parameters as viscosity and density of this solution is observed.

Figure 2 graphically shows the relationship between the dynamic viscosity and density of these solutions in dependence on the p-EGM content in them.

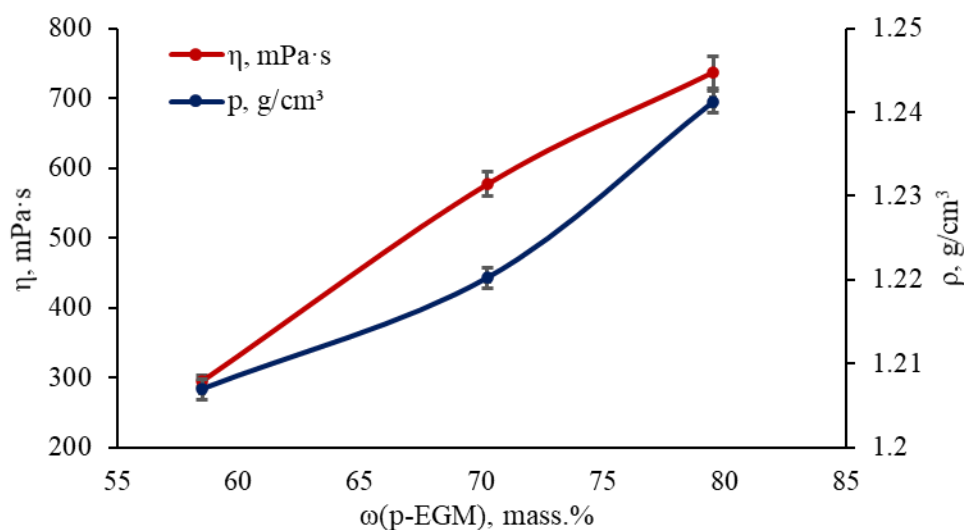
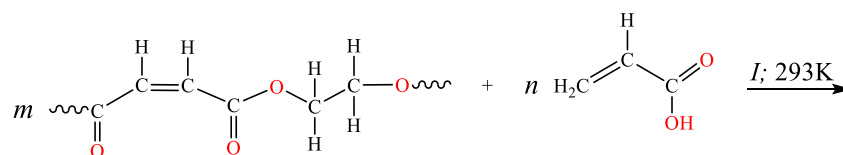


Figure 2. Dependence of dynamic viscosity and density of initial solutions of p-EGM–AA on the content of p-EGM in them

“Cold” curing of the synthesized polyethylene glycol maleate with acrylic acid was carried out in the presence of an initiating system consisting of benzoyl peroxide (initiator) and dimethylaniline (activator). The reaction was carried out at a temperature of 293 K. Schematically, the radical copolymerization reaction is presented in Figure 3.



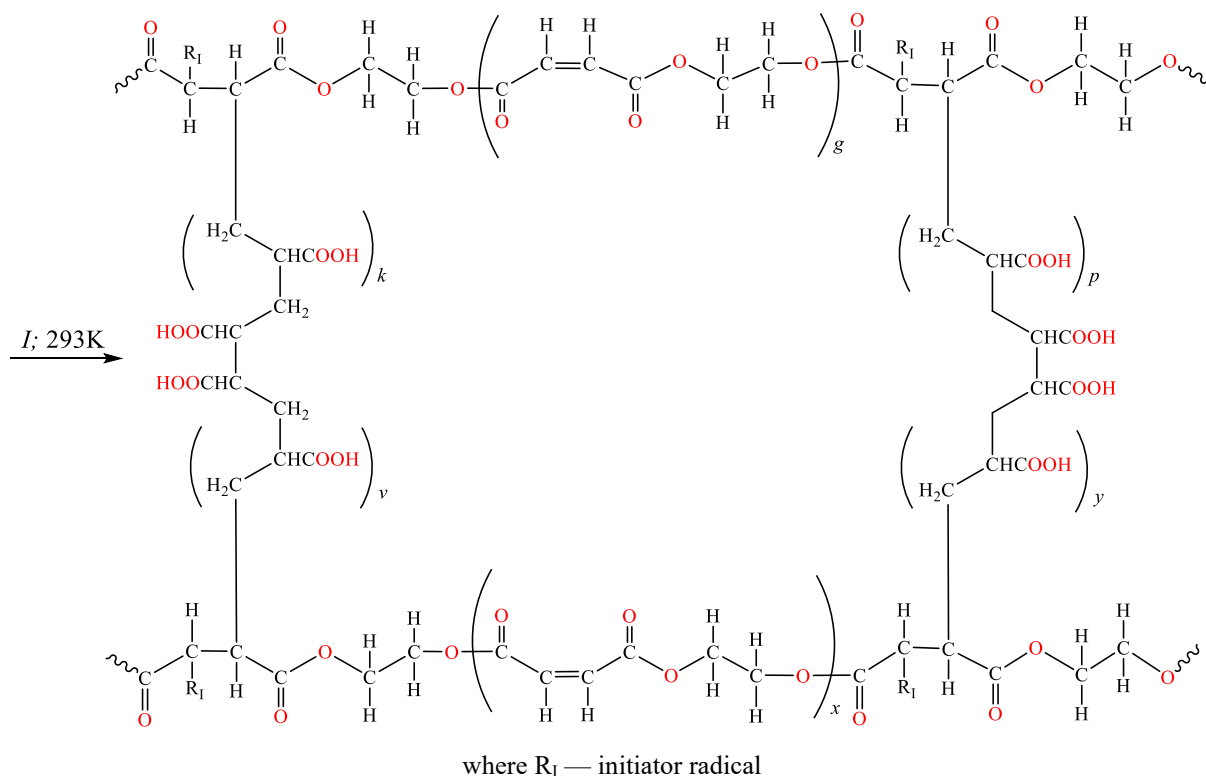


Figure 3. Copolymer structural fragments of the p-EGM-AA

Insoluble net polymers were obtained as a result of the reaction. The obtained solutions in dioxane were analyzed by HPLC, which allowed to establish the true composition of the obtained copolymers. The results of HPLC-analysis are presented in Table 2:

Table 2

**Dependence of copolymer composition and some of their parameters  
on the composition of the initial mixtures of p-EGM ( $M_1$ ) with AA ( $M_2$ )**

Composition of the initial solution, mass. %		Composition of the copolymers, mass. %		Yield, %	Degree of unsaturation, %
$M_1$	$M_2$	$m_1$	$m_2$		
58.48	41.52	57.44	42.56	91.5	37.7
70.22	29.78	68.18	31.82	90.2	42.4
79.51	20.49	77.36	22.64	89.4	54.8

The obtained copolymers were transferred into Petri dishes and dried under vacuum in a desiccator at 30 °C. After establishing constant mass, the yield of the copolymers was calculated. The obtained data is also presented in Table 2. Analysis of the yield values of p-EGM copolymers with AA indicates an increase as the content of unsaturated polyester in the initial solutions decreases. The degree of unsaturation of the cured copolymers was determined by bromatometry, which correlates well with the p-EGM content in the initial reaction mixture. The degree of unsaturation of the cured products is in direct dependence on the content of unsaturated polyester (p-EGM) in the reaction mixture.

The products were identified using IR and NMR spectroscopy. Figure 4 shows the IR spectra of the initial p-EGM and its copolymers with AA of the compositions of 57.44:42.56 mass.% and 77.36:22.64 mass.%. The analysis of the obtained IR spectrum of unsaturated polyester reveals characteristic peaks in the 1570–1590  $\text{cm}^{-1}$  range, indicating the presence of unsaturated double bonds in p-EGM. The peaks between 1400–1440  $\text{cm}^{-1}$  correspond to the ether group.

Similar absorption bands, but of lower intensity, are present in the IR spectra of the cured products. This confirms the presence of some amount of unsaturated bonds that have not reacted. Also, peaks at

1715  $\text{cm}^{-1}$ , 1742  $\text{cm}^{-1}$ , 3439  $\text{cm}^{-1}$  and 1711  $\text{cm}^{-1}$ , 1736  $\text{cm}^{-1}$  and 3432  $\text{cm}^{-1}$  are present in both IR-spectra of the cured products, respectively, indicating the presence of  $-\text{COOH}$  carboxyl groups [24].

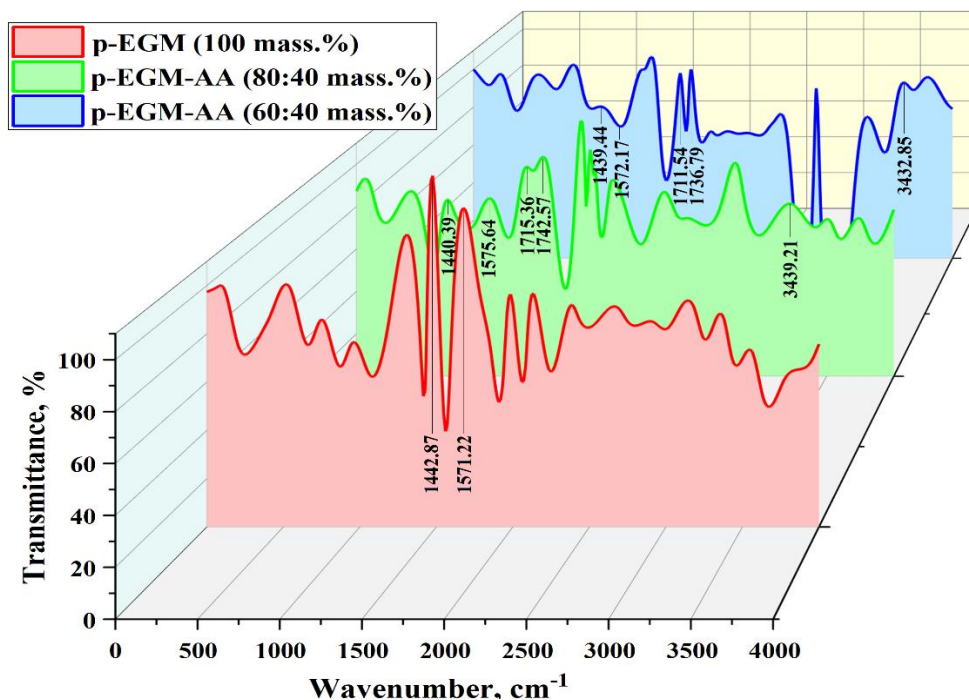


Figure 4. IR spectra of the initial p-EGM and p-EGM-AA copolymers

The  $^1\text{H}$  NMR spectrum (Fig. 5) of the p-EGM-AA copolymer shows a pronounced multiplet expressed by signals in the range of 1.14–1.31 ppm. The appearance of the multiplet signal in this range confirms the presence of protons of methylene groups Ha in the polymer linkage, which are bound to oxygen atoms. The presence of multiplet signals in the ranges of 2.94–3.05 ppm and 3.65–4.42 ppm indicates the presence of protons of simple and ester methine and methylene groups of aliphatic series (Hb and Hc). In the range of 5.06–5.23 ppm there is a signal of comparatively lower intensity than in the range of 6.63–6.85 ppm where a signal of high intensity corresponding to a considerable amount of ethylene protons Hd in the copolymer structure appears. The separation of ethylene protons into two multiplet signals is due to their cis- and partially trans-orientation, with neighboring functional groups also having an influence. The high signal intensity in the range of 5.06–5.23 ppm indicates the dominance of cis-isomerism, confirming the structure of p-EGM. Similarly, the  $^1\text{H}$  NMR spectrum of the original p-EGM is dominated by ethylene proton signals corresponding to cis-isomerism. The fixation of ethylene protons on the  $^1\text{H}$  NMR spectrum, determined at the multiplet in the range of 6.68–6.85 ppm, indicates the presence of more electronegative atoms (neighboring oxygen atoms) in comparison with the protons of the multiplet in the region of 5.06–5.23 ppm [25].

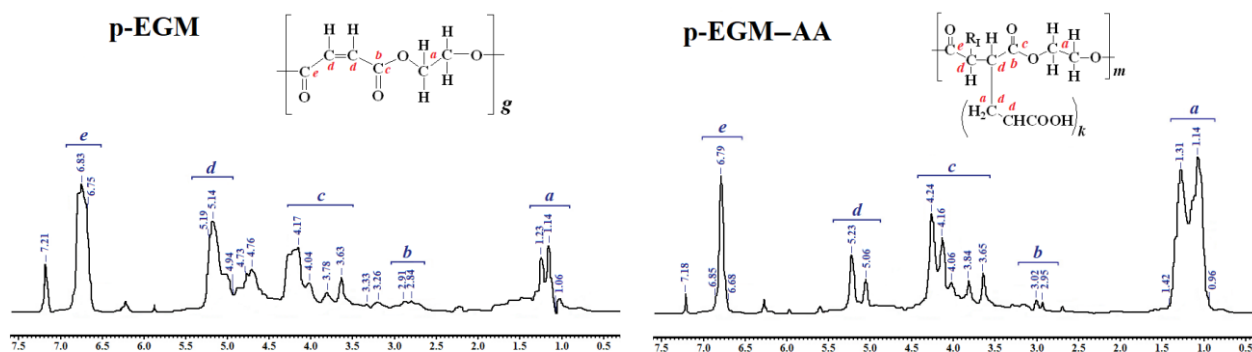


Figure 5.  $^1\text{H}$  NMR-spectra of initial p-EGM and p-EGM-AA copolymers (77.36:22.64 mass.%)



“Cold” curing of p-EGM was carried out in mass without solvent, due to which the total volumetric shrinkage of the final product is insignificant. To obtain numerical values of this index, the density of the cured unsaturated polyester was determined using the hydrostatic method. The obtained values of density and, accordingly, the calculated volumetric shrinkage, are given in Table 3.

Table 3

**Dependence of copolymer properties on mass composition of p-EGM–AA, PB + DMA (1 % + 0.15 %), T = 293K**

Composition of initial solution, mass. %		Density ( $\rho$ ), g/cm <sup>3</sup>	Volume shrinkage, %	Swelling degree, %
p-EGM	AA			
57.44	42.56	1.3195±0.074	9.3	144.6
68.18	31.82	1.3257±0.074	8.6	127.9
77.36	22.64	1.3324±0.075	7.3	78.4
100	0	1.3414±0.075	4.4	12.8

Analysis of the density values of the synthesized p-EGM-AA copolymers and the dynamic viscosity and density values of p-EGM solutions in AA shows their good correlation. Thus, the maximum content of p-EGM (79.51:20.49 mass.%) in the initial solution leads to a cured product (77.36:22.64 mass.%) with a higher density of 1.3489 g/cm<sup>3</sup>. According to the values of the densities of the solutions of p-EGM in AA and the obtained cured copolymers, their total volume shrinkage indices were calculated depending on the composition of the initial reaction mixture. Based on the data obtained, it was found that this parameter does not exceed 10 %, while the lowest total volume shrinkage is observed for the solution with the maximum p-EGM content (79.51:20.49 mass.%). This parameter, not exceeding 10 %, corresponds to low shrinkage, which characterizes these solutions as suitable raw materials for the production of bulk products [1, 12].

Further, the degree of water sorption by synthesized copolymers was determined by gravimetric method. The results are also presented in Table 3. The data obtained indicate a low moisture absorption index, which defines these compounds as hydrophobic. At the same time, the minimum sorption capacity is observed for the copolymer with the highest unsaturated polyester content.

The surface topography of the initial p-EGM and its copolymers with AA was analyzed using SEM, with the results presented in Figure 6 [26].

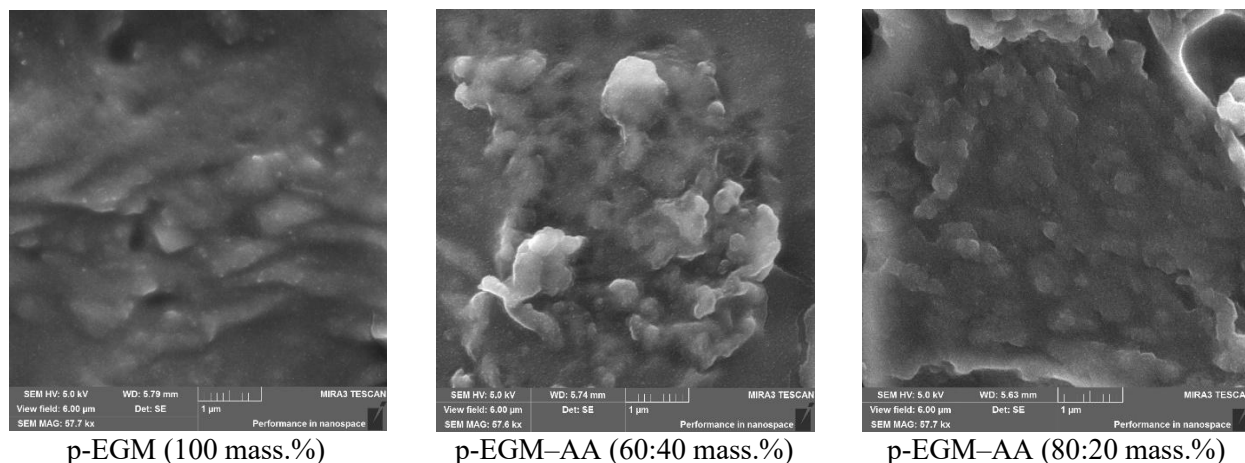


Figure 6. SEM-images of the initial p-EGM and p-EGM-AA copolymers

SEM images of the initial polyether confirm its low porosity. On the contrary, in the SEM images of the p-EGM-AA copolymers, it can be seen that the formation of a porous mesh structure is observed as a result of the curing of the unsaturated polyester with acrylic acid. Comparing SEM images of copolymers with minimum (57.44:42.56 mass.%) and maximum (77.36:22.64 mass.%) content of p-EGM, it should be noted that a greater number of pores and their larger size is observed in the copolymer with a lower content of unsaturated polyether. This correlates well with the results of gravimetric analysis to establish the moisture absorption capacity of the cured copolymers. In particular, the polymers having a more porous structure

(p-EGM–AA composition 57.44:42.56 mass.%) show greater moisture adsorption capacity in contrast to compounds with a high content of unsaturated polyester (the composition of p-EGM–AA is 77.36:22.64 mass.%), which have less pronounced porosity. Regarding the initial unsaturated polyester, it should be noted that this compound is hydrophobic, which is confirmed by the almost complete absence of pores on SEM-images and gravimetric analysis, which showed a swelling degree of 12.8 %.

### Conclusions

Analyzing the obtained results of studying the physicochemical properties of solutions of polyethylene glycol maleate in acrylic acid of various composition, as well as cured copolymers on their basis, we can conclude that their use in the manufacture of filler products with a minimum content of mineral bulk fillers is promising. In particular, the dependence of the increase in such indicators as density and viscosity of the analyzed solutions on the content of unsaturated polyester in their composition was demonstrated. Thus, an increase in the unsaturated polyester content in the initial solutions promotes an increase in their dynamic viscosity and density. At the same time, cured products with a high content of p-EGM show higher density and degree of unsaturation.

The analysis of the total volume shrinkage index as well as swelling of cured products shows a similar correlation of these indicators on the content of unsaturated polyester in composition of the copolymers. Thus, the degree of swelling of cured products decreases with increasing p-EGM content. In general, studies of the degree of swelling showed a low moisture sorption capacity of the synthesized copolymers, which allows us to attribute these cured products to hydrophobic polymers. Taking into account that acrylic acid in the initial solution was used simultaneously as a solvent and hardener, the cured solutions had low volumetric shrinkage values (more than 10 %).

Obtained values are important parameters for polymer solutions that can be used to produce fill products for the construction industry. In particular, the production of polymer tiles. Because of this, the most preferable composition with the optimal complex of physical and chemical properties (including high hydrophobicity) for the manufacture of bulk products is the solution of p-EGM–AA (~80:20 mass.%).

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### Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. **CRedit**: **Gulsym Kabayevna Burkeyeva** conceptualization, validation, visualization, supervision & editing; **Anna Konstantinovna Kovaleva** conceptualization, data curation, methodology, validation, visualization, writing-original draft, writing-review & editing; **Danagul Magazovna Muslimova** data curation & methodology; **David Havlicek** supervision & editing; **Nurken Meiramuly Zhumabek** data curation & investigation; **Nurlan Aleksandrovich Nukin** data curation & investigation.

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### Conflicts of Interest

The authors declare no conflict of interest.

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