Original Article

THE STUDYING SYNTHESIS AND RESEARCH OF NICKEL AND TIN ACRYLATE ON THE BASIS OF THE COPOLYMERS

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Abstract

In this study, we compared the relationship between the synthesis methods, structure and properties that have a good effect on the structure and size, morphology of the copolymer matrix, as well as solubility of copolymers based on nickel and tin acrylates. in water, electricity and heat properties. allows. Based on the results of this work, the possibilities of application in the production of hydrogel and heat-resistant adhesive coatings were considered. To achieve this goal, copolymers based on nickel and tin acrylates were analyzed using TGA, DTA, IR-spectrum, SEM, scanning electron microscopy and elemental analysis.

Keywords: Acrylic acid, methyl methacrylate, hydrogel, $SnCl_2$, $NiCl_2$.

1. Introduction

In the synthesis of copolymers, it is possible to improve their thermal, mechanical and physicochemical properties mainly by adding a basic function to monomers [1]. Acrylates on the basis of the metal salts, and copolymers on the basis of the methyl methacrylate monomers can be used as coatings due to their ease of production, low cost, and high heat resistance and refractory properties [2-3]. The use of coatings made of metal acrylate copolymers is a more effective method in increasing the heat and external corrosion resistance of steel equipment in industrial processes, which is effective in the coating industry [4]. The composition of copolymers used for adhesives is mainly due to the ratio of metal acrylates and monomers [5]. Hydrogel on the basis of the synthesized copolymers is a promising (bio-) material from them due to its bio-flexibility, biological degradation and excellent mechanical properties [6-7]. The biological properties, nature and use of hydrogels have been of great interest to biomaterials for many years [8]. In the synthesis of hydrogels of copolymers on the basis of the acrylates and their monomers, various compositions of acrylic monomers were prepared. To separate various metal ions (Cu2 +, Cd2 + and Ni2 +) from wastewater, the pH and exposure time of this type of hydrogel are tested under different conditions [9-10]. When the copolymer contains a small number of metal acrylates, the activation energy depends on the conversion, which indicates many mechanisms of degradation. When the copolymer contains a large number of metallic acrylates, the activation energy increases and remains independent of conversion, indicating a degradation process through a single-step mechanism [11-12]. The differences between the nutrient compositions of the monomers bound to the copolymer chain were compared [13]. It is distinguished by its ease of use and versatility in the use of various monomer mixtures in the production of copolymers. The properties of copolymers derived from different monomers play an important role in the selection or development of a material for a specific purpose, and the rapid polymerization process plays an important role in the production of a number of materials

such as optical fiber coatings, photolithography and microelectronics [14 -15]. In our study, the mutual copolymerization properties and polymerization kinetics of the synthesized nickel acrylate and tin acrylate and methyl methacrylate monomers were compared with other acrylate monomers [16]. The synthesis of acrylate copolymers depends mainly on the composition of monomers, which corresponds to the catalytic activity of metal salts [17]. The composition of copolymers formed by the course of polymerization reactions is constantly changing, as the active monomer reacts more, and the copolymer is enriched with it [18]. Currently, acrylate copolymers of various compositions are used to obtain environmentally friendly materials [19]. The synthesized copolymers are characterized by properties such as good adhesion, flexibility, waterproofing and heat resistance [20-21]. Today, more than 50% of methyl methacrylate is used in the chemical industry in the production of acrylic copolymers [22]. The regulation of the synthesis conditions of copolymers used in production, as well as their use in a wide range of compositions, ie the combination in the macromolecular chain of monomer chains containing the -COOH and -O-CH3 groups allows obtaining materials with different hydrophobic properties [23]. The copolymer is also used in the restoration and conservation of archaeological sites [24]. The thermoplastic properties of acrylates, butyl methacrylates and methyl methacrylate monomer-based copolymers reveal the lacquer properties of polyacrylate-based copolymers, as the coating properties are preserved even at room temperature [25-26]. Given these properties, acrylic monomers have been studied for their application as paints [27]. It can also be used on the basis of the organometallic coordination chemistry of macromolecular compounds [28]. In our country, the demand for metal-containing copolymers in the manufacturing sector has increased significantly. This makes it possible to obtain promising nanocomposite materials [29]. Nickel and tin acrylate-based copolymers are used as additives in the preparation of paints for various structures (metal, ferrous metal, slate) to improve the quality of films and thickeners [30-31]. It can also be used, especially in the form of plastic sheets, cast powders, surface coatings, emulsion polymers, fibres, inks, latex paints, adhesives and films [32]. Simple methods can lead to the formation of heterometallic compounds with a regular structure between metallic binders and monomers [33]. Copolymers based on it are characterized by good adhesion and high heat resistance properties [34].

The purpose of our study

Synthesis of copolymers based on new nickel and tin acrylates, analysis of the composition, structure, thermal and physicochemical properties of the obtained copolymers using IR spectrum, TDA, TGA, SEM (scanning electron microscope) and elemental analysis. Also, study the properties of hydrogels.

Research methods and tools

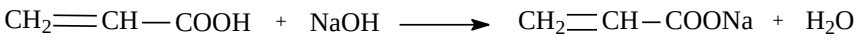
In this study, the results of the analysis obtained by IR-spectroscopic on SHIMADZU IR spectrophotometer and scanning electron microscope on MIRA 2 LMU SEM were used to study the synthesis of a new metal-containing copolymer. In addition, the results of the analysis obtained from the derivatogram of the differential thermal analysis have been used.

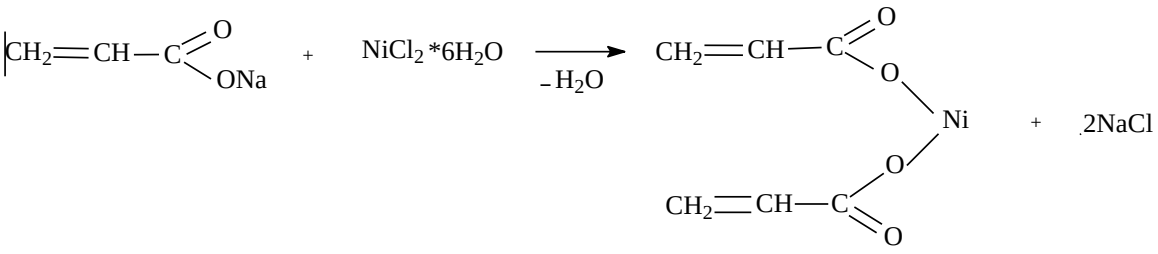
2.Experimental Part

2.1. Copolymer synthesis on the basis of the nickel acrylate

A three-mouthed 250ml flask equipped with an automatic stirrer, a return cooler and a thermometer were filled with 150ml of distilled water and 40g of acrylic acid. 16 g of alkali was then added to the mixture and mixed well. When the alkali was completely dissolved, 20 g of NiCl2 crystal hydrate was added. The tube was heated to 50–55 ° C in a thermostat by mixing and transferring nitrogen. After 60 min, 45 g of methyl methacrylate, as well as a solution of potassium persulfate (0.25 g in 10 ml of water) was added dropwise as the initiator. The temperature rose to 60-70 ° C. The reaction took place over a period of 3 hours, resulting in the formation of a light yellow

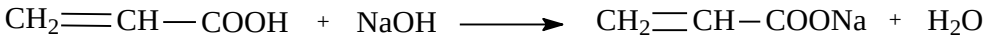
porous, viscous copolymer [35]. The physical properties of the new type of copolymer were analyzed, according to which the copolymer was dissolved in water, proved to have a very high viscosity, and its resistance to high temperatures and viscosity properties have been studied.

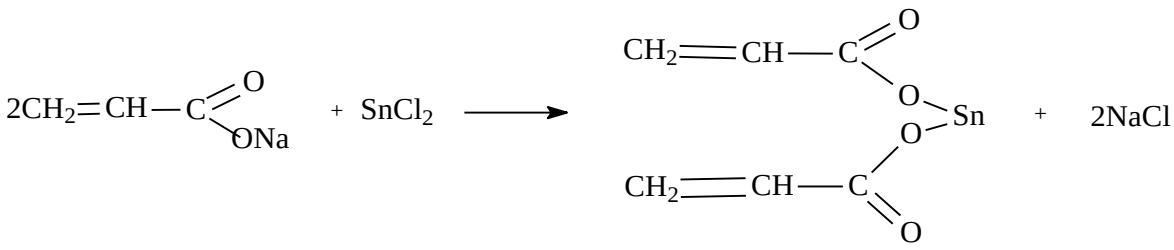




2.2. Copolymer synthesis on the basis of the tin acrylate

A round-bottomed flask with three nozzles, a return cooler, a mechanical stirrer and a thermometer was installed. 150 ml of distilled water and 49.7 g of acrylic acid were added dropwise to the flask and hydroquinone was added to the flask to prevent the acrylic acid from polymerizing rapidly. 16 g of sodium hydroxide were added to the flask while stirring. The solution was heated for one hour at a temperature of 35-400C until the alkali was completely dissolved, and after the alkali was completely dissolved 22 g of tin (II) chloride salt was added to it, the heating in the mixture was continued to 500C until it was completely dissolved. was brought. Once the tin salt was completely dissolved, 50 g of methyl methacrylate and 0.5 g of benzoyl peroxide were slowly added dropwise to the flask using a Buchner funnel. The temperature rose to 70 ° C. Copolymerization lasted 4 hours. The reaction resulted in the formation of a white copolymer with a porous structure. Dissolved in organic solvents, low solubility was observed. When the copolymer was dissolved in water, it swelled, absorbing water without dissolving.





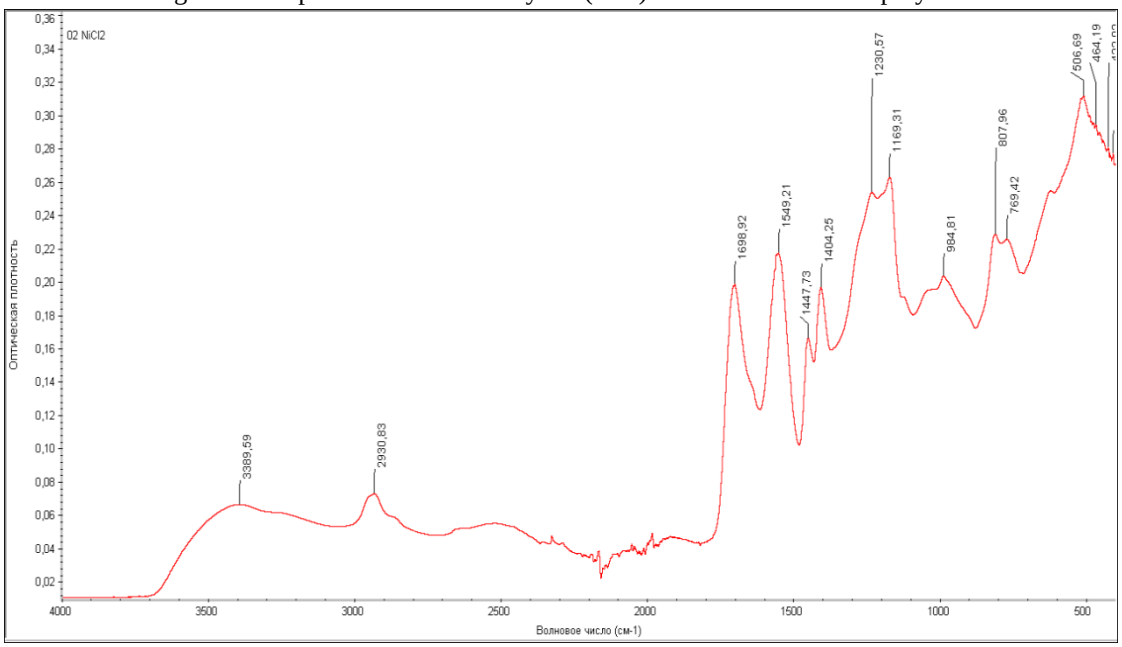
3.Result and its discussion

3.1. IR spectral studies of nickel acrylate on the basis of the copolymers.

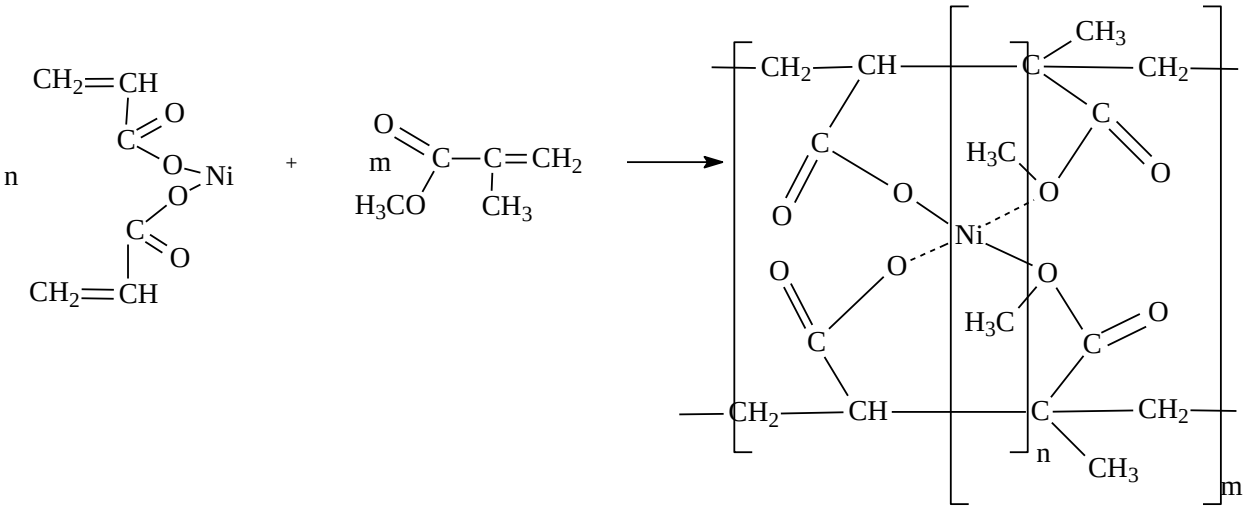
IR-spectral studies of nickel acrylate on the basis of the copolymers have been conducted at room temperature in the IR spectrum of SHIMADZU. The spectra are recorded at 400 to 4000 cm^{-1} at the same intensity and at the same scanning speed [36]. The IR spectrum of the synthesized product is given in (Figure 1). OH, valence oscillations in the high-frequency part of the spectrum were observed in the 3389.59 cm^{-1} regions. The mutual valence oscillations of the CH_3 and CH_2 groups in the monomers were observed in the area of 2930.83 cm^{-1}. The mutual elongation of C = O bonds of acrylic acid and methyl methacrylate is observed in the area of 1698.92 cm^{-1}. Activated C-O oscillations occurred in the frequency range 1442.73-1169 cm^{-1} of the carbonyl-oxygen bond of the carbonyl groups. The oxygen binding of nickel in nickel acrylate to methyl methacrylate in oxygen was

observed mainly in the donor-acceptor bond in the Ni-O bond characteristic frequencies in the 506.69-469.19 cm^{-1} regions.

Figure 1. IR spectrum of nickel acrylate (NiA) on the basis of the copolymer



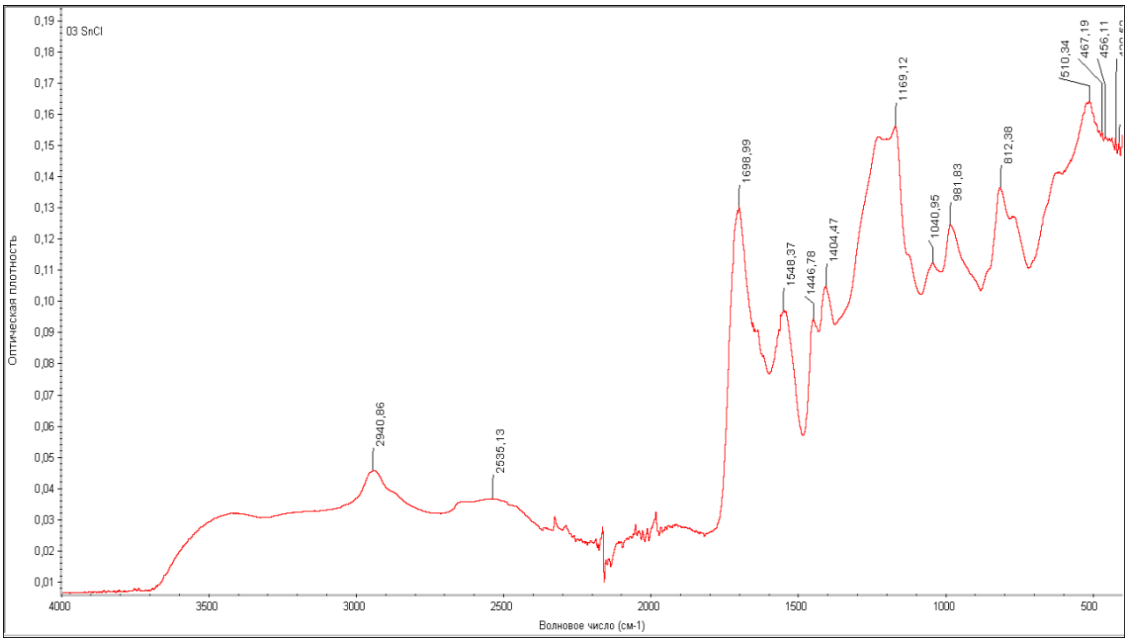
Depending on the results of the IR spectral analysis of the nickel acrylate (NiA) based copolymer obtained by synthesis, the copolymerization reaction proceeds as follows.



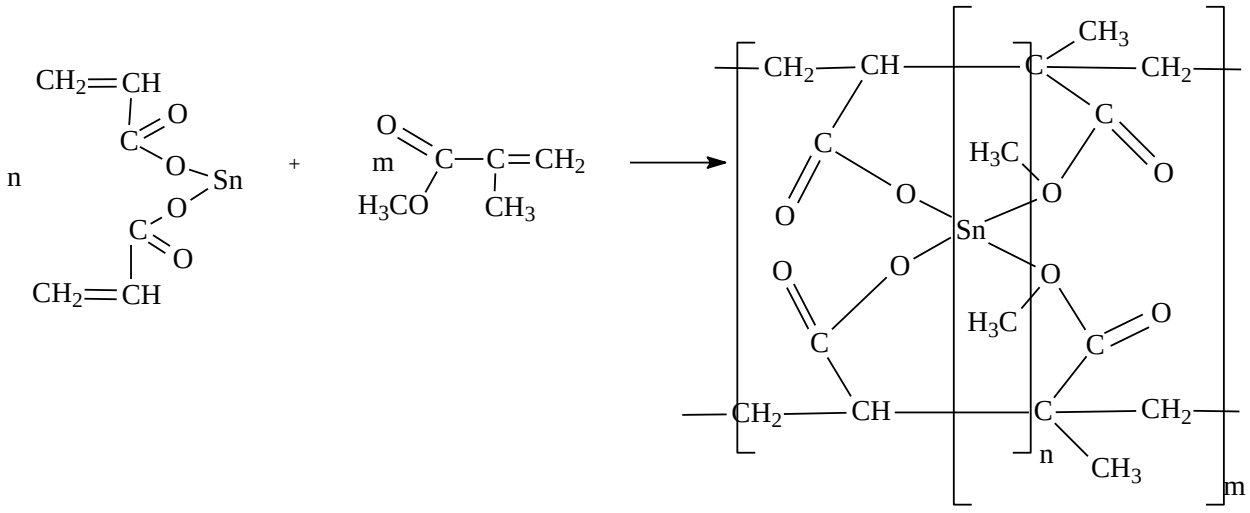
3.2. IR spectral studies of tin acrylate on the basis of the copolymers.

The IR spectrum of tin acrylate-based copolymer synthesis is shown in Figure 2, and the results of the analysis are as follows. Very high intensity of valence oscillation of the C = O group was observed in the area of 1698.99 cm^{-1}. The asymmetric and symmetric deformation oscillations of the CH_3 and CH_2 groups and the shear oscillations of the methylene group correspond to the range of groups in the range of 1446.78 -1404.47 cm^{-1}. Valence oscillations of the acrylate-containing $(COO)_2Sn$ group tin metal and the $-COOCH_3$ oxygen-containing methyl methacrylate group were observed in the 510–467 cm^{-1} region.

Figure 2. IR spectrum of the tin acrylate-based copolymer



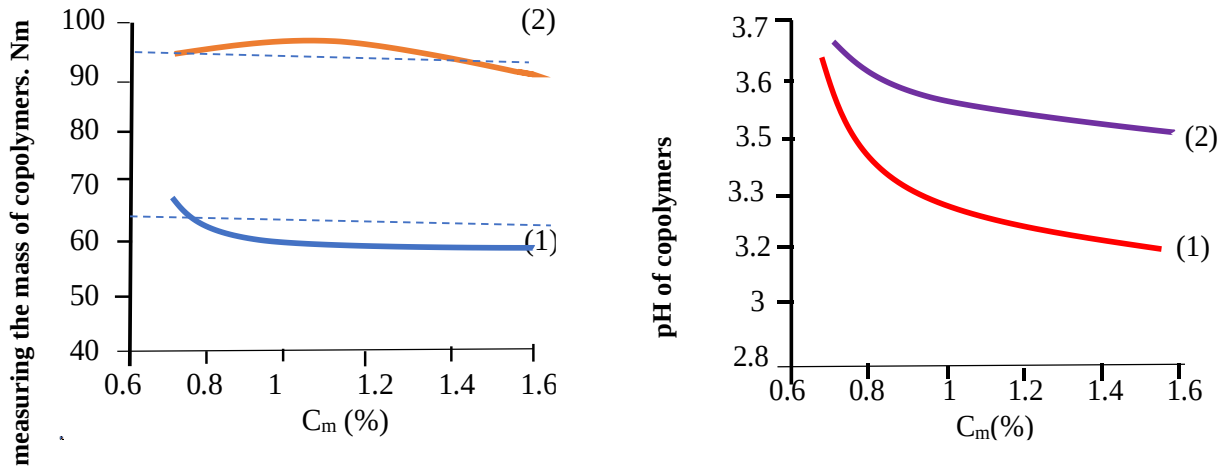
Depending on the results of the IR spectral analysis of the synthesized tin acrylate (SnA) copolymer, the copolymerization reaction is as follows.



3.3. Physicochemical, kinetic and technological properties of copolymers on the basis of the nickel and tin acrylates.

The ratio between the components of the polymerization system and the mixing rate of the synthesized nickel and tin acrylates-based copolymers also has a significant effect on the particle size of the metal. Figure 3 shows the mass values and pH values of copolymers of nickel and tin acrylates and methyl methacrylate formed under the conditions of polymerization.

Figure 3. Interaction of nickel and tin acrylates and methyl methacrylate under polymerization conditions and copolymer properties: (a) t; (b) pH (1). 400 K; (2). 450 K.

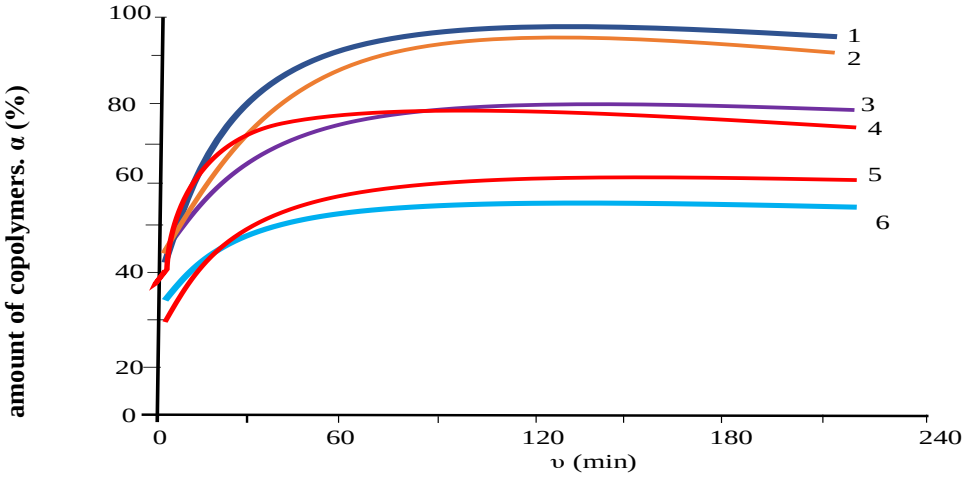


Depending on the nature of the methyl methacrylate monomer, and the mixing rate, the size of Ni and Sn acrylate particles is characterized by:

As the particle radius of nickel and tin acrylates increases, a decrease in their total amount may lead to an increase in the viscosity of the polymerization system due to an increase in the initial concentration of the methyl methacrylate monomer. A decrease in the mobility of radicals formed during the decomposition of potassium persulfate used as an initiator to accelerate the polymerization process of methyl methacrylate monomer to Ni and Sn acrylate is observed, resulting in a decrease in the initial amount of copolymer-monomer particles. It should also be noted that according to the classical theory of copolymerization when the conversion rate reaches 15-20%, the rate increases as a result of its constant decrease. This leads to an increase in the particle size of the synthesized copolymer and a change in their structure and properties.

Depending on the nature of the copolymers based on nickel and tin acrylate (NiA, SnA) and the conditions of its formation, the amounts of copolymers formed as a result of cross-polymerization of methyl methacrylate (MMA) in the presence of dispersed NiA, SnA monomer 4- shown in the figure .

Figure 4. The change in the number of copolymers relative to monomers over time, as well as the change in the amount of copolymer over time as a result of the interaction of mole ratios of NiA, SnA and MMA. NiA, SnA and MMA (1), (2), (4); MMA copolymer (3); mole ratio of copolymer to monomers: (1) - (3) 3: 1 (4) 1: 1. NiA, SnA, MMA, SP: (1), (3), (5) (6) 450; (2), (4), 482



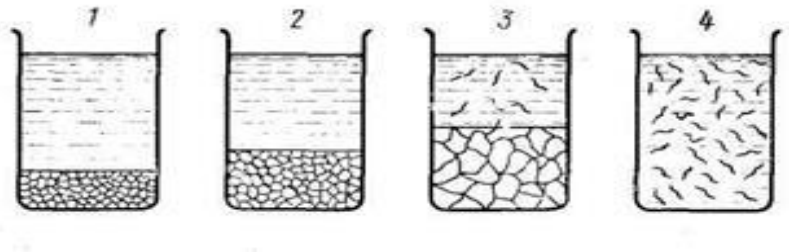
The rate of polymerization of Ni and Sn acrylate-based copolymers and the degree of conversion of monomers largely depend on the nature of the polymer matrix (Figure 4). MMA polymerization occurs at high speeds and, depending on the nature of the polymer matrix, the conversion rate of the monomers is achieved in 120 minutes. Ni, the maximum conversion rate and the highest polymerization rate are observed when using SnA-gr-MMA copolymer. The activity of the copolymer composition was also found to depend on its synthesis conditions [37].

3.4. The solubility and water absorption of copolymers based on nickel and tin acrylates have been studied.

Nikel va qalay akrilatlari asosidagi sopolimerlar metall tarkibiga qarab har xil eruvchanlik xususiyatlarini namoyon qiladi. NiA asosidagi sopolimer organik erituvchilarda kam eriydi, lekin suvda juda yaxshi eriydi va yopishqoqlikka ega. SnA sopolimeri dimetilformamidda erimaydi, lekin dioksanda yaxshi eriydi. Suvda erimaydi, lekin u suvni yutishi va shishish tezligi yuqori bo'lganligi isbotlangan.

Calculation of swelling of SnA copolymer in water. 1.5 g of SnA copolymer was added to 127ml of water and the swelling process in water was studied in approximately four steps. In the first (initial) stage, the system consists of two components: a polymer and a low molecular weight liquid. The second stage of the solution is the swelling process, as a result of which low molecular weight liquid molecules enter the polymer intensively, forming strong solvate complexes with it. In the third stage of copolymer melting, as the polymer swells, the size of the polymer and the distance between the macromolecules increase, individual macromolecules begin to separate, and a low molecular weight liquid layer is formed [38]. In the final (fourth) stage of the solution, the molecules of the high molecular weight substance are evenly distributed due to their dispersion into the low molecular weight solvent and form a true solution (Fig. 5).

Figure 5. Solubility and swelling of tin acrylate-based copolymer in water.

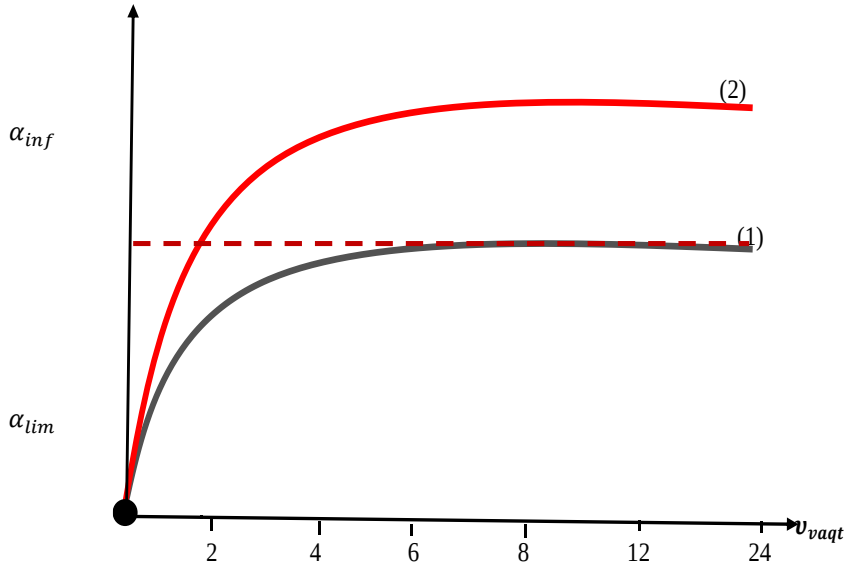


Calculate the swelling rate of a tin acrylate-based copolymer.

$$\alpha = \frac{(m-m_0)}{m_0} = \frac{128,5-1,5}{1,5} = 84,67$$

The limited and infinite state of the swelling rate of the SnA copolymer over time can be seen in the diagram below (Figure 6).

Figure 6. Time dependence of the kinetic curves of tin acrylate-based copolymer:1 - limited swelling; 2 - Infinite swelling



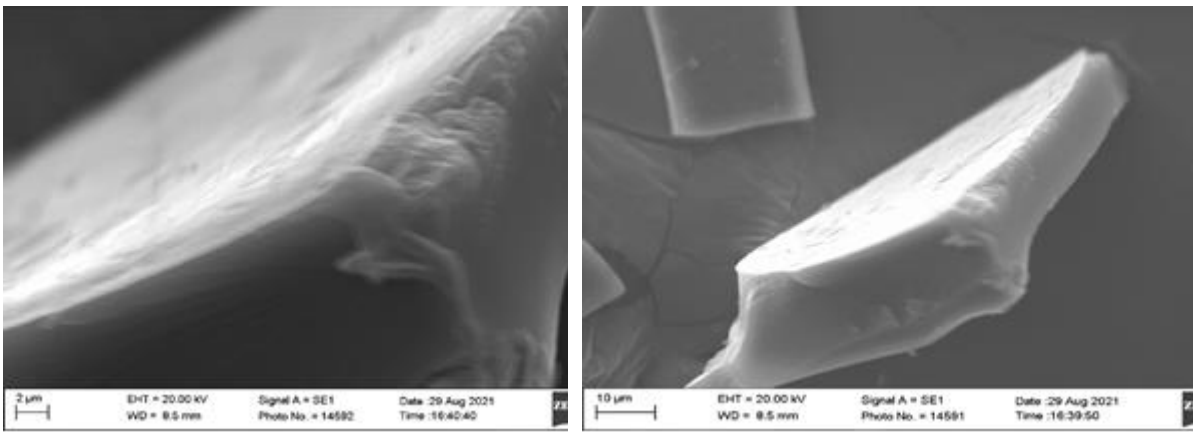
According to the results of studying the swelling rate of tin acrylate copolymer, we used it in the production of hydrogels because of the high water solubility of the resulting copolymer.

3.5. Analysis of nickel and tin acrylate-based copolymers under a scanning electron microscope.

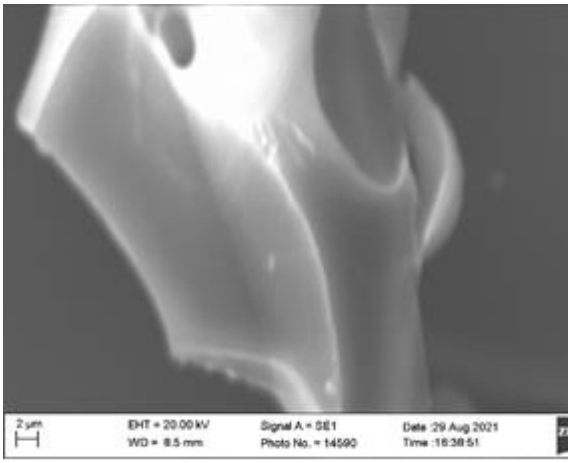
The synthesized copolymer has been analyzed by MIRA 2 LMU scanning electron microscopy. The results of the analysis show that the starting materials are completely dispersed. An image of the copolymer in 4 different sizes is shown in Figure 7, according to which the dimensions are a) 3000; b) 2000; d) 1000; c) magnified 500 times to describe the reaction rate of the substances, the composition and structure of the layers close to the surface. It can be seen from the obtained images that the high porosity of the copolymer crystals and the absence of additives increase the intensity of the copolymer.

Figure 7. Imaging of copolymers on a scanning electron microscope.

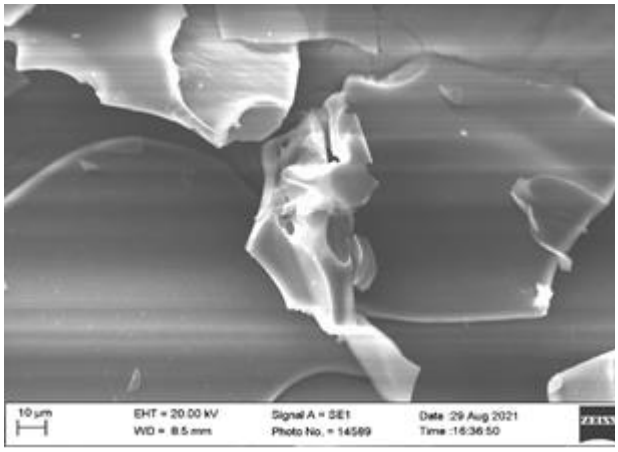
a) b)



d)

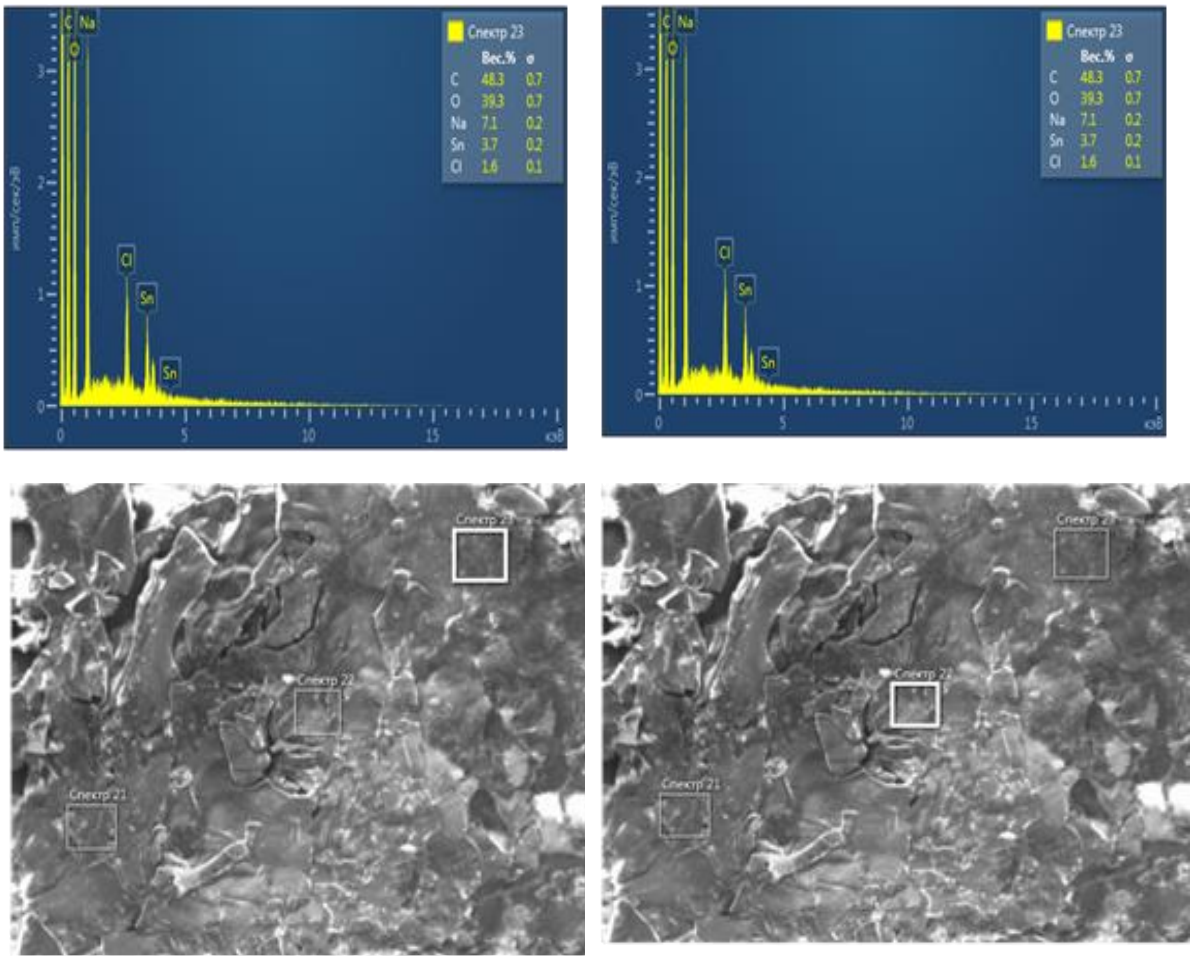


c)



Scanning electron microscope analyzes have been performed under a high vacuum. In the same device, microanalysis of the elements in the copolymer has been carried out in the fields with an accelerating voltage of 20 keV and a current of 1 nA. This allows us to know that it occurred at the end of the reaction, as well as the elemental composition of the substances formed in parallel in the reaction. (Figure 8).

Figure 8. Imaging of the copolymer in a scanning electron microscope and element analysis.



View the results of elemental analysis of the copolymer in tabular form.

Table-1.

Element	Weight%	Weight of sigma%
C	49.01	0.70
O	38.04	0.67
Na	6.92	0.19
Cl	2.10	0.09
Fe	0.26	0.08
Sr	0.24	0.10
Ni, Sn	3.44	0.18

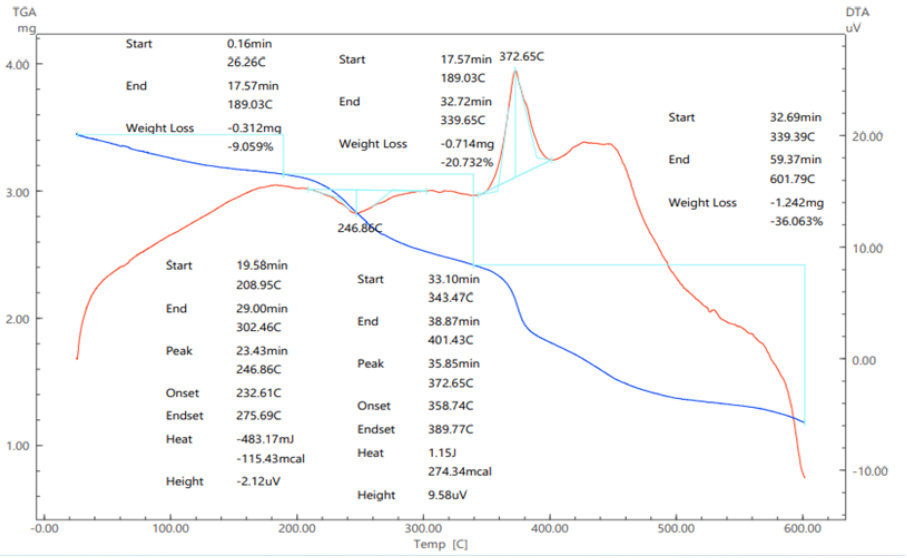
Several copolymer derivatives can be synthesized by replacing the three hydrogen atoms at the centre of the Ni and Sn acrylate-based copolymer molecule with almost all the metallic and nonmetallic ions of the periodic table. However, the thermal conductivity properties of copolymers are altered by electron donors or electron acceptor groups.

3.6. Thermal analysis of Ni and Sn acrylate-based copolymers.

Thermal analysis of nickel and tin acrylate-based copolymer has been studied and analyzed at 20–602ºC.

3.6.1. 4.56 mg of the copolymer was obtained for thermogravimetric analysis of Ni acrylate-based copolymer synthesis and the process has been studied in the temperature range 20–600ºC. the results of the thermal analysis of the copolymer are shown in Figure 9**.**

Figure 9. Thermogravimetric (TGA) and Differential Thermal Analysis (DTA) of nickel acrylate-based copolymer synthesis



In the DTA analysis of the synthesized polymer based on the synthesized Ni acrylate, the mass loss occurred in three areas and one exothermic and one endothermic process. The first phase of decomposition of the substances started at 26.26°C and 0.312 mg or 9.059% mass loss was observed at 189.03°C. This decomposition is explained by the release of bound water in the form of crystal hydrate.

The second phase started at 189.03°C and ended with 0.714 mg, 20.732% mass loss at 339.65°C. At these temperatures, carbon dioxide is released as a result of the decomposition of carboxyl groups.

The third stage was the main decomposition stage, starting at 339.39°C and a loss of 1.242 mg or 36.063% mass was observed at 601.79°C. Oxides remain from the decomposition of organic compounds and from the decomposition of carbon and metal carbonates.

In the DTA analysis of the synthesized product, heat absorption i.e. decomposition of substances at 246.89°C in the endothermic process was observed. During this process, carbon monoxide decomposes. It is observed with the release of heat (exothermic) at 372.65°C, which forms metal carbonates and carbides.

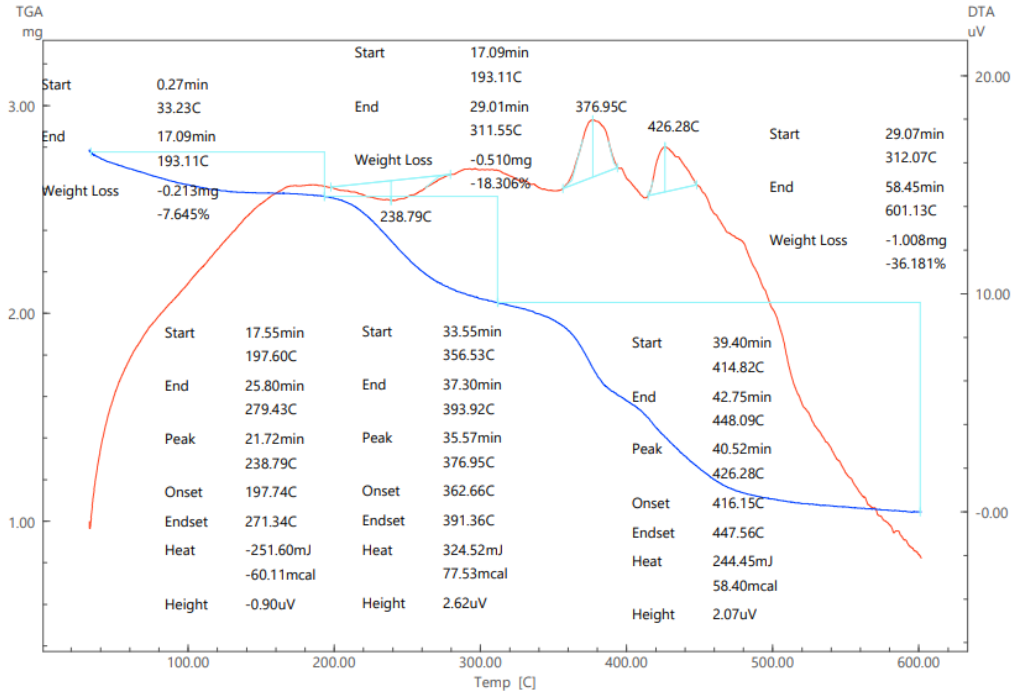
Results analysis of TGA and DTA curves of Ni acrylate copolymer

Table-2.

№	Temperature °C	Lost weight,mg (4.56)	Lost weight, %
1	100	0.245	5,37
2	200	0.312	6,84
3	300	0,714	15,66
4	400	1,242	27,23
5	500	2,56	56,14
6	600	3,241	71,08

3.6.2. 3.82 mg of the copolymer for the analysis of thermogravimetric analysis of Sn acrylate-based copolymer synthesis. was obtained and the process has been studied in the temperature range of 20–600°C. The results of the thermal analysis of the copolymer are shown in Figure 10.

Figure 10. Thermogravimetric (TGA) and Differential Thermal Analysis (DTA) of Sn acrylate-based copolymer synthesis



In the DTA analysis of Sn acrylate-based copolymer synthesis, the weight loss occurred in three areas and two exothermic processes occurred.

In the first decomposition phase of the substances obtained for testing, a process of 33.23°C was initiated and 0.213 mg or 7.645% weight loss has been observed at a temperature of 193.11°C. This decomposition is explained by the leakage of water containing alkali.

The second phase of the process began at 193.11°C and ended with a loss of 0.510 mg, or 18.306%, at a temperature of 311.55°C. At these temperatures, carbon dioxide is released as a result of the decomposition of carboxyl groups.

The third stage of the process was the main decomposition stage, starting at 312.07°C and a loss of 1.008 mg or 36.181% of the mass was observed at 601.13°C. This leaves metal carbonates from the decomposition of organic compounds and oxides from the decomposition of carbon.

In the DTA analysis of the synthesized product, heat is released at a temperature of 376.95°C in the first stage of the exothermic process, which results in the formation of carbon and metal oxides. The second stage of the exothermic process is observed at a temperature of 426.28°C, which results in the formation of metal carbonates and carbides.

Results analysis of TGA and DTA curves of Sn acrylate-based copolymer

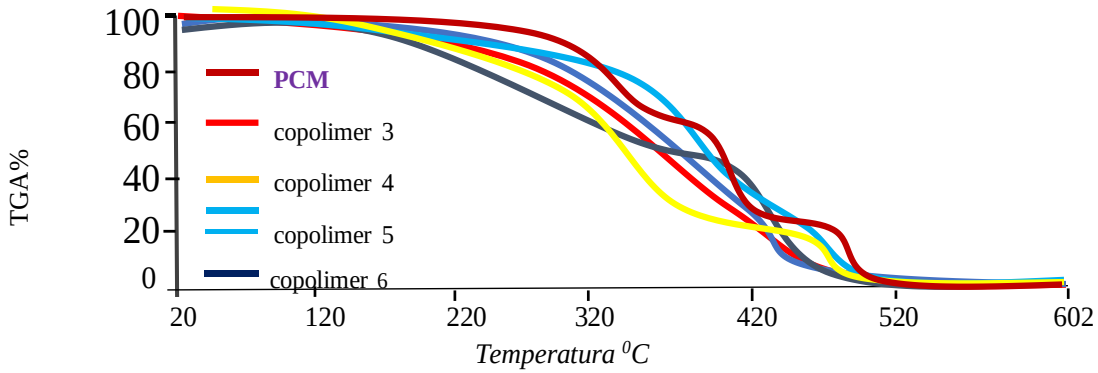
Table-3.

№	Temperature,°C	Lost weight,mg (3,82)	Lost weight, %
1	100	0,213	5,61

2	200	0,510	13,35
3	300	0,895	29,42
4	400	0,754	43,063
5	500	1,008	64,869
6	600	0.44	82,329

The heat resistance properties of copolymer materials on the basis of the nickel and tin acrylates obtained have been studied using the STA method. The TG and DTG curves for copolymer materials at 20–600°C are shown in Figure 11 [39-40].

Figure 11. TGA / DTG curves (20-602°C) for copolymer materials on the basis of nickel and tin acrylates.



Studies have shown that the obtained Sn acrylate-based copolymer is well soluble in water and the obtained copolymer can be used in the production of hydrogels. The heat resistance of Ni acrylate-based copolymer was found to meet the specified requirements. Given these properties, it has been applied to production as heat-resistant coatings.

Conclusion

The formation of a copolymer on the basis of the synthesized nickel and tin acrylates has been proven to have functional groups in the IR spectrum of the product and explains its good quality. The technological and kinetic properties of the nickel and tin acrylate-based copolymer were good, and the SEM analysis was analyzed by spectral analysis and comparison with existing copolymers. The thermogravimetric analysis of the copolymer revealed that it was thermally stable. It was also studied for its solubility and heat resistance properties in various solvents. The synthesized copolymer has unique properties and was synthesized in a new way, which allows achieving high economic efficiency in the process of copolymer synthesis. The possibility of using the synthesized copolymer in practice as heat-resistant foams and films and as a hydrogel was studied.

Studies have shown that the resulting tin acrylate copolymer has been used in the production of hydrogels due to its high solubility in solvents. The heat resistance of nickel acrylate copolymer was found to meet the specified requirements. Given these properties, it has been applied to the production of heat-resistant materials, i.e. foams.

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