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SYNTHESIS OF POLYVINYLPYROLIDONE COPOLYMERS IN THE PRESENCE OF TWO-COMPONENT INITIATION SYSTEMS

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The paper presents the results of the study of copolymerization of 2-hydroxyethylmethacrylate (HEMA) with polyvinylpyrrolidone (PVP) under the action of two-component initiation systems of iron (II) sulfate/radical type initiator. The influence of the nature of the radical initiator (In) in the $FeSO_4$/In system on the behavior of the polymerization of HEMA/PVP compositions, structural parameters of the polymer matrix network and properties of hydrogels based on pHEMA-gr-PVP copolymers were established.

Key words: polyvinylpyrrolidone; 2-hydroxyethylmethacrylate; hydrogel; polymerization; initiator; iron sulfate; graft copolymer; crosslinked polymer; polymer network.

Introduction

In recent years, the synthesis and research of polymer hydrogel materials is one of the priority areas of polymer science and practice, which is actively developing. Hydrogels are characterized by high sorption capacity for low molecular weight substances, permeability to liquids and gases, which are a prerequisite for their use in various fields of application [1–5]. Such materials have acquired special practical application in medicine and biotechnology [6–12]. Among a wide range of polymers for biomedical purposes, copolymers based on (meth)acrylic esters of glycols with polyvinylpyrrolidone (PVP) are promising. These materials are characterized by technological advantages of obtaining and a combination of unique properties, which opens the prospect of their use, for example, as hydrogel medical dressings [14], contact lenses [15], hydrogel membranes [16], materials for regeneration of damaged tissues [17], as enzyme immobilization systems [18], drug delivery systems [19], vascular prostheses [20] restoration of works of art paintings [21], electrically controlled elements of optical systems [22], for conductometric moisture gages [23] etc.

Preliminary studies conducted by the staff of the Department of Chemical Technology of Plastics Processing in Lviv Polytechnic National University [24–27] developed hydrogels based on PVP copolymers with 2-hydroxyethylmethacrylate (HEMA), polymerization of which occurs under the action of metal ions of variable oxidation state. Polymerization occurs at high speed at room temperature, in air, which greatly simplifies and reduces the cost of the process, reduces its duration and expands the possibilities of its use. Depending on the composition formulation, nature and content of the solvent, the nature and concentration of the metal salt, the duration of formation (with a limiting yield of polymer 98–99 %) of such compositions can be varied within wide limits – from 15 to 150 min. It was found, that the most effective in terms of the rate of polymerization of the studied metal salts is iron sulfate (II) [26]. Copolymers of pHEMA-gr-PVP have a spatially crosslinked structure formed by blocks of HEMA grafted on a PVP matrix and contain hydrophilic groups: hydroxyl and carbonyl HEMA, as well as peptide PVP. The number of such groups, as well as the unique porous structure provide high permeability to low molecular weight compounds and swelling of pHEMA-gr-PVP copolymers in water and are the main factors that determine their distribution and use. At the same time, the improvement of synthesis methods and the structure of hydrogel materials largely depends on the nature of the initiation system. The choice of the initiation system is a necessary condition for the process of polymer synthesis, as the initiator affects the behavior of the polymerization process and the formation process of a polymer matrix, thereby affecting its structure and, consequently, the properties [27–31]. At the same time, the choice of the initiation system largely determines the technological features of obtaining materials, namely the pot life of HEMA/PVP compositions and the duration of formation of products based on pHEMA-gr-PVP copolymers and their hydrogels.

The aim of the research

The aim of the study was to investigate the influence of the nature of the two-component initiation system on the features of the synthesis of pHEMA-gr-PVP copolymers.

Materials and methods of research

The following substances were used: 2-hydroxyethylmethacrylate (Sigma Chemical Co), which was purified and distilled in vacuum (residual pressure = 130 N/m^2, T_B = 351 K); polyvinylpyrrolidone (AppliChem GmbH) of high purity with MM 28000 was dried at 338 K in vacuum for 2–3 hours before use; iron (II) sulfate was used of p.a. grades., benzoyl peroxide (PBO) and azobisisobutyronitrile (AIBN) were purified by recrystallization from ethanol, potassium persulfate (KPS) was recrystallized twice from aqueous solution. Obtaining of PVP with HEMA and hydrogel materials based on them were carried out according to the method described in [24]. In order to combine the stages of synthesis of the hydrophilic polymer and its subsequent swelling, the polymerization of HEMA in the presence of PVP was performed in a solvent at 293 K, in air, in daylight. The kinetics of polymerization of the composition was studied by changing the volume of the reaction mixture during polymerization of the monomer by dilatometric method [32, 33]. The amount of unbound PVP in the polymer network was determined by photocolorimetry of the aqueous extract [24]. Grafting efficiency (*f*, %) was calculated as the ratio of the amount of grafted PVP to the total amount of PVP in the original composition, the grafting degree (*p*, %) – as the ratio of the amount of grafted PVP to the total weight of the copolymer [24]. The molecular weight between cross-links in the polymer network (*Mc*, kg/mol) was determined by the equilibrium modulus of high elasticity [24].

Water content (*W*, wt.%) was investigated by weight method of difference between the mass of dry and swollen samples, the swelling coefficient (*k*) – by changing the size of dry and swollen samples [24]. Strain-resilience characteristics: the hardness number (*H*, MPa) and the elasticity index (*E*, %) were determined on the hardness meter TShR-320 by measuring the difference between the depth of immersion of the indenter in the swollen sample under the action and after removal of the load [24].

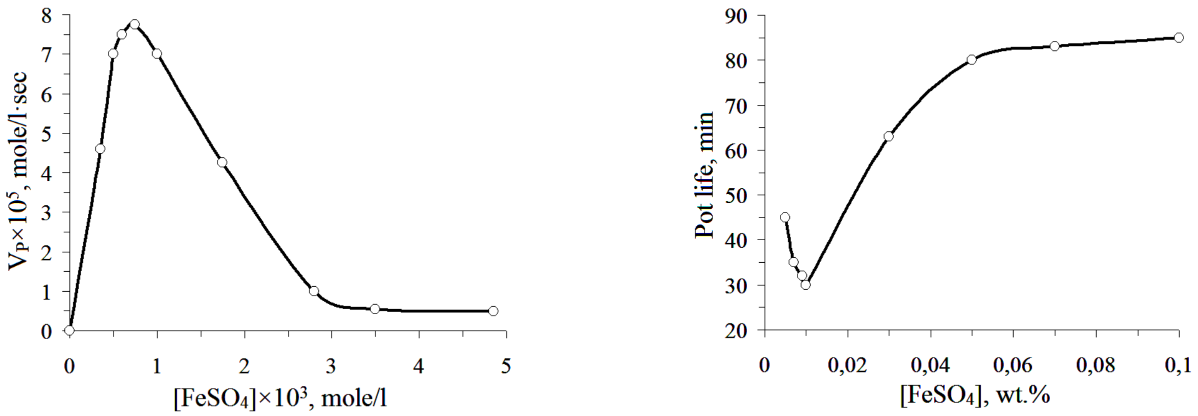
Research results and their discussion

In addition to the formation of the structure and performance properties of copolymers, the efficiency of the initiation system will primarily determine the rate of the polymerization process, which, in its turn, will determine the technological characteristics of HEMA/PVP compositions, in particular – pot life, i.e. the period of time in a viscous-flow state, and the duration of the formation of hydrogel materials. It is established that the copolymerization of HEMA with PVP in the presence of ions of metals of variable oxidation state occurs by a complex-radical or ionic mechanism [35]. The most effective in terms of polymerization rate of the studied metal salts are iron salts (II, III). Depending on the composition formulation, content, nature of the solvent and the concentration of the metal salt, the pot life of such compositions can be varied widely – from 5 to 90 minutes, the duration of formation (with a limiting yield of polymer of 98–99 %) – from 15 to 150 minutes. Compositions with a higher PVP content and a minimum solvent content or those obtained by mass polymerization are characterized by the highest polymerization rate [26]. It is found, that with increasing PVP content more than 20 mass fractions, compositions become non-technological – they are characterized by high viscosity and there are difficulties with the removal of air bubbles, which requires additional vacuumation. Polymerization in solution in most cases is a necessary condition for several reasons: the presence of solvent increases the fluidity of compositions with higher PVP content, hydrogel materials for metal reduction should be operated in a swollen state, furthermore the presence of solvent significantly increases the porosity of hydrogels.

In order to intensify the polymerization process, taking into account the ion-radical mechanism of copolymerization of HEMA with PVP, in the work a two-component initiating system was used to initiate polymerization – metal salt of variable oxidation state (iron sulfate (II) with traditional radical initiators of polymerization of acrylic monomers – BPO, KPS and AIBN. The content of the components of the initiation system was selected on the basis of previous kinetic studies [26, 35]. It was found that with increasing $FeSO_4$ content, the rate of polymerization of HEMA/PVP compositions in the mass increases, but in the case of polymerization in solution – has an extreme behavior with a maximum value for $[FeSO_4]$ = 0,01 wt. % (Fig. 1, *a*) [26]. The change of pot life on salt content has the same dependence (Fig. 1, *b*). Therefore, the concentration of $FeSO_4$ – 0.01 wt. % from the formulation of original composition was selected for research.

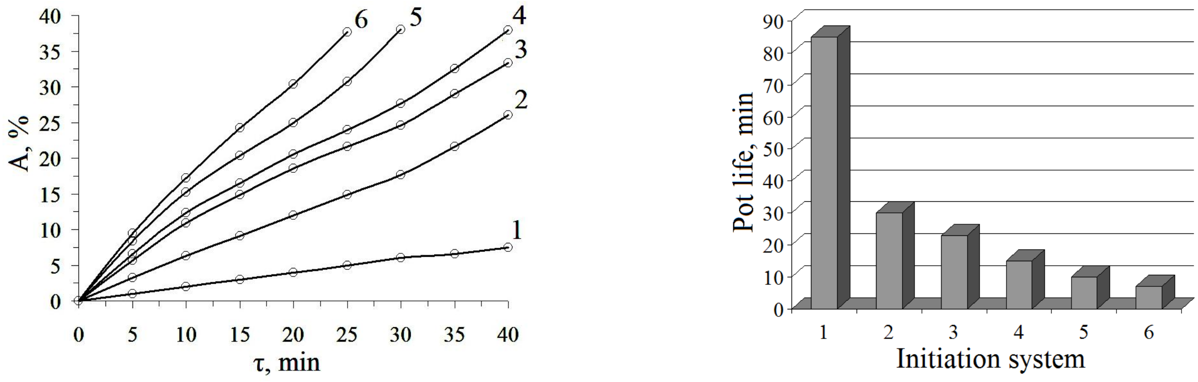
Therefore, for the research was chosen the concentration of $FeSO_4$ – 0,01 wt. % from the formulation of the original composition, the ratio of $FeSO_4$: initiator – 1 : 1.

It was found with a study of the kinetics of polymerization in the initial stages in the presence of a two-component initiation system, that with the usage of radical initiators BPO, KPS and AIBN, in each case the rate of polymer formation increases compared to $FeSO_4$ (Fig. 2, *a*), in particular, the process rate increases in a row: $FeSO_4$ – $FeSO_4$/AIBN – $FeSO_4$/BPO – $FeSO_4$/KPS. Naturally, the pot life of composition changes in similar way (Fig. 2, *b*). The results of kinetic studies showed, that the addition of radical-type initiators increases the rate of the polymer formation process, which is an additional confirmation of the proposed from the previous works – the ion-radical mechanism of the copolymerization process of HEMA with PVP [27, 35].



a *b*

Fig. 1. Effect of $FeSO_4$ concentration on the rate of copolymerization (a) and pot life (b) of HEMA/PVP compositions (T = 298 K, MM_{PVP} = 28000)
Composition formulation, mass parts: a – HEMA:PVP:H_2O = 70:30:100; b – HEMA:PVP:H_2O = 80:20:100



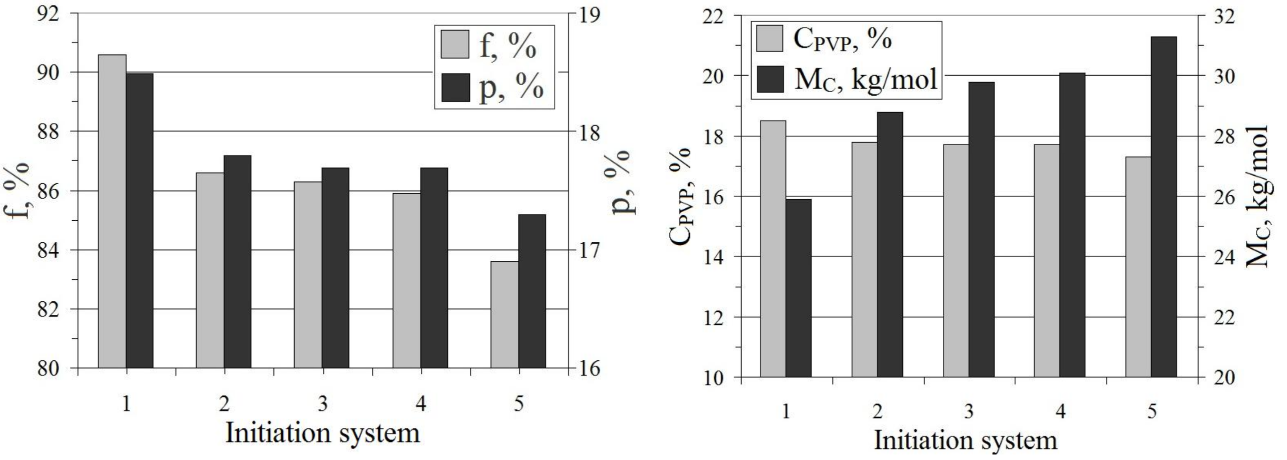
a *b*

Fig. 2. Effect of the composition of the initiation system on the monomer conversion A, % (a) and pot life (b) (HEMA:PVP:H_2O = 80:20:100 mass parts; $[FeSO_4]$ = 0,01 wt.%; $FeSO_4$: In = 1 : 1; T = 298 K):
1 – $FeSO_4$ (0,1 %); 2 – $FeSO_4$; 3 – $FeSO_4$ (0,1 %)/BPO; 4 – $FeSO_4$/AIBN; 5 – $FeSO_4$/BPO; 6 – $FeSO_4$/KPS

With an increase in the concentration of $FeSO_4$ by 10 times (Fig. 3, *a*, curve 3) there is no sharp decrease in the rate of polymerization compared to pure $FeSO_4$ (curve 1), which indicates the advantage in this case of the radical component of the polymerization mechanism.

The high hydrophilicity and sorption capacity of PVP-containing hydrogels relative to low molecular weight compounds were determined both by the ability of PVP to form various complexes with many compounds and the formed structure of the spatially crosslinked copolymer containing PVP units. The formation of the ternary complex between HEMA, PVP and Fe^{n+} ions, the course of grafted polymerization and the formation of spatially crosslinked pHEMA-gr-PVP copolymer were confirmed by IR spectroscopy, conductometry, DTA and TGA [24, 26, 27]. However, not all polyvinylpyrrolidone was involved in graft polymerization. Unreacted PVP can be washed away during hydration, which affects the physical and mechanical properties of hydrogels such as water content, sorption capacity and permeability. Therefore, of practical interest are studies of the influence of the initiation system nature on the amount of PVP that reacted to the formation of the copolymer.

As can be seen from the obtained results (Fig. 3, *a*), the presence of radical-type initiators leads to a decrease in the efficiency (*f*, %) and the degree of grafting (*p*, %) of PVP and its content (C_{PVP}, %) in the copolymer (Fig. 3, *b*).



a *b*

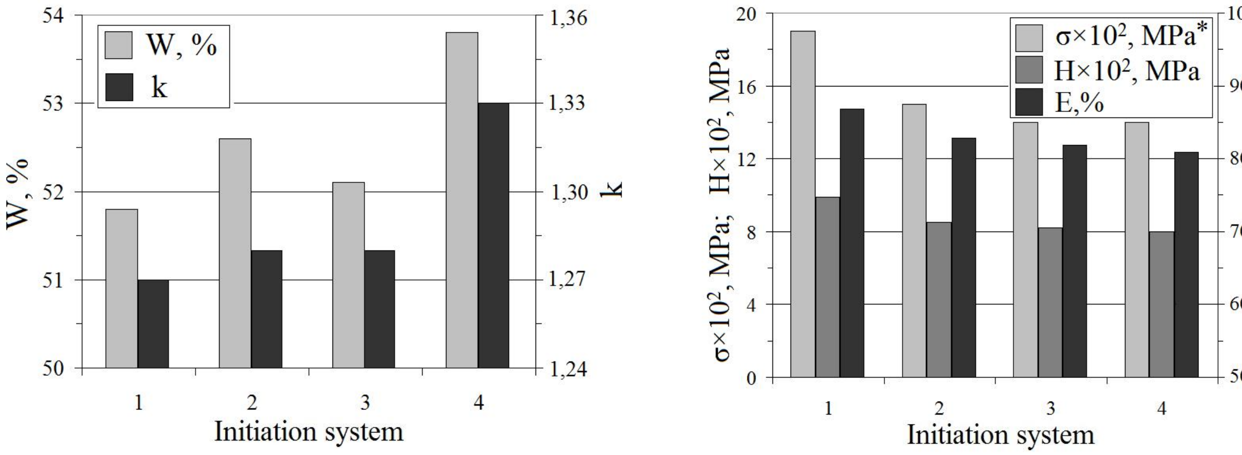
Fig. 3. Effect of the initiator nature on the efficiency, grafting degree (a) and content of PVP in the copolymer, molecular weight of the internodal segment (b) (HEMA : PVP:H_2O = 80 : 20 : 100 mass parts; [$FeSO_4$] = 0,01 wt. %; $FeSO_4$: In = 1 : 1): 1 – $FeSO_4$; 2 – $FeSO_4$ (0,1 %)/BPO; 3 – $FeSO_4$/AIBN; 4 – $FeSO_4$/BPO; 5 – $FeSO_4$/KPS

The difference in the parameters of copolymers synthesized in the presence of two-component initiation systems is also found in the structural characteristics of their network, the degree of crosslinking of which is characterized by the molecular weight of the internodal segment (M_C, kg/mol) (Fig. 3, *b*).

The possibility of practical use of hydrogel materials based on pHEMA-gr-PVP copolymers is largely determined by the performance properties of the polymer matrix.

To compare the influence of the composition of initiation system on the properties of hydrogels, their physico-mechanical characteristics such as tensile strength, hardness number, elasticity index, plasticity number, water content and swelling coefficient were studied (Fig. 4).

According to the results of research, adding of the additional initiators to the composition in each case leads to a change in the properties of hydrogel materials. At the same time, there is an improvement in the sorption capacity of hydrogels relative to water and a decrease in physico-mechanical properties – a decrease in tensile strength, hardness number, elasticity of samples and increase of their plasticity. Obviously, this is caused due to the changes in both the composition and structure of the copolymers. Studies of the structural parameters of copolymers (Fig. 3) have shown that the use of double initiation systems reduces the effectiveness of PVP grafting, which is washed away from the copolymer during hydration, reduces the degree of crosslinking of the copolymer, which is the cause of increase of water content and swelling coefficient. However, the looser polymer is characterized by fewer crosslinking nodes that take the load, resulting in a decrease in strength and elasticity properties.



a *b*

*Fig. 4. Effect of the initiation system nature on the properties of copolymers (HEMA : PVP : H_2O = 80 : 20 : 100 mass parts, [$FeSO_4$] = 0,01 wt. %, $FeSO_4$: In = 1 : 1; *– for film samples): 1 – $FeSO_4$; 2 – $FeSO_4$/AIBN; 3 – $FeSO_4$/BPO; 4 – $FeSO_4$/KPS*

Conclusions

Therefore, the obtained results showed the many-sided effect of two-component initiation systems on the synthesis process of pHEMA-gr-PVP copolymers and their properties. It was found that the increasing the rate of polymerization and, accordingly, such technological characteristics as pot life and duration of formation of hydrogel products based on pHEMA-gr-PVP copolymers, is accompanied by improved sorption properties, however, the loss of strength and elasticity of hydrogels are observed. Therefore, the choice of the initiation system must be made depending on the operating conditions of the hydrogel material in a particular product, predicting its properties. The obtained research results will be used in the development of technology for obtaining pHEMA-gr-PVP copolymers and composite metal-filled hydrogels based on them by the method of chemical reduction of metal ions in the polymer network.

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СИНТЕЗ КОПОЛІМЕРІВ ПОЛІВІНІЛПІРОЛІДОНУ В ПРИСУТНОСТІ ДВОКОМПОНЕНТНИХ ІНІЦІЮВАЛЬНИХ СИСТЕМ

Викладено результати дослідження кополімеризації 2-гідроксіетилметакрилату (ГЕМА) з полівінілпіролідоном (ПВП) під дією двокомпонентних ініціювальних систем феруму (ІІ) сульфат/ініціатор радикального типу. Встановлено вплив природи радикального ініціатора (In) в системі $FeSO_4$/In на перебіг полімеризації ГЕМА/ПВП композицій, структурні параметри сітки полімерної матриці та властивості гідрогелів на основі пГЕМА-пр-ПВП кополімерів.

Ключові слова: полівінілпіролідон; 2-гідроксіетилметакрилат; гідрогель; полімеризація; ініціатор; феруму сульфат; прищеплений полімер; просторово зшитий полімер; полімерна сітка.