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Research Article

Hydrophilic Polyacrylamide Gels Modified with Poly-N-vinylpyrrolidone

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Abstract

In modern medicine, there is a need to create biologically active polymer materials for the controlled release of drugs with improved characteristics compared to existing materials. Hydrogels are an ideal basis for creating such materials due to their high water content, excellent biocompatibility, and soft consistency, which is similar to that of body tissues. Therefore, this study aimed to develop and investigate hydrogels modified with poly-N-vinylpyrrolidone (PVP) for their potential use as drug carriers. A series of polyacrylamide gels (PAAGs) were synthesized, modified with PVP at concentrations of 0.3 wt.%, 0.5 wt.% and 1.0 wt.% relative to the weight of acrylamide. The synthesis was performed through free radical polymerization of acrylamide, N,N'-methylene-bis-acrylamide, and PVP in a sodium phosphate buffer solution (pH 8.0–8.5) in the presence of an oxidation-reduction initiation system (ammonium persulfate and N,N,N′,N′-tetramethylethylenediamine). The structural characteristics, water absorption, rheological, and sorption properties of these hydrogels were analyzed. IR spectroscopic analysis revealed that the incorporation of PVP into the PAAG structure leads to alterations in the hydrogen bonding of NH groups within the polymer matrix and changes in the skeletal chain, associated with varying contents of CH, CH_2, CH_3 groups. Rheological studies demonstrated that PVP-modified hydrogels exhibit a reduction in viscosity by 22.4-35.3% compared to unmodified PAAGs, depending on the PVP content. Despite this decrease in viscosity, the structural stability remained sufficient for the hydrophilic matrix to function effectively in drug immobilization. The water absorption studies indicated absorption values ranging from 761.9 to 1059.8%. Sorption properties were assessed using the drug dacarbazine, revealing that increasing the PVP content in the hydrogel to 0.5 wt.% and 1.0 wt.% enhanced the sorption capacity of PAAGs by 21.1% and 27.1%, respectively. Thus, the synthesized hydrogel materials exhibit sorption capacity for dacarbazine and demonstrate high water absorption values, indicating that they are promising materials for use as drug carriers in medical practice; thus, they require further medical and biological research.

Keywords

Polyacrylamide Gels, Poly-N-vinylpyrrolidone, Hydrophilicity, Sorption, Dacarbazine, Drug Delivery Systems

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1. Introduction

In recent years, the synthesis of biologically active polymer materials for the controlled release of drugs based on hydrogels has attracted the attention of many scientists due to their hight water content, excellent biocompatibility, and soft consistency, which is similar to the body tissues [1, 2]. In addition, hydrogels are capable of integrating with the surrounding biological tissues [1].

Hydrophilic gels, known as hydrogels, are cross-linked polymer networks that can absorb large amounts of water or biological fluids. Due to their soft consistency, porosity and highwater content, they imitate natural living tissues better than any other class of synthetic biomaterials. The ability of hydrogels to absorb water results from the presence of hydrophilic functional groups attached to the main polymer chain, such as –OH, –CONH–, –$CONH_2$–, and –SO_3H. The water inside the hydrogel allows free diffusion of dissolved molecules, while the polymer serves as a matrix that holds the water together. Additionally, the polymer network macro chains in the gel are interconnected, forming a single large molecule on a macroscopic scale. A hydrogel is a state that is neither completely liquid nor completely solid. Because of these properties, hydrogels are considered unique materials [3].

Hydrogels have been successfully used for several decades in tissue engineering as implant materials [1, 4, 5], drug delivery systems [6, 7], and for the sustained release of genes and cells [8, 9]. They have also been used as injectable treatments, particularly for stress urinary incontinence in women [10], as extracellular matrices for *in vitro* studies of cell behavior [11], and in other biomedical applications, including wound [12, 13] and burn [14] treatments, as well as in the manufacture of contact lenses [15, 16] etc.

Polyacrylamide-based hydrogels are widely utilized and have a various application, including drug delivery, plastic surgery, and ophthalmology. Polyacrylamide hydrogel are non-toxic, stable, non-absorbable, sterile aqueous gels consisting of cross-linked polyacrylamide and water [17].

Semi-interpenetrating polyacrylamide-gelatin cross-linked hydrogels with a gelatin content of up to 0.3% are known to be biocompatible (with fibroblast viability exceeding >70%) and hemocompatible, as they do not cause erythrocyte lysis. The synthesized hydrogels are promising candidates for soft tissue engineering applications, serving as biosynthetic extracellular matrices for *in vitro* cell behavior studies [11].

Polyacrylamide hydrogels have been developed as coatings for subcutaneously implanted devices to reduce foreign body reaction and local tissue inflammation. Applying polyacrylamide gels as thin coatings on polydimethylsiloxane disks or silicone catheters significantly enhanced their biocompatibility. Insulin infusion catheters coated with this hydrogel have demonstrated improved biocompatibility and extended service life [18].

Researchers [4] have developed hybrid polyacrylamide hydrogel implants capable of sustained release of anticancer drugs (chlorhexidine, 5-fluorouracil and doxorubicin), maintaining effective concentration at the site of the disease. There are also hybrid polymer systems based on pH-sensitive polyvinyl formal hydrogels and gold nanoparticles designed for filling postoperative cavities, particularly after tumor resection, with simultaneous targeted release of the bacteriostatic agent albucid [19].

The chemical structure of hydrogels in particular the presence of hydrophobic and hydrophilic groups, affects their swelling behavior. Hydrophilic groups are responsible for containing a significant amount of water, while the cross-linking of the polymer chains allows the hydrogel to retain water without dissolving. Hydrogels with higher hydrophilic group content swell more than those with hydrophobic groups [20].

For this reason, it is advisable to introduce a hydrophilic modifier into the hydrogel structure that participates in the polymerization process, forming an additional cross-linked structure containing more hydrophilic groups. A suitable modifier is the water-soluble polymer poly-N-vinylpyrrolidone (PVP), which is biocompatible and exhibits good adsorbing capacity. Introducing PVP into polymers increases their hydrophilicity, heat resistance, and mechanical properties [21]. It is also known that by incorporating vinylpyrrolidone copolymer into the polymer structure and varying its content results in composite materials with different drug release properties [22].

Thus, the objective of this study was to develop hydrophilic PAAGs modified with PVP and to investigate their structure, rheological properties, water absorption, and sorption capacity for dacarbazine, aiming at their potential use as drug carriers.

2. Experimental

2.1. Materials

Acrylamide (AA) C_3H_5NO (Merck, Germany) (MM = 71.07), N,N'-methylene-bis-acrylamide (MBAA) $C_7H_{10}N_2O_2$ (Merck, Germany) (MM = 154.17), ammonium persulfate $(NH_4)_2S_2O_8$ (Merck, Germany) (MM = 228.20), N,N,N′,N′-tetramethylethylenediamine (TEMED) $C_6H_{16}N_2$ (Merck, Germany) (MM = 116.21), poly-N-vinylpyrrolidone (PVP) $(C_6H_9NO)_n$ (M = 12600 ± 2700 g/mol), sodium phosphate buffer solution (pH 8.0–8.5) were used for the synthesis of PAAGs modified with PVP.

Dacarbazine (Medac) (DAC) $C_6H_{10}N_6O$ (Pharm., Germany) (MM = 182.18, melting temperature = 205 ℃) and citrate-phosphate buffer solution (pH 5.0) were used to study the sorption properties.

2.2. Method of Synthesis

2.2.1. Method of PAAG Synthesis

4.8 g (4.8%) of AA and 0.018 g (0.018%) MBAA were dissolved in 95 ml (95%) sodium phosphate buffer solution and placed in a three-necked reactor equipped with a mechanical stirrer and an argon supply system. The reaction was carried out in the presence of an oxidation-reduction initiating system consisting of 0.13 g (0.13%) ammonium persulfate and 0.05 ml (0.052%) TEMED. Polymerization took place in an inert environment under vacuum with constant stirring at room temperature for 10-20 minutes. The resulting gel was kept under vacuum for 24 hours.

2.2.2. Synthesis of PAAG Modified with 0.3 wt.% PVP (PAAG-0.3 PVP)

4.5 g (4.5%) of AA, 0.018 g (0.018%) MBAA, and 0.3 g (0.3%) PVP were dissolved in 95 ml (95%) sodium phosphate buffer solution and placed in a three-necked reactor equipped with a mechanical stirrer and an argon supply system. The reaction was carried out in the presence of an oxidation-reduction initiating system consisting of 0.13 g (0.13%) ammonium persulfate and 0.05 ml (0.052%) TEMED. Polymerization took place in an inert environment under vacuum with constant stirring at room temperature for 10-20 minutes. The resulting gel was kept under vacuum for 24 hours.

2.2.3. Synthesis of PAAG Modified with 0.5 wt.% PVP (PAAG-0.5 PVP)

The synthesis was carried out similarly to the synthesis of PAAG-0.3 PVP, using 4.3 g (4.3%) of AA, 0.018 g (0.018%) MBAA, 0.5 g (0.5%) PVP, 0.13 g (0.13%) ammonium persulfate and, 0.05 ml (0.052%) TEMED.

2.2.4. Synthesis of PAAG Modified with 1.0 wt.% PVP (PAAG-1.0 PVP)

The synthesis was carried out similarly to the synthesis of PAAG-0.3 PVP using 3.8 g (3.8%) of AA, 0.018 g (0.018%) MBAA, 1.0 g (1.0%) PVP, 0.13 g (0.13%) ammonium persulfate and 0.05 ml (0.052%) TEMED.

2.3. Study Methods

2.3.1. Fourier Transform Infrared (ATR FTIR) Spectroscopy

The structure was investigated on a Tensor-37 FTIR spectrometer in the range 650–4000 cm^{-1} by the MATR method with the aid of a diamond crystal trapezoidal prism (a number of reflections of N = 1, an incidence angle of φ = 39°).

2.3.2. Rheological Test

Rheological properties were studied using a rheometer (Rheometer TA AR 2000ex) to measure the drop in hydrogel viscosity depending on the shear rate.

2.3.3. Absorption Test

Water absorption was studied by determining the amount of water absorbed by the hydrogel after exposure to distilled water at a temperature of 37 ℃ for 24 hours. The samples were pre-treated with ethyl alcohol and dried to a constant weight. After 24 hours of exposure in distilled water, the samples were removed and gently dried using filter paper to eliminate surface water. Three measurements were conducted for each sample and calculate the mean and standard deviation.

The degree of water absorption (W, %) was calculated using the expression:

$$W = \frac{m_1 - m_0}{m_0} \cdot 100\% \quad (1)$$

where, m_1 –mass of the sample after soaking in water for 24 hours, g; m_0 –dry sample mass, g.

2.3.4. Sorption Test

The sorption properties of the samples were studied concerning the drug DAC. Samples with an average weight of 0.2658-0.2794 g were swollen in a 0.01% DAC solution at room temperature. The samples were placed in dark glass bottles with lids, and 20 ml of 0.01% DAC solution in a buffer with pH 5 was added. The volume of the solution absorbed by the samples was measured after specific time intervals (1, 3, 7, 14 days).

The stability of DAC solutions in aqueous and citrate-phosphate buffer solution was assessed using a spectrophotometric method. Absorption spectra of the tested solutions were obtained using "SPECORD M-40" spectrophotometer in cuvettes with a path length of 1 cm, relative to the comparison solutions.

3. Results and Discussion

3.1. Synthesis

The synthesis of PAAG (5%) was carried out by free radical polymerization of AA and MBAA in a sodium phosphate buffer solution (pH 8.0–8.5) in the presence of an oxidation-reduction initiating system (ammonium persulfate, TEMED). The synthesis of 5% PAAGs modified with PVP (Figure 1) was carried out by free radical polymerization of AA, MBAA and PVP in a sodium phosphate buffer solution (pH 8.0–8.5) in the presence of an oxidation-reduction initiating system (ammonium persulfate, TEMED).

A series of polyacrylamide gels modified with PVP in amounts of 0.3 wt.%, 0.5 wt.% and 1.0 wt.% relative to the weight of acrylamide were synthesized (PAAG; PAAG-0.3

PVP; PAAG-0.5 PVP; PAAG-1.0 PVP).

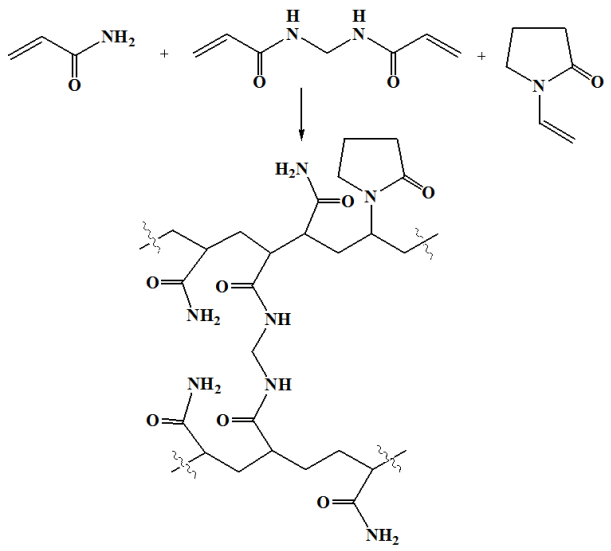

Figure 1. *Synthesis scheme of PAAGs modified with PVP.*

3.2. FTIR Spectroscopy

According to IR spectroscopic studies, PAAG (Figure 2, curve 1) in the frequency range of 3000-3600 cm^{-1} is characterized by the absorption band of $\nu_{NH\text{-}bond}$ with a maximum of 3186 cm^{-1} and the absorption band of $\nu_{NH\text{-}free}$ with a maximum of 3329 cm^{-1}.

The IR spectra of PAAG show that absorption bands corresponding to the valence vibrations of the =CH bond at 3033 cm^{-1} and 840-820 cm^{-1} disappear, indicating the opening of the vinyl group's double bond (–CH=CH_2) during polymerization process. This results in changes in the $\delta_{C\text{-}H}$ absorption bands at 1470-1375 cm^{-1} and 900-600 cm^{-1}, associated with the formation of the –CH–CH_2–CH– chain.

Additional characteristic absorption bands observed in the IR spectra include the C-H valence vibrations.

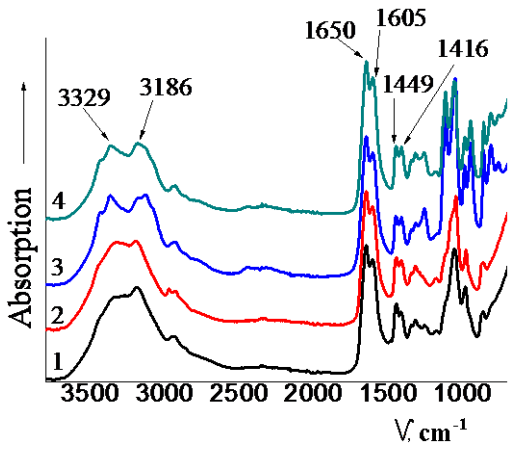

Figure 2. *IR spectra of synthesized hydrogels: PAAG (1), PAAG-0.3 PVP (2), PAAG-0.5 PVP (3), PAAG-1.0 PVP (4).*

(3000-2800 cm^{-1}), the $\nu_{C=O}$ absorption band of the $CONH_2$ group of acrylamide at 1650 cm^{-1} (amide I), the δ_{NH} absorption band at 1605 cm^{-1} (amide II), and the $\delta_{C\text{-}H}$ bands of bond at 1449 cm^{-1} and 1416 cm^{-1}. The region from 1350-700 cm^{-1} also contains bands corresponding to $\nu_{C\text{-}C}$ and $\nu_{C\text{-}N}$, $\delta_{C\text{-}H}$, and $\delta_{N\text{-}H}$ vibrations.

In the IR spectra of PAAG modified with PVP in the range of 3000-3600 cm^{-1} a decrease in the intensity of the $\nu_{NH\text{-}bond}$ absorption band at 3186 cm^{-1} and an increase in the intensity of the $\nu_{NH\text{-}free}$ absorption band at 3329 cm^{-1} are observed. Both absorption bands ($\nu_{NH\text{-}bond}$ and $\nu_{NH\text{-}free}$) are more intensively manifested on the profile of the main band of valence vibrations of NH-groups.

In the interval of valence vibrations of C-H bond 3000-2800 cm^{-1} on the IR spectra of PAAGs modified with PVP there is a redistribution of the intensity of the absorption bands, which indicates a different content of CH, CH_2, CH_3 groups in the skeletal chain. There is also a redistribution of the intensities of the $\delta_{C\text{-}H}$ absorption bands in the IR spectra interval of 1470-1375 cm^{-1}. The intensity of the absorption band at 1449 cm^{-1} decreases and the intensity of the absorption band at 1416 cm^{-1} increases.

These finding suggest, the introduction of PVP into the PAAG structure alters the hydrogen bonding network of NH groups in the polymer matrix, as well as the intensity distribution of valence and deformation vibrations of the C-H bond, pointing to different contents of CH, CH_2, and CH_3 groups in the skeletal chain.

3.3. Rheological Test

Since viscosity plays an important role in quality control of the material and its characteristics, studies of the rheological properties of polyacrylamide gels with varying PVP content were conducted. The rheological properties were evaluated according to the drop in the hydrogel viscosity depending on the shear rate (Figure 3).

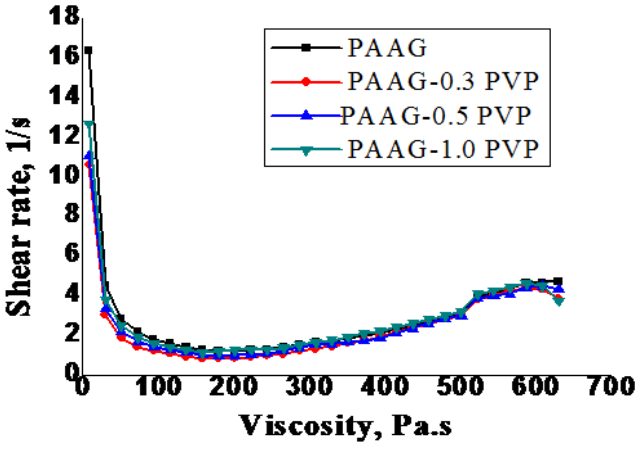

Figure 3. *Dependence of PAAG viscosity on the shear rate.*

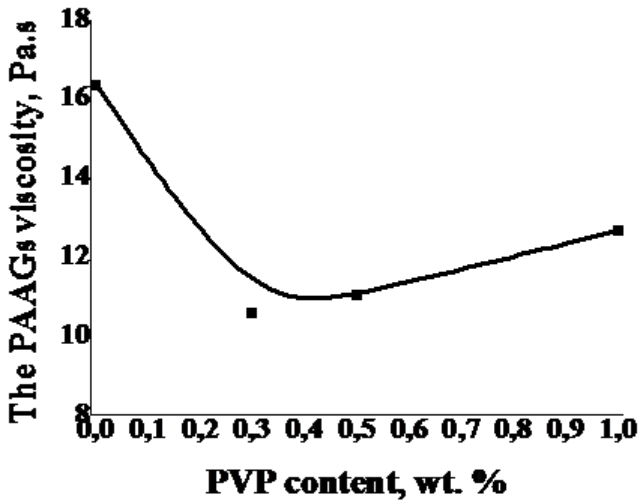

Figure 4. *Dependence of the hydrogels viscosity on PVP content.*

According to the results of rheological studies, hydrogels modified with PVP have a lower viscosity compared to PAAG by 22.4-35.3% depending on the PVP content. However, the stability of the structure remains sufficient to perform the functions of hydrophilic matrix for the immobilization of drug. At the same time, the dependence of viscosity on the amount of PVP in the hydrogel structure is monitored. An increase in the amount of PVP to 0.5 wt.% and 1.0 wt.% causes an increase in PAAGs viscosity by 4.3% and 19.8% respectively (Figure 4).

3.4. Absorption Test

Water absorption is one of the important characteristics of hydrogels, as it indicated their capacity to absorb and retain large amounts of water within their structures without disintegrating. Since the obtained PAAGs are modified with different amounts of PVP, that is, they have different amounts of hydrophilic and hydrophobic groups. This will affect the water absorption values.

The study results (Table 1) show that the water absorption ranges from 761.9 to 1059.8%. The addition of PVP into the PAAG structure and increasing its content causes an increase in water absorption values by1 9.6-39.1%

Table 1. *Water absorption of synthesized hydrogels.*

Samples	PAAG	PAAG-0.3 PVP	PAAG-0.5 PVP	PAAG-1.0 PVP
Water absorption,%	761.9	911.0	1044.8	1059.8

3.5. Sorption Test

The sorption properties of the synthesized hydrogels were tested using DAC. DAC is a well-known cytostatic antitumor chemotherapeutic drug of the alkylating type action, commonly used for treating metastatic melanoma [23].

First of all, the possibility of DAC sorption by polymer samples from aqueous solutions was studied. For this purpose, a study of the stability over time of a 0.002% DAC solution in distilled water was conducted. The solution was kept in a dark place at room temperature. The values of the optical density of the aqueous solution of DAC over time at the maximum of the band at a wavelength of λ = (329±2) nm are presented in Table 2. According to research results, the optical density of the DAC solution in distilled water decreases over time, that is, this solution is unstable.

Table 2. *Stability study of DAC solution over time.*

Time, day	λ_{max}, nm	Optical density of solution, $D_{avg.}$
0.002% DAC solution in distilled water		
0	328.1	0.6267
2	328.2	0.5755
6	328.5	0.5385
10	328.3	0.5249
14	328.5	0.5119
0.001% DAC solution in citrate-phosphate buffer (pH = 5)		
0	328.5	0.4842
2	328.4	0.4902
7	328.4	0.4856
14	328.3	0.4890

The possibility of DAC sorption by polymer samples from a citrate-phosphate buffer solution with pH 5 was investigated. For this purpose, the stability of a 0.001% DAC solution in the buffer kept at room temperature in a dark place was studied. The values of the optical density of the buffer solution over time are presented in Table 1. The stability of the optical density of this solution indicates the stability over time of a

0.001% DAC solution in a buffer (pH 5). This allows us to conduct a study of sorption from this solution.

To study the sorption properties of polymers, the PAAG samples were swollen in a 0.01% DAC solution buffered at pH 5. The volume of the absorbed solution was measured, enabling the calculation of the specific absorbed solution volume (in ml/g) and DAC-specific sorption (mg/g), that is the amount of sorbted DAC during the swelling of 1 gram of sample. The results of measurements and calculations of DAC sorption by the studied samples are presented in Table 3.

Table 3. Sorption properties of the hydrogels relative to dacarbazine.

Time, day	Volume of solution absorbed by the sample during swelling, ml			
	PAAG	PAAG-0.3 PVP	PAAG-0.5 PVP	PAAG-1.0 PVP
1	3.0	4.5	4.0	4.0
3	4.0	5.0	5.5	5.5
7	4.5	5.0	6.5	6.5
14	5.5	5.5	7.0	7.0
Specific absorbed volume for 14 days, ml/g	20.69	20.49	25.05	26.30
Specific sorption of DAC, mg/g	2.07	2.05	2.51	2.63

The results show (Table 3) that increasing the PVP content to 0.5 wt.% and 1.0 wt.% boosts the sorption capabilities of PAAGs by 21.1% and 27.1% respectively. Therefore, it is possible to obtain hydrogels for medicine with different sorption capacity by varying of the PVP content in the structure of materials.

The hydrogel samples modified with 1.0 wt% PVP are characterized by the highest sorption capacity relative to DAC.

Thus, during the experiment it was found that the DAC solution in the citrate-phosphate buffer (pH = 5) is stable, which made it possible to use it to study the sorption properties of the studied polymers. A study of DAC sorption by polymer samples was carried out for 14 days (until full saturation). It has been established that increasing the PVP content in the hydrogel to 0.5 wt.% and 1.0 wt.% leads to an increase in the sorption capacity of PAAGs.

Based on the obtained results, hydrophilic PAAGs modified with PVP show sorption capacity for dacarbazine and high water absorption values, making them suitable for potential use as drug carriers in medicine. Thus, these hydrogels should undergo further biomedical research.

4. Conclusions

PAAGs modified with PVP (0.3 wt.%, 0.5 wt.% and 1.0 wt.%) were synthesized using free radical polymerization of acrylamide, N,N'-methylene-bis-acrylamide and PVP in a sodium phosphate buffer solution. The modified hydrogels' structure, water absorption, rheological, and sorption properties were studied. It has been established that addition of PVP into the PAAG structure leads to changes in the structure of hydrogen bonds of NH-groups of the polymer matrix and changes in the skeletal chain (different content of CH, CH_2, and CH_3 groups), a decrease in viscosity and an increase in water absorption and sorption properties. The obtained results indicted that the obtained hydrogels have potential for further development as drug carriers.

Abbreviations

PAAG	Polyacrylamide Gel
PVP	Poly-N-vinylpyrrolidone
AA	Acrylamide
MBAA	N,N'-methylene-bis-acrylamide
TEMED	N,N,N′,N′-tetramethylethylenediamine
DAC	Dacarbazine

Author Contributions

Tetiana Vislohuzova: Investigation, Writing – original draft

Rita Rozhnova: Conceptualization, Resources, Writing – review & editing

Tetiana Kiselova: Investigation

Galyna Kozlova: Investigation

Liudmyla Nechaeva: Investigation

Natalia Galatenko: Conceptualization, Resources

Conflicts of Interest

The authors declare no conflicts of interest.

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