

EFFECT OF POLYMER COMPOSITION AND TEMPERATURE ON THE RHEOLOGICAL PROPERTIES OF CHITOSAN-CARBOXYMETHYL CELLULOSE HYDROGELS

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Abstract. *Introduction.* Over the past decade, superabsorbent hydrogels based on natural polymers (cellulose, chitosan) have been widely applied in biomedicine. Despite their high stability and biocompatibility, these hydrogels exhibit several drawbacks associated with insufficient pseudoplasticity and thixotropy. Consequently, the development of hydrogel systems with tailored rheological properties has acquired critical medical relevance. *The objective of this study* was to develop and comprehensively investigate the rheological properties of hydrogels based on chitosan (CS) and carboxymethylcellulose (CMC) with different component ratios, as well as to evaluate the effects of shear rate and temperature on their rheological characteristics in order to identify promising formulations for topical application. *Results and Discussion.* Chitosan/carboxymethylcellulose (CS/CMC) hydrogels with different polymer ratios were investigated by rheometry at temperatures of 20, 25, and 35°C over a shear-rate range of 3-120 s⁻¹. The highest apparent viscosity under all investigated conditions was observed for the CS2.0/CMC0.5 formulation, whereas increasing temperature resulted in a consistent decrease in viscosity. To quantitatively evaluate the rheological behavior, the experimental data were fitted using the Power-law, Herschel-Bulkley, and Casson models. *Conclusion.* CS/CMC hydrogels exhibited pronounced shear-thinning behavior at 20-35°C and shear rates of 3-120 s⁻¹, facilitating their application to the skin. The CS2.0/CMC0.5 formulation showed the highest viscosity and stable rheological behavior ($K = 16.90 \text{ Pa}\cdot\text{s}^n$, $n = 0.389$, $R^2 = 0.987$ at 25°C), making it a promising matrix for topical hydrogel systems.

Keywords: chitosan, carboxymethylcellulose, hydrogel, rheological characteristics, pseudoplasticity, topical application

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1. Introduction

Hydrogels' capacity to hold water and their structural resemblance to the natural extracellular matrix have made them popular as prospective scaffolds for tissue engineering in recent years. Owing to these properties, hydrogels have found broad applications in biomedicine, including wound and burn dressings, due to their excellent biocompatibility, low toxicity, elasticity, and high swelling capacity in aqueous media [1-3].

For topical applications, not only the biological properties of hydrogels but also their rheological characteristics are of particular importance, as these properties determine the application, spreading, and retention of the material on the skin surface. Specifically, system stability and uniform spreading during application may be facilitated by a combination of sufficiently high viscosity at low shear rates and a reduction in viscosity with increasing shear rate. Therefore, investigation of the dependence of viscosity on shear rate is an important step in the development of hydrogel systems for topical application [4,5].

Hydrogels based on chitosan (CS) and carboxymethylcellulose (CMC) are of particular interest in this field due to their high hydrophilicity, biocompatibility, and water-retention capacity. Chitosan is a natural cationic polysaccharide [6,7], whereas CMC is a water-soluble cellulose derivative with pronounced thickening and water-retention properties [8-11]. Varying the ratio of these polymers makes it possible to modify the structural and rheological characteristics of the hydrogel system and thereby select formulations with the desired flow behavior [12,13].

The rheological properties of polymeric hydrogels depend not only on their composition but also on temperature, as changes in temperature may affect molecular mobility and intermolecular interactions within the polymer system [14,15]. Quantitative characterization of rheological behavior can be performed using the Power-law, Herschel-Bulkley, and Casson models, which allow flow parameters to be determined and different formulations to be compared [16-18].

2. Materials and Experimental Methods

2.1. Materials

Chitosan (CS, Cat. No. C804729) was obtained from Shanghai Macklin Biochemical Technology Co., Ltd. (China). The manufacturer's specs state that chitosan has a viscosity of 200-400 mPa·s in 1% acetic acid and a degree of deacetylation (DDA) of 88-92%. The polymer was recovered as an off-white to yellow powder.

Carboxymethyl cellulose (CMC, Cat. No. C889437) was obtained from Shanghai Macklin Biochemical Technology Co., Ltd. (China) and employed as the second polymer component for the manufacture of CS/CMC hydrogel systems.

Acetic acid was utilized as a solvent for the dissolving of chitosan in aqueous medium. Polymer solutions and hydrogel compositions were prepared using purified water. All materials and reagents were used as received without any further purification.

2.2. Chitosan-Carboxymethyl Cellulose Hydrogels Preparation

Chitosan-carboxymethyl cellulose (CS/CMC) hydrogels were synthesized with polymer concentration from 0.5 to 2.0%. Table 1 shows the results of six formulations with different polymer ratios.

Table 1 - Composition of the investigated CS/CMC hydrogel formulations

№	Sample	Chitosan (CS), %	Carboxymethyl cellulose (CMC), %
1	CS0.5/CMC1.0	0.5	1.0
2	CS0.5/CMC1.5	0.5	1.5
3	CS0.5/CMC2.0	0.5	2.0
4	CS1.0/CMC0.5	1.0	0.5
5	CS1.5/CMC0.5	1.5	0.5
6	CS2.0/CMC0.5	2.0	0.5

For the preparation of the hydrogel formulations, CS was dissolved in an aqueous acetic acid solution at the minimum concentration required for complete polymer dissolution. CMC was separately dissolved in purified water. The CS and CMC solutions so obtained were combined in prescribed ratios and the volume was made up to the final volume with filtered water. The solutions were well homogenized to generate homogenous hydrogel compositions.

2.3. Rheological Measurements

The rheological properties of the prepared CS/CMC hydrogel formulations were investigated using a Rheotest II rotational viscometer equipped with a cylindrical measuring system. Measurements were performed at 20, 25, and 35°C over a shear rate range of 3-120 s⁻¹ to evaluate the effects of polymer composition and temperature on the rheological behavior of the hydrogels. The apparent viscosity was recorded at each shear rate, and viscosity-shear rate curves were subsequently plotted. The measuring system was selected according to the viscosity range of the samples, taking into account the corresponding instrument constants. The mean ± standard deviation (SD) was used to express the findings of all measurements, which were done in triplicate (n=3).

2.4. Data Analysis and Rheological Modeling

The experimental rheological data were fitted using the power-law, Herschel-Bulkley and Casson models to characterize the rheological behavior of the CS/CMC hydrogel compositions. Consistency index (K) and flow behavior index (n) were estimated using power-law model. The flow behavior index (n) reflects how the viscosity of the hydrogel depends on the shear rate: n<1 means shear-thinning, n=1 means Newtonian behavior, and n>1 means shear-thickening. Yield stress and other rheological parameters were obtained by using the Herschel-Bulkley and Casson models.

The model parameters were determined by fitting the matching models to the experimental data for each formulation and temperature. The coefficient of determination, R², was used to evaluate the goodness of fit and to compare the adequacy of the models studied to represent the rheological behavior of the

hydrogel formulations. The results are shown as mean $\pm$ SD based on three replicate measurements (n=3).

3. Results and discussion

3.1. Effect of Polymer Composition on Apparent Viscosity

Rheological behavior of CS/CMC hydrogel compositions relied on polymer mix, shear rate and temperature. All formulations showed a progressive decrease in the apparent viscosity with the increase of the shear rate and the level of viscosity depended on the polymer content.

At 25°C and 3s⁻¹, increasing the CMC concentration from 1.0 to 2.0% at a constant CS concentration of 0.5% increased the apparent viscosity from 1.43 $\pm$ 0.04 to 5.21 $\pm$ 0.15 Pa·s. Similarly, at a constant CMC concentration of 0.5%, increasing the CS concentration from 1.0 to 2.0% increased the viscosity from 2.79 $\pm$ 0.12 to 8.09 $\pm$ 0.20 Pa·s. The highest apparent viscosity was therefore observed for CS2.0/CMC0.5. The viscosity–shear rate profiles of the six formulations at 25°C are presented in Figure 1.

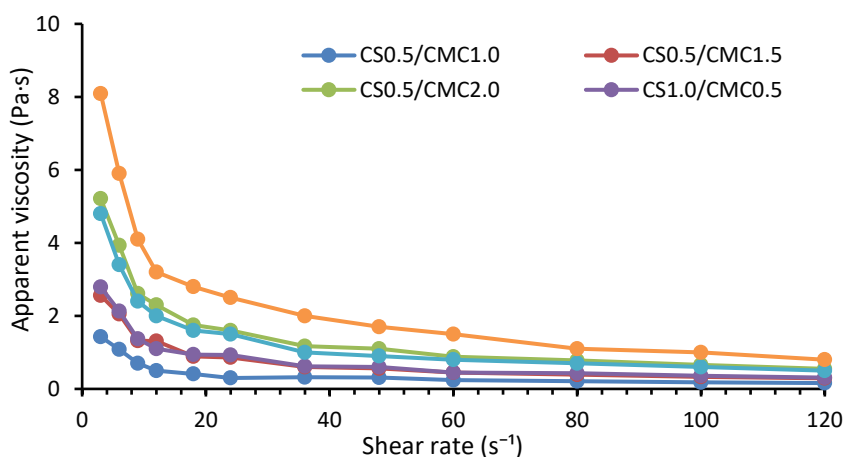


Figure 1 – Apparent viscosity as a function of shear rate for CS/CMC hydrogel formulations with different polymer compositions at 25°C. Data are presented as the mean $\pm$ SD.

The increase of the polymer concentration leads to a rise of the apparent viscosity due to the lower mobility of the polymer chains and the higher intermolecular interactions between CS and CMC that favor the creation of a more interconnected polymeric structure and increase the resistance to flow.

3.2. Shear rate effect on flow behavior

Figure 1 demonstrates that all CS/CMC formulations displayed a significant reduction in apparent viscosity with increasing shear rate from 3 to 120 s⁻¹, confirming the characteristic non-Newtonian shear-thinning behavior. At 25°C, the apparent viscosity of CS2.0/CMC0.5 declined from 8.09 $\pm$ 0.20 Pa·s at 3 s⁻¹ to 0.89 $\pm$ 0.02 Pa·s at 120 s⁻¹, which was approximately 89% reduction, while

CS0.5/CMC1.0 decreased from 1.43 ± 0.04 to 0.15 ± 0.01 Pa·s. The decrease could be linked with orientation and restructuring of polymer chains under shear, resulting in reduced flow resistance [17]. The substantial shear thinning tendency seen in all the formulations should be beneficial for topical administration since the increased viscosity at low shear could increase the retention, whilst the decrease of the viscosity under shear facilitates the spreading over the skin surface [19].

3.3 Influence of Temperature on Rheological Properties

The rheological behavior of the CS/CMC formulations was significantly affected by temperature. The overall apparent viscosity decreased with increasing temperature from 20 to 35 °C in the studied shear-rate range. For CS2.0/CMC0.5 at 3 s^{-1} , the apparent viscosity decreased from 10.14 ± 0.60 Pa·s at 20°C to 8.09 ± 0.20 Pa·s at 25°C and 7.42 ± 0.19 Pa·s at 35°C; the corresponding viscosity–shear rate curves are presented in Figure 2.

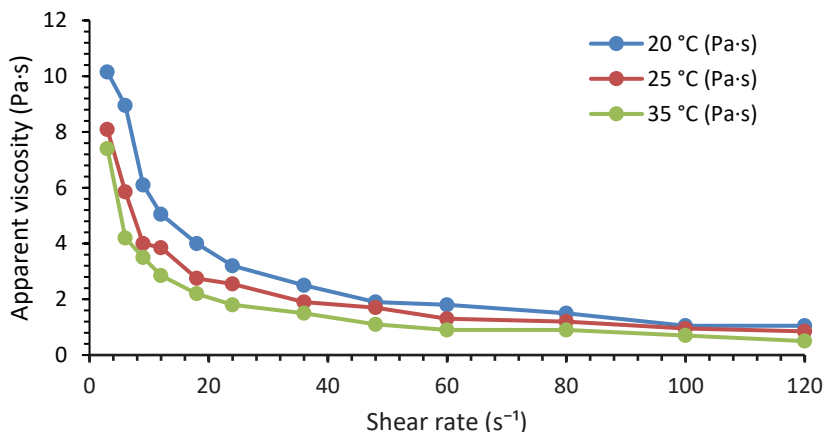


Figure 2 – Effect of temperature on the apparent viscosity of the CS2.0/CMC0.5 hydrogel formulation as a function of shear rate at 20, 25, and 35°C. Data are presented as the mean $\pm$ SD.

The decrease in apparent viscosity with increasing temperature may be attributed to increased molecular mobility and reduced resistance to polymer-chain rearrangement, while the characteristic shear-thinning behavior was maintained at all investigated temperatures. Therefore, throughout the temperature range of 20–35 °C, the overall flow behavior of the hydrogels, which is relevant to the consistency and spreading in topical application, was not altered by the temperature except for to the magnitude of viscosity.

3.4 Power-Law Analysis of the CS/CMC Formulations

The experimental rheological data were fitted to the power-law model to characterize the shear-dependent behavior of the CS/CMC formulations. The flow behavior index (n) was below unity for all formulations and temperatures, confirming their pseudoplastic (shear-thinning) character. The calculated power-law parameters are summarized in Table 2.

Table 2 – Power-law parameters of CS/CMC hydrogel formulations at various temperatures

Formulation	Temperature, °C	K, Pa·s ⁿ	n	R ²
CS0.5/CMC1.0	20	3.384	0.389	0.981
	25	3.126	0.378	0.987
	35	2.436	0.392	0.985
CS0.5/CMC1.5	20	6.400	0.384	0.982
	25	5.711	0.382	0.988
	35	4.603	0.388	0.984
CS0.5/CMC2.0	20	12.440	0.387	0.981
	25	11.253	0.381	0.987
	35	9.168	0.386	0.984
CS1.0/CMC0.5	20	6.580	0.395	0.980
	25	5.890	0.391	0.983
	35	4.881	0.391	0.981
CS1.5/CMC0.5	20	11.464	0.386	0.981
	25	9.958	0.390	0.984
	35	8.388	0.381	0.986
CS2.0/CMC0.5	20	18.533	0.396	0.988
	25	16.899	0.389	0.987
	35	14.102	0.385	0.985

The flow behavior index (n) ranged from 0.378 to 0.396 and remained well below unity for all formulations, confirming consistent shear-thinning behavior. In contrast, the consistency index (K) strongly depended on polymer composition and temperature. CS2.0/CMC0.5 exhibited the greatest K values, which declined from 18.53 Pa·sⁿ at 20 °C to 16.90 Pa·sⁿ at 25 °C and 14.10 Pa·sⁿ at 35 °C. The decrease in K with increasing temperature was in agreement with the observed decrease of apparent viscosity. Figure 3 shows a representative power-law fit for CS2.0/CMC0.5 at 25 °C.

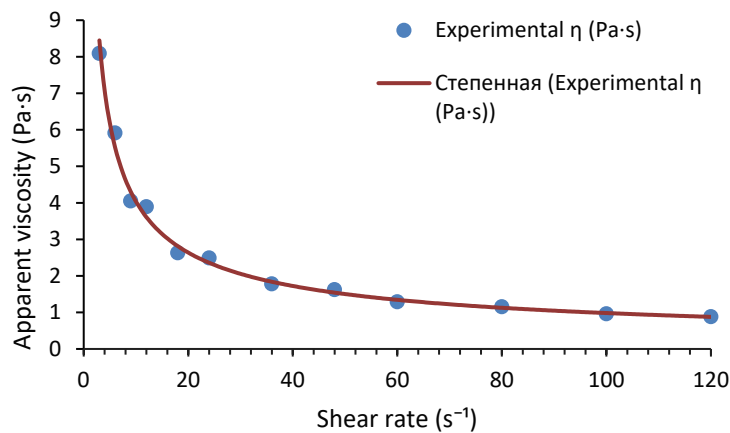


Figure 3 – Power-law fitting of the experimental rheological data for the CS2.0/CMC0.5 hydrogel formulation at 25°C. Experimental values are presented as the mean ± SD ($n = 3$).

For this formulation, the power-law model yielded $K = 16.90 \text{ Pa}\cdot\text{s}^n$, $n =$

0.389, and $R^2 = 0.987$. The low n value quantitatively confirmed the pronounced shear-thinning behavior observed experimentally.

3.5. Comparison of Rheological Models

The experimental data were additionally fitted using the Herschel-Bulkley and Casson models, and a representative comparison of these models for CS2.0/CMC0.5 at 25°C is presented in Figure 4.

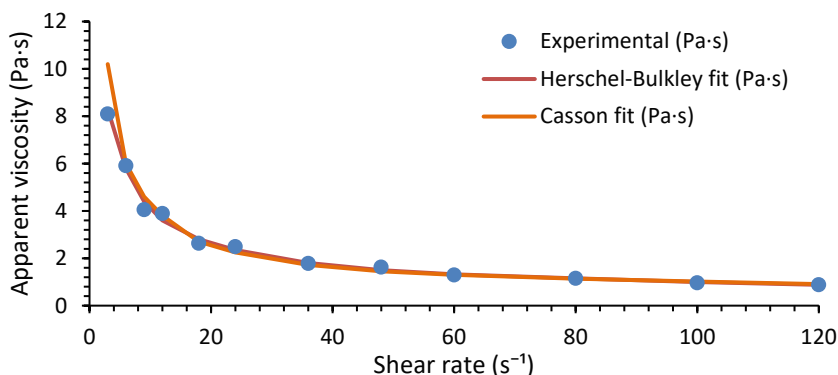


Figure 4 – Comparison of Herschel-Bulkley and Casson model fits for the CS2.0/CMC0.5 hydrogel formulation at 25°C. Experimental points represent mean values from three independent measurements ($n = 3$).

For the representative CS2.0/CMC0.5 formulation at 25 °C, the power-law model provided a satisfactory fit to the experimental data ($R^2 = 0.987$), comparable to that obtained with the Herschel–Bulkley model ($R^2 = 0.987$). The Casson model showed a lower goodness of fit ($R^2 = 0.961$), indicating a less adequate description of the experimental flow behavior. These results support the predominantly pseudoplastic nature of the formulation. Therefore, the power-law model was selected as the principal model for the quantitative comparison of the investigated formulations.

Overall, increasing the concentration of either polymer increased the apparent viscosity and consistency index, whereas increasing temperature reduced these parameters. All formulations retained pronounced shear-thinning behavior. CS2.0/CMC0.5 exhibited the highest apparent viscosity and consistency index together with substantial shear-dependent viscosity reduction, supporting its potential as a topical hydrogel formulation for further investigation [20,21].

4. Conclusion

The rheological properties of CS/CMC hydrogels were strongly influenced by polymer composition and temperature. Increasing the concentration of either polymer increased the apparent viscosity, whereas increasing temperature reduced it. All investigated formulations exhibited pronounced non-Newtonian shear-thinning behavior, confirming the pseudoplastic nature of the developed hydrogel

systems. Among the investigated formulations, CS2.0/CMC0.5 demonstrated the highest apparent viscosity and consistency, making it a promising formulation for topical applications.

The experimental data were adequately described by the applied rheological models, supporting the quantitative characterization of the flow behavior of the CS/CMC systems. Overall, the findings demonstrate that adjustment of the CS/CMC ratio and temperature provides an effective approach for controlling hydrogel rheology. These results provide a basis for the further development and optimization of CS/CMC hydrogel systems for topical and biomedical applications.

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ПОЛИМЕР ҚҰРАМЫ МЕН ТЕМПЕРАТУРАНЫҢ ХИТОЗАН- КАРБОКСИМЕТИЛЦЕЛЛЮЛОЗА ГИДРОГЕЛЬДЕРІНІҢ РЕОЛОГИЯЛЫҚ ҚАСИЕТТЕРІНЕ ӘСЕРІ

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Түйіндеме. *Kіріспе.* Соңғы онжылдықта биомедицинада табиғи полимерлер (целлюлоза, хитозан) негізіндегі аса жоғары сіңіргіш гидрогельдер кеңінен қолданылуда. Жоғары тұрақтылығы мен биосәйкестігіне қарамастан, бұл гелидер псевдопластикалық және тиксотропиялық қасиеттерінің жеткіліксіз болуына байланысты бірқатар кемшіліктерге ие. Осыған байланысты берілген реологиялық қасиеттері бар гидрогельдік жүйелерді әзірлеу медициналық тұрғыдан өзекті міндет болып табылады. *Зерттеудің мақсаты* – хитозан (CS) мен карбоксиметилцеллюлоза (CMC) негізіндегі гидрогельдерді әртүрлі компоненттік қатынаста алу және олардың реологиялық қасиеттерін кешенді зерттеу, сондай-ақ перспективалы сыртқа қолдануға арналған құрамдарды анықтау мақсатында ығысу жылдамдығы мен температураның олардың құрылымдық-тұтқырлық сипаттамаларына әсерін бағалау. *Нәтижелер және талқылау.* Әртүрлі полимерлік қатынастағы хитозан және карбоксиметилцеллюлоза (CS/CMC) негізіндегі гидрогельдер 20, 25 және 35°C температураларда, 3-120 с⁻¹ ығысу жылдамдығы диапазонында реометрия әдісімен зерттелді. Зерттелген барлық жағдайларда ең жоғары көрінерлік тұтқырлық мәндері CS2.0/CMC0.5 құрамында байқалды, ал температураның жоғарылауы тұтқырлықтың заңды түрде төмендеуіне әкелді. реологиялық қасиеттерін сандық бағалау үшін эксперименттік деректер Power-law, Herschel-Bulkley және Casson модельдерімен аппроксимацияланды. *Қорытынды.* 20-35°C температура аралығында және 3-120 с⁻¹ ығысу жағдайында CS/CMC гидрогельдері теріге жағуды жеңілдететін айқын сұйылу қасиетін көрсетті. CS2.0/CMC0.5 құрамы ең жоғары тұтқырлықты және тұрақты реологиялық қасиеттерін көрсетті (25°C температурада $K=16,90 \text{ Па} \cdot \text{с}^n$, $n=0,389$, $R^2=0,987$). Алынған нәтижелер CS2.0/CMC0.5 құрамын тері сыртына қолдануға арналған топикалық гидрогельдік жүйелерді әзірлеу үшін перспективалы негіз ретінде қарастыруға мүмкіндік береді.

Түйінді сөздер: хитозан, карбоксиметилцеллюлоза, гидрогель, реологиялық сипаттамалар, псевдопластикалық, топикалық қолданыс

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ВЛИЯНИЕ СОСТАВА ПОЛИМЕРОВ И ТЕМПЕРАТУРЫ НА РЕОЛОГИЧЕСКИЕ СВОЙСТВА ХИТОЗАН-КАРБОКСИМЕТИЛЦЕЛЛЮЛОЗНЫХ ГИДРОГЕЛЕЙ

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Резюме. *Введение.* В последнее десятилетие в биомедицине широко применяются сверхпоглощающие гидрогели на основе природных полимеров (целлюлоза, хитозан). Несмотря на высокую стабильность и биосовместимость, данные гели обладают рядом недостатков, связанных с недостаточной псевдопластичностью и тиксотропностью. В связи с этим критическую медицинскую актуальность приобретает разработка гидрогелевых систем с заданными реологическими свойствами. *Целью исследования* является получение и комплексное реологическое исследование гидрогелей на основе хитозана (CS) и карбоксиметилцеллюлозы (CMC) с различным соотношением компонентов, а также оценка влияния скорости сдвига и температуры на их структурно-вязкостные характеристики для определения перспективных составов для наружного применения. *Результаты и обсуждение.* Гидрогели на основе хитозана и карбоксиметилцеллюлозы (CS/CMC) с различным соотношением полимеров были исследованы методом реометрии при температурах 20, 25 и 35°C в диапазоне скоростей сдвига 3-120 с⁻¹. Наибольшие значения кажущейся вязкости во всех исследованных условиях наблюдались для состава CS2.0/CMC0.5, тогда как повышение температуры приводило к закономерному снижению вязкости. Для количественной оценки реологического поведения экспериментальные данные были аппроксимированы моделями Power-law, Herschel-Bulkley и Casson. *Заключение.* Гидрогели CS/CMC при 20-35 °C и сдвиге 3-120 с⁻¹ проявили выраженное разжижение, облегчающее их нанесение на кожу. Состав CS2.0/CMC0.5 показал максимальную вязкость и устойчивое реологическое поведение ($K=16,90$ Па·сⁿ, $n=0,389$, $R^2=0,987$ при 25°C), что делает его перспективной основой для топических гидрогелевых систем.

Ключевые слова: хитозан, карбоксиметилцеллюлоза, гидрогель, реологические характеристики, псевдопластичность, топическое применение

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