
<https://doi.org/10.15407/ujpe69.3.207>

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FEATURES OF GELATION AND AGGREGATION IN AQUEOUS SOLUTIONS OF HYDROXYPROPYL CELLULOSE WITH NaCl, NaI, AND AgNO₃ SALTS

Using the turbidimetric method and the method of static light scattering, the features of gelation and aggregation in 2 wt% and 0.2 wt% aqueous solutions of hydroxypropyl cellulose (HPC) with NaCl, NaI, and AgNO₃ salts have been studied. It is found that the introduction of these salts into the 2% solution reduces the coefficient of surface tension at the interface between the sol and gel phases, decreases the lower critical solution temperature (LCST), and slows down the sol-gel transition rate. From the data on static light scattering, it follows that in the dilute (1 : 10) aqueous solutions of HPC at temperatures above the LCST and in the presence of salts, there arise supramolecular associates (clusters) that are several times larger than the wavelength of visible light. The precipitation of polymer is observed. It is shown that the intensity of reflected light can be described as a function of the cluster size in the framework of the Mie scattering theory. It is found that the density of polymer aggregates in the AgNO₃ solution was the highest among the studied specimens, which can be explained by the reduction of silver nitrate to metal. The sedimentation of HPC clusters containing silver nanoparticles can be used for creating composite polymer films with the bactericidal and fungicidal activities.

Keywords: hydroxypropyl cellulose, salts, gel formation, aggregation.

1. Introduction

Cellulose is the most widespread renewable organic material produced in the biosphere. The cellulose structure is represented by an elongated linear chain-like macromolecule composed of hundreds or tens of thousands of D-glucose residues. The use of cellulose together with its derivatives has wide applications in various fields, including the food industry,

building, hydrocarbon extraction, aerospace materials, medicine, and pharmaceuticals [1–3]. Although cellulose is a hydrophilic material [4, 5], it is insoluble in water and most common solvents. Therefore, there was a need to modify the structure of cellulose in order to improve its solubility, which led to the synthesis of various cellulose derivatives.

A characteristic feature of many water-soluble cellulose ethers is the thermoreversible volume transition. Its essence consists in the formation of a polymer gel network when the temperature increases, but the system returns to the state of isotropic solution when cooled down. The threshold temperature of this transition is called the lower critical solution temperature (LCST). The start of the gel formation is initiated by the aggregation of macromolecules into polymer associates, which leads to the formation of

Citation: Kovalchuk V.I., Zabashta Yu.F., Bulavin L.A. Features of gelation and aggregation in aqueous solutions of hydroxypropyl cellulose with NaCl, NaI, and AgNO₃ salts. *Ukr. J. Phys.* **69**, No. 3, 207 (2024). <https://doi.org/10.15407/ujpe69.3.207>.

Цитування: Ковальчук В.І., Забашта Ю.Ф., Булавін Л.А. Особливості гелеутворення і агрегації у водних розчинах гідроксипропілцелюлози з солями NaCl, NaI і AgNO₃. *Укр. фіз. журн.* **69**, №3, 207 (2024).

ISSN 2071-0194. *Ukr. J. Phys.* 2024. Vol. 69, No. 3

a single homogeneous cluster that fills the entire volume of the system. At the same time, the structure of the emerging polymer network is determined by the influence of various factors, for example, electrolyte impurities.

The study of the specific features of macromolecule aggregation and gel formation in systems containing cellulose derivatives is a challenging task owing to the wide application of those materials in biomedical technologies [6–8]. Hydroxypropyl cellulose (HPC) as an object of research was chosen due to the attractiveness of its physical and chemical properties. In addition to the well-known unique properties of a “green polymer”, such as availability, cheapness, and biocompatibility, HPC has a low LCST value of (40–42) °C [9–12]. Such a temperature is close to the physiological interval of temperatures, which opens prospects for the application of HPC in the systems of controlled drug delivery and wound treatment.

In this work, the thermotropic gelation and aggregation processes in aqueous solutions of HPC with NaCl, NaI, and AgNO₃ admixtures were studied. The latter two salts demonstrate a pronounced bactericidal activity. Therefore, the physical and biological properties of polymer systems created making use of those salts have been the subject of intensive research for a long time [13–17].

2. Materials and Methods

2.1. Materials

The cellulose derivative, hydroxypropyl cellulose (HPC), was manufactured by the Alfa Aesar company [18]. The manufacturer’s specification stated that the average molecular weight of HPC was 10⁵, the degree of substitution was 75.7%, and the viscosity of the 5 wt% aqueous solution of the polymer at 25 °C was 112 cPs.

Analytical-grade NaCl, NaI, and AgNO₃ salts were produced by the Sigma-Aldrich company.

2.2. Specimen preparation

The initial aqueous HPC solution with a concentration of 2 wt% was prepared by dissolving the necessary amount of polymer in deionized water under continuous stirring for 4 h at a temperature of 60 °C to ensure the complete dissolution of the polymer. As a result, a homogeneous and transparent solution was obtained.

This initial solution was divided to prepare four specimens. The salts were introduced into three of them, and the specimens were mixed until the complete salt dissolution. The molar concentrations of the salt in the specimens were identical and equal to that of the physiological solution (154 mmol/l).

By diluting 2% solutions with water to a ratio of 1:10, specimens with a polymer concentration of 0.2 wt% and a salt concentration of 15.4 mmol/l were fabricated.

2.3. Static light scattering

The temperature dependences of the intensities of transmitted and reflected light were measured on the installation described in works [19, 20].

3. Results and Their Discussion

The studies were performed using the device described in detail in works [19, 20]. When falling on the surface of the examined solution, the primary beam of light with a wavelength of 525 nm formed two beams, transmitted and reflected. The corresponding intensities J_T and J_R of these beams were measured at a set of temperatures within an interval from 25 to 70 °C. The specimen heating rate was 1.1 °C/min. The obtained experimental data were calibrated in each specimen to a transparency level of 100%. As such the corresponding value of J_T at a temperature of 25 °C was selected.

3.1. Specimens with the 2-wt% HPC concentration and the 154-mmol/l salt concentration. Transparency of solutions

Figure 1 exhibits the measurement results obtained for the temperature dependences of the relative intensities of the transmitted light beam (the solution transparency) for specimens with NaCl, NaI, and AgNO₃ salts. To interpret the results of the turbidimetric experiment, let us use the formalism developed by us to describe the kinetics of sol-gel transitions [21, 22].

Let phases A and B be in equilibrium at the temperature $T = T_0$, and the transition $A \rightarrow B$ occur at $T > T_0$. Provided that the system temperature changes in time according to a linear law

$$T(t) = T_0 + vt, \quad (1)$$

where v is the heating rate, the following formula was obtained in work [21]:

$$\ln \theta_A(t) = -\xi \int_0^t \exp \left[-\frac{M}{(v\tau)^2} \right] (t - \tau)^\alpha d\tau. \quad (2)$$

Here, θ_A is the relative volume of phase A; and ξ , α , and M are parameters to be determined from the experiment

In work [22] it was shown that the ratio between the surface tension coefficients at the interface between the sol and gel phases in the experimental specimens can be calculated using the formula

$$\frac{\sigma'}{\sigma_1} = \left(\frac{M'}{M_1} \right)^{1/3}, \quad (3)$$

where the primed quantities are related to the solutions with ions, and the quantities with the subscript 1 to the ion-free solution. By minimizing the mean square deviation function

$$\chi^2(N) = N^{-1} \sum_{j=1}^N [\theta_A(T_j) - \theta_{\text{exp}}(T_j)]^2, \quad (4)$$

where N is the number of temperature points, and θ_{exp} is the experimental value of the relative volume of phase A, we calculated the values of the parameters ξ , α , and M .

In Table, the LCST values corresponding to the inflection points in the temperature dependences of J_T depicted in Fig. 1 and the relative changes in the interfacial surface tension coefficient (3) are quoted for each solution. From the analysis of the presented data, it follows that the coefficients of interphase surface tension are lower if the specimens contain ions in comparison with the case of ion-free solution.

Qualitatively, this result can be explained as follows. Cellulose derivatives are moderately rigid chain polymers [23], whose macromolecules in various solvents are characterized by certain conformations. The latter depend on the solvent type, the polymer concentration, and so forth. For instance, HPC macromolecules have an elongated shape in ethanol solutions [24] and in diluted aqueous solutions [25]. At the same time, in work [26], it was shown that its polymer chain has a statistical coil conformation in aqueous solutions with HPC concentrations of (1÷5) wt%.

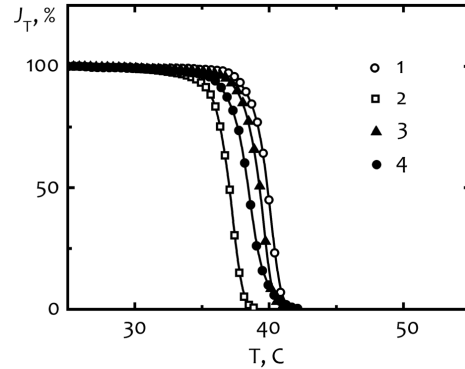


Fig. 1. Temperature dependences of the relative intensities of transmitted light, J_T , for specimens with an HPC concentration of 2 wt% and ions of various salts: ion-free (1), NaCl (2), NaI (3), and AgNO₃ (4)

Lower critical solution temperature (LCST) and the change of the interfacial surface tension coefficient σ when the salt is introduced into the polymer solution

n	Salt	LCST, °C	σ_n/σ_1
1	–	40.1	1
2	NaCl	37.2	0.899
3	NaI	39.5	0.933
4	AgNO ₃	38.7	0.815

The equilibrium state of a polymer coil (at $T = T_0$ [21]) is characterized by its root-mean-square radius, which is determined by a balance between the electrostatic repulsion forces between the monomers, which leads to the coil swelling, and the elastic forces preventing this swelling. The transition from the equilibrium state to the gel one is accompanied by a temperature growth and a corresponding swelling of the polymer coil [27]. The presence of ions in the solution gives rise to an additional swelling of the coil due to the screening of the interaction forces between the polymer chain links. This effect manifests itself in the LCST reduction and the decrease of the work that must be done to form a unit area of the sol-gel interface. In our opinion, this can be a possible mechanism that qualitatively describes the decrease of the interfacial surface tension coefficient in the presence of the NaCl, NaI, and AgNO₃ salt ions in the solution.

3.2. Dilute (1 : 10) solutions. Light reflection

Figure 2 illustrates the measurement results for the temperature dependences of the relative intensities

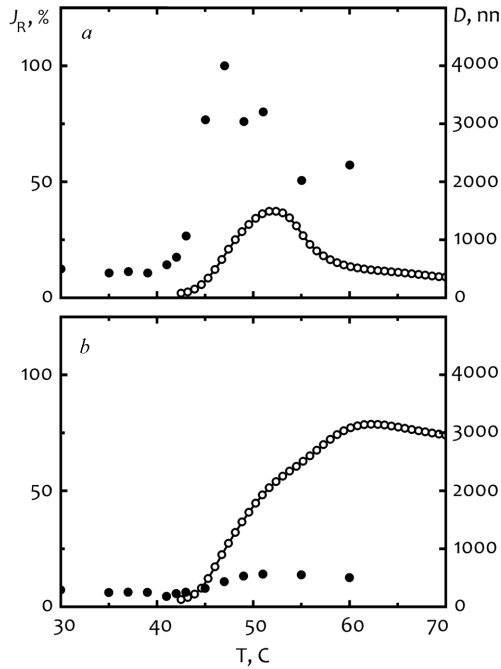


Fig. 2. Temperature dependences of the normalized intensities J_R of the light reflected by dilute HPC solutions: with NaCl salt ions (a), without ions (b). The values of the hydrodynamic diameter D of polymer aggregates (black circles) were obtained in work [28] using the method of dynamic light scattering for the same solutions

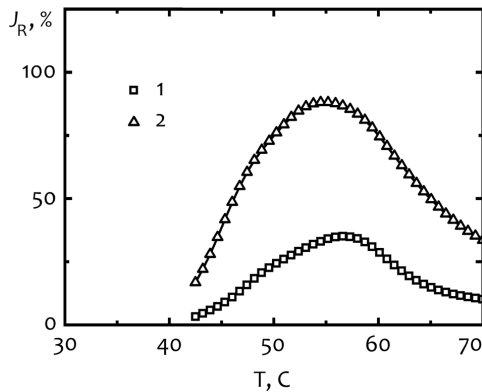


Fig. 3. Temperature dependences of the relative intensities J_R of reflected light for dilute HPC solutions with ions of various salts: NaI (1) and $AgNO_3$ (2)

of reflected light for dilute HPC solutions with and without ions of the NaCl salt. These systems have already been studied by us earlier in work [28], and it was established that in 0.2-wt% HPC solutions at temperatures higher than the LCST and in the pres-

ence of ions, there appear polymer aggregates whose size can be several times larger than the wavelength of visible light.

Let us find a relationship between the size of the particles and the intensity of the light scattered by them. Let a be the radius of a particle, and k be the wave vector magnitude of an incident photon. Taking into account the size of the clusters, we proceed from the Mie scattering theory [29].

The cross-section of the inverse Mie scattering for a single particle has the form [30]

$$\sigma(a) = \frac{1}{a^2 k^2} \left| \sum_{j=1}^{\infty} (-1)^j (2j+1) (A_j(a, k) - B_j(a, k)) \right|^2, \quad (5)$$

where A_j and B_j are the Mie coefficients [31]. According to the calculation results obtained for cross-section (5) in the framework of the method [32, 33], for light with a wavelength of 525 nm and the refractive index value taken from [34] for the aqueous HPC solution, it follows that for $a = (10^2 \div 10^4)$ nm, the dependence $\sigma(a)$ has the scaling behavior

$$\sigma(a) \sim a^s, \quad (6)$$

with the power exponent $s = 2$.

The total cross-section of the inverse Mie scattering by a system of N identical spherical particles equals

$$\Sigma \sim N a^2. \quad (7)$$

Since the amount of polymer in the solution is a constant parameter, the number of such particles equals

$$N \sim 1/a^3, \quad (8)$$

and, therefore,

$$\Sigma \sim 1/a. \quad (9)$$

It follows from formula (9) that

$$J_R \sim 1/a, \quad (10)$$

that is, the intensity of backscattered light increases as the particle size decreases. The same conclusion can be drawn, if we compare the experimental values of J_R and D (see Fig. 2).

From Fig. 2, a, one can see that the experimental dependence $J_R(T)$ for the solution with NaCl ions

has a non-monotonic character: as the temperature increases, the J_R -values first grow, then reach a maximum at T' , and afterward decrease. The sizes of the polymer clusters (see the same figure) have a similar temperature behavior. There are two explanations for the decrease of cluster sizes at $T > T'$. The first of them consists in that the clusters collapse at $T > T'$ and form numerous small fragments. But in this case, as follows from Eq. (10), instead of the reduction of the intensity J_R , we would observe its growth, which contradicts the experiment. The second explanation is that large polymer aggregates disappear from the solution at $T > T'$ owing to their sedimentation, which is confirmed by the experiment: whenever the measurements were concluded, some polymer sediment was found at the bottom of the cell with the solution. This sediment did not dissolve well in a hot solution, but it dissolved easily in cold water.

Note that the viscosity of the 0.2-wt% aqueous solutions of HPC at (50–60) °C exceeds the viscosity of water by only 5–7% [35]. Therefore, the sedimentation of polymer clusters occurred quite quickly: from Fig. 2, *a*, it follows that J_R decreased by a factor of two during about 10 min.

Figure 2 also demonstrates that the sizes D of clusters in the ion-free specimen are several times smaller than in the specimen with ions. By order of magnitude, they are equal to the wavelength of visible light. In Fig. 2, *b*, the intensity of reflected light is larger than that in Fig. 2, *a*, as it should be according to formula (10). The polymer clusters in the ion-free solution were in a suspended state, and practically no sediment was observed in the cell after the measurements.

Figure 3 illustrates the experimental temperature dependences of J_R for dilute HPC solutions with ions of the NaI and AgNO₃ salts. As one can see, these dependences are also non-monotonic, the nature of which was clarified above. At the same time, the intensity of reflected light in the case of the solution with AgNO₃ salt ions (curve 2 in Fig. 3) reaches even larger values than those for the ion-free solution (see Fig. 2, *b*). According to Eq. (10), this fact means that the size of polymer aggregates in the solution with AgNO₃ is the smallest among all four studied specimens. However, unlike the ion-free specimen where small clusters remain suspended for a long time, a noticeable sedimentation occurred in the solution with AgNO₃. This fact indicates that the density

of polymer aggregates in the solution with AgNO₃ was higher than the density of clusters in the ion-free solution. This conclusion is confirmed by the results of works [13–17], where it was shown that silver nitrate is reduced by cellulose derivatives to metal; as a result, polymer aggregates contain silver nanoparticles tens of nanometers in size.

4. Conclusions

The turbidimetric method is applied to study the thermal behavior of HPC aqueous solutions (2 wt%) with additions of NaCl, NaI, and AgNO₃ salts (154 mmol/l). It is found that the introduction of these salts into the solution reduces the surface tension coefficient at the interface between the sol and gel phases, decreases the LCST, and diminishes the rate of sol-gel transition. This result can be used in the controlled drug delivery systems fabricated on the basis of the polymer gel matrix.

Using the method of static light scattering, dilute aqueous solutions of HPC (0.2 wt%) with the admixtures of NaCl, NaI, and AgNO₃ salts (15.4 mmol/l) are studied. It is found that, at temperatures higher than the LCST, there arises inverse scattering of light via the light reflection from the supramolecular structure of the researched solutions. In the solutions with ions, there emerge large macromolecular associations (clusters) whose size is several times larger than the wavelength of visible light. The temperature dependence of the intensity J_R of backward scattering is non-monotonic for specimens with salts; namely, as the temperature grows, the values of J_R increase, reach a maximum, and finally decrease. There is a relation between the values of J_R and the sizes of polymer clusters, which was found in the framework of Mie scattering theory. In particular, it is shown that the reduction of $J_R(T)$ in an interval of (55–70) °C occurs due to the sedimentation of clusters rather than their decay, which was confirmed experimentally.

In our opinion, the aggregation and sedimentation of polymer clusters from dilute solutions in the presence of ions has an important application value for the development of promising technologies, such as catalysts with adjustable activity and film nanocomposite materials [36–40]. In works [13–17], it was shown that composite films based on water-soluble cellulose derivatives with metallic silver nanoparticles have a

high fungicidal and antibacterial efficiencies against both gram-positive and gram-negative bacteria. Taking this fact into account, the aggregation in diluted aqueous solutions of HPC with the ions of NaI and AgNO₃ salts can be used in modern technologies for the production of bactericidal polymeric films used for wound treatment.

The work was supported by the Ministry of Education and Science of Ukraine (project No. 0123U101955 “Molecular mechanisms of physical processes determining the application of hydrogels in military-medical technologies”).

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Received 22.02.24.

Translated from Ukrainian by O.I. Voitenko

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ОСОБЛИВОСТІ ГЕЛЕУТВОРЕННЯ
І АГРЕГАЦІЇ У ВОДНИХ РОЗЧИНАХ
ГІДРОКСИПРОПІЛЦЕЛЮЛОЗИ
З СОЛЯМИ NaCl , NaI І AgNO_3

Турбідиметричним методом та методом статичного розсіяння світла досліджено особливості гелеутворення та агрегації у 2% та 0,2% (мас.) водних розчинах гідроксипропілцелюлози (ГПЦ) з солями NaCl , NaI і AgNO_3 . Встановлено, що введення вказаних солей у 2% розчин зменшує коефіцієнт поверхневого натягу на межі золь- та гель-фази, знижує нижню критичну температуру розчину (НКТР) та сповільнює швидкість золь-гель переходу. З даних по статичному розсіянню світла випливає, що у розбавлених (1:10) водних розчинах ГПЦ при температурі, вищій за НКТР, у присутності солей утворюються значні надмолекулярні асоціації (кластери), розмір яких перевищує довжину хвилі видимого світла у декілька разів. Спостерігалось випадання осадів полімеру. Показано, що інтенсивність відбитого світла може бути описана як функція розміру кластера у рамках теорії розсіяння Мі. Встановлено, що густина полімерних агрегатів у розчині з AgNO_3 є найвищою порівняно з рештою досліджених зразків, що пояснюється відновленням нітрату срібла до металу. Осадження кластерів ГПЦ з наночастинками срібла може бути використано у технологіях створення композитних полімерних плівок з бактерицидною та фунгіцидною активністю.

Ключові слова: гідроксипропілцелюлоза, солі, гелеутворення, агрегація.