

Article

Molecular masses and rheological properties of chitosan and sodium carboxymethylcellulose polymers

M.Sh. Fayziyeva ¹ , J.M. Khakkulov ^{*1} , Xuqing Liu ¹ Faculty of Physics, National University of Uzbekistan named after Mirzo Ulugbek, Tashkent, 100174, Uzbekistan² Northwestern Polytechnical University, Xi'an, 710072, China

j.xaqulov@nuu.uz (M.F.), jmkhakkulov@mail.ru (J.Kh.)

* Correspondence: jmkhakkulov@mail.ru; Tel.: +998 93 0062227 (J.Kh.)

Abstract: This study investigates the molecular characteristics of Na-SMC (sodium carboxymethyl cellulose) and chitosan (ChS), as well as the rheological properties of their polyelectrolyte solutions and mixtures. The molecular characteristics were determined using an Ubbelohde viscometer, while the rheological behavior of the gels under shear conditions was examined using a Reotest-2 rheometer. These studies are primarily related to the fields of materials science, polymer chemistry, and rheology, and are aimed at an in-depth investigation of the physical and chemical properties of these polymer systems.

Keyword: sodium carboxymethylcellulose (Na-SMC), chitosan (ChS), hydrodynamic methods, rheological methods, molecular weight.

Introduction

Today, water-soluble functionally active compounds derived from natural polymers, including polysaccharides such as Na-carboxymethyl cellulose (Na-CMC) [1] and chitosan (CH₃), are widely used in various sectors of the economy. The functional activity of these polysaccharides manifests as polyelectrolyte properties in solutions [2] and causes various phase states and structural formation in chemical reagents, deformation flows, and structural transformations. Therefore, a comprehensive study of these processes and changes, including the study of rheological properties in a stationary thermo-mechanical displacement field [3] and the determination of important parameters of these polymer systems necessary for practice, is one of the urgent tasks of polymer physics.

Scientific research in the field of polymers focuses on determining the relationship between their physicochemical properties and molecular structure. Natural and synthetic polymers such as chitosan and Na-CMC are distinguished by their molecular weight and rheological properties, as well as their wide application in various industrial and biomedical fields. Chitosan, as a deacetylated chitin derivative, possesses properties such as bioactivity, biocompatibility (working in harmony with a biological system), and biodegradability [4] (the material's natural decomposition), and is used in pharmaceuticals, food, and agriculture. At the same time, Na-CMC polymers, as water-soluble materials sensitive to ionic transformations, allow for the control of rheological properties due to their dispersion and gel-forming properties [5].

The molecular weight of the polymer directly affects its rheological behavior in solutions and solutions; high molecular weight often leads to higher viscosity [6] and a strong gel-forming capacity. Therefore, the study of the molecular masses and rheological properties of chitosan and Na-CMC polymers is of great importance for optimizing their technological and biomedical applications.

Materials and Methods

Determining the molecular properties of polymers using the Ubbelode viscosimetry method [6] is usually carried out using hydrodynamic methods. For conducting the research, we chose the Ubellode viscometry method, which is one of the simplest and most convenient hydrodynamic

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methods [7]. Using this traditionally widely used viscometry method, the relative viscosity (η_{rel}) of the polymer solution is determined at several concentrations, i.e.,

$$\eta_{rel} = \tau_i / \tau_0 \quad (1)$$

where τ_i is the flow time of a solution of a specific concentration (Ci) and τ_0 is the flow time of the solvent through the viscometer capillary.

Usually, the flow time of the solvent in the selected viscometer is required to be within the range of 50–150 s. Furthermore, the initial concentration of the solution (C_1) must have a flow time (τ_i) at least twice as long as that of the solvent, i.e., $\tau_i = 2\tau_0$. Determining the specific viscosity as $\eta_{spec} = \eta_{rel} - 1$ and calculating its ratio to the concentration (C), we find the reduced viscosity $\eta_{red} = \eta_{spec} / C$.

In dilute solution systems, the dependence of the reduced viscosity on concentration is expressed by the Huggins equation [?], i.e.

$$\eta_{spec} / C = [\eta] + k[\eta]^2 C \quad (2)$$

In this context, k is the Huggins coefficient; $[\eta]$ is the intrinsic viscosity, which is determined by progressively diluting the polymer solution and measuring its viscosity. This is done by plotting the dependence of η_{spec} / C versus concentration C , and extrapolating the graph to $C \rightarrow 0$. At the hypothetical concentration $C = 0$, $(\eta_{spec} / C) = [\eta]$ is obtained. In this case, the η_{spec} / C versus C plot is required to be linear.

In polyelectrolytes, decreasing the concentration usually leads to changes in electrostatic interactions between polymer molecules, which in turn causes conformational changes. As a result, the dependence of η_{spec} / C on concentration C becomes nonlinear (curved). To eliminate this effect, a certain amount of salt ions is added to the solvent. For example, the addition of 2% NaCl is sufficient to suppress the polyelectrolyte behavior in Na-CMC and chitosan solutions.

Application of the rheological method Rheological studies were carried out using a “Reotest-2” (Germany) instrument.

The device consists of two types of working units: the first is a thermostatic chamber, and the second is a cone-plate system. The thermostatic chamber is a coaxial cylinder system, while the cone-plate configuration provides a working range of shear rates from 0.2 to $4.86 \times 10^3 \text{ s}^{-1}$.

The measurable viscosity ranges from 1 to $4 \times 10^7 \text{ mPa} \cdot \text{s}$. Rheology studies the deformation and flow of materials and also includes their elastic, viscous, and plastic properties.

The studies were carried out in a cylinder-in-cylinder system cell, where one cylinder is placed inside the other ($R_2 > R_1$) (Figure 1). The inner cylinder (R_1) acts as a rotor, and changes in its rotation frequency (ω) are recorded according to the indicator reading (α) on the measurement panel. In this setup, a polymer solution or mixture is filled between the cylinders ($\Delta R = R_2 - R_1$) up to a height h .

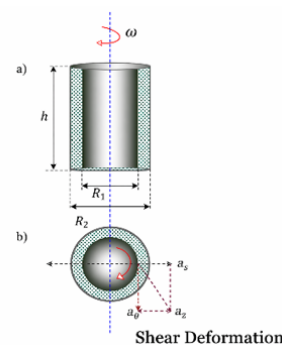


Figure 1. Schematic cross-sections of a coaxial “cylinder–cylinder” cell: a – side view; b – top view

Using this method, shear flow of polymer solutions with different concentrations is generated, in which macromolecules [?] become deformation-oriented and non-Newtonian flow is observed.

In this case, the shear stress (τ) and shear rate (γ – velocity gradient) can be determined using the M. Reiner formula:

$$\tau \approx M/2\pi R_1^2 h \quad (3)$$

$$\gamma \approx 2\pi\omega R/(R_2 - R_1) \quad (4)$$

Here, M is the torque; $R = (R_2 + R_1)/2$ is the average radius; and h is the height of the liquid near the inner cylinder (rotor).

According to Newton's law of viscosity, the effective viscosity (η_{eff}) is determined as the ratio of shear stress to the velocity gradient.

$$\tau/\gamma \approx \eta_{eff} \quad (5)$$

In the Reotest-2 device, the shear stress (τ) is determined at 12 measurement points over the shear rate (γ) range from 0.5 to 1320 s⁻¹ by multiplying the indicator reading (α) by the cylinder constant (z): $\tau \approx \alpha z$. From the plot of $\ln \eta_{eff}$ versus γ , the dynamic viscosity (η) is determined by extrapolating to $\gamma \rightarrow 0$.

Typically, η is measured at different temperatures (T), and the activation energy of viscous flow (E_a) is calculated according to the Frenkel–Eyring equation.

$$\ln \eta \approx \ln A + E_a R/T \quad (6)$$

Here, A is a coefficient, and $R = 8.31$ is the universal gas constant.

Results

To determine the molecular characteristics of the samples, hydrodynamic (viscometric) studies were carried out. First, a 0.2% solution of Na-CMC was prepared using a 2% NaCl aqueous solvent.

The experiments were conducted in the following sequence. Initially, the flow time (τ_0) of the pure solvent (2% NaCl) through the viscometric capillaries was measured. Then, the flow time (τ_i) of the Na-CMC solution through the capillary was determined at five different concentrations, obtained by stepwise dilution. For each concentration, measurements were repeated four times, and the average values were calculated.

From the obtained results, the relative viscosity ($\eta_{rel} \approx \tau_i/\tau_0$) was determined. Based on this, the specific viscosity ($\eta_{spec} = \eta_{rel} - 1$) was calculated. The reduced viscosity ($\eta_{red} = \eta_{rel}/C$) was then obtained from the ratio of specific viscosity to concentration (see Table 1).

Table 1. Calculated viscosity values

| Solution volume, mL | τ_0 , s | τ_i , s | η_{rel} | η_{spec} | η_{red} | C |
|---------------------|--------------|--------------|--------------|---------------|--------------|------|
| 8 | 74,2 | 105,8 | 1,43 | 0,43 | 2,13 | 0,20 |
| 10 | 74,2 | 100,0 | 1,35 | 0,35 | 2,19 | 0,16 |
| 12 | 74,2 | 94,8 | 1,28 | 0,28 | 2,15 | 0,13 |
| 16 | 74,2 | 89,6 | 1,20 | 0,20 | 2,00 | 0,10 |
| 20 | 74,2 | 85,5 | 1,15 | 0,15 | 1,88 | 0,08 |

Based on the Huggins equation, $\eta_{spec}/C = [\eta] + k[\eta]^2 C$, and since $\eta_{rel}/C = \eta_{red}$, a graph of η_{red} versus C was plotted.

By extrapolating to $C \rightarrow 0$, where $\eta_{red} \approx [\eta]$, the intrinsic viscosity was determined to be $[\eta] \approx 1.64$ dL/g (see Figure 2).

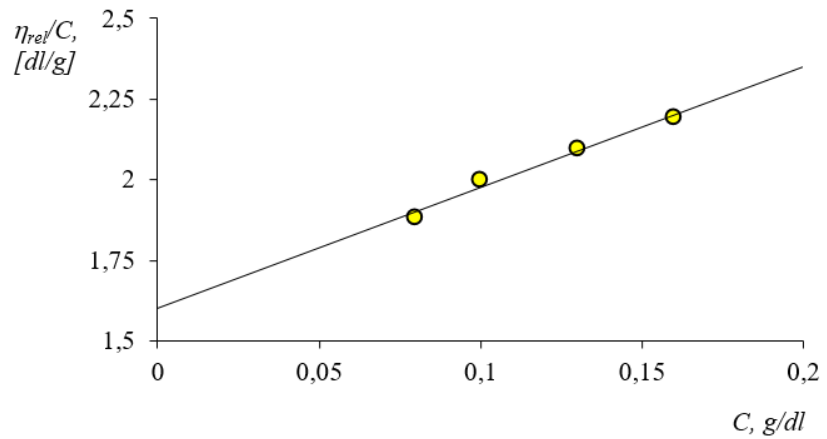


Figure 2. Dependence of reduced viscosity (η_{red}) on concentration (C) for the Na-CMC solution

$$M_{\eta} = ([\eta]/K)^{1/a} = (1.64/2.33 \times 10^{-4})^{1/0.78} = (7039)^{1.28} = 84100$$

The molecular mass (M_{η}) of Na-CMC was calculated based on the Mark–Kuhn–Houwink equation, and it was determined to be $M_{\eta} = 84100$.

Also, the Ubbelohde viscometry method was used to determine the molecular mass (M_{η}) of the obtained chitosan. For this purpose, a 0.2 g/dL chitosan solution was prepared in 2% acetic acid (2% CH₃COOH–water). Due to the polyelectrolyte nature of the solution, 2% NaCl was added to eliminate concentration anomalies. Viscosity measurements were carried out at different concentrations (C), and based on the Huggins equation:

$$\eta_{rel}/C = [\eta] + k[\eta]^2C$$

The η_{rel}/C –C dependence graph was plotted (Figure 3). By extrapolating the linear plot to $C \rightarrow 0$, the intrinsic viscosity $[\eta] = 1.8$ dL/g was obtained. Using this value, the molecular mass (M_{η}) of chitosan was determined based on the Mark–Kuhn–Houwink equation:

$$M_{\eta} \approx ([\eta]/K)^{1/a} \approx ([\eta]/1.4 \times 10^{-4})^{1/0.83} = 89200$$

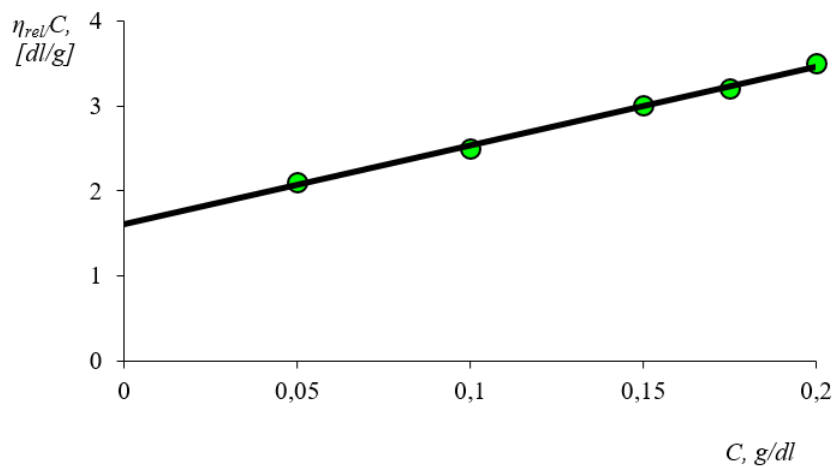


Figure 3. Dependence of reduced viscosity (η_{red}) on concentration (C) for chitosan solution

The rheological properties of the Na-CMC:Chitosan (90:10) based gel were studied. The investigations were carried out in a shear field at different temperatures. The deformation-induced

breakdown of the gel and its transition to a viscous flow state, as well as the dependence of its effective viscosity on the shear rate, are presented in Figure 4.

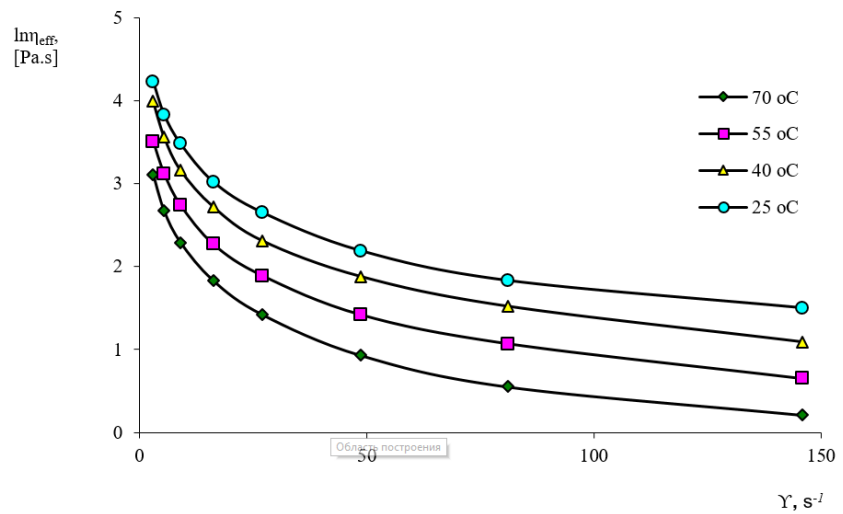


Figure 4. Rheograms of Na-CMC:Chitosan (90:10) gel (5%) in a shear field at different temperatures: 25°C, 40°C, 55°C, and 70°C

It was found that with an increase in temperature (t), the dynamic viscosity (η) decreases, and this behavior is presented graphically in Figure 5. This phenomenon is explained by the weakening of intermolecular interactions within the gel under thermal influence. Since the studied gels belong to the group of physical gels, the junction points forming their structure are easily disrupted under external thermomechanical effects.

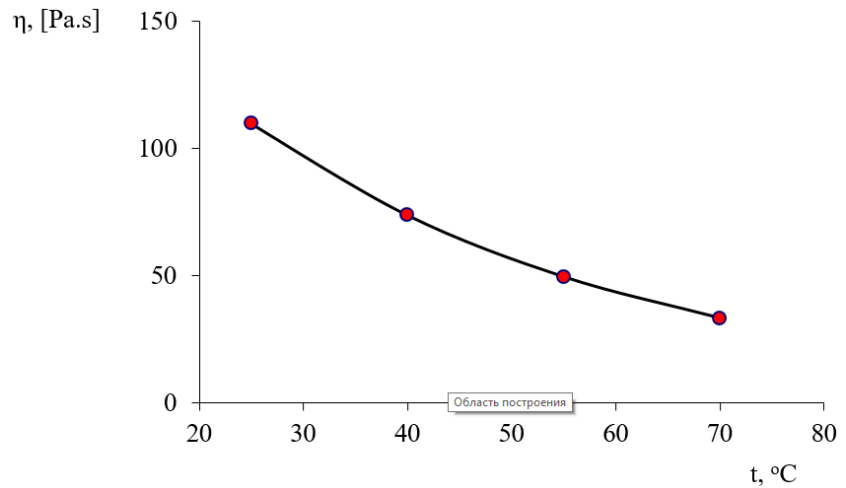


Figure 5. Temperature (t) dependence of the dynamic viscosity (η) of Na-CMC gel

The dependence of the effective viscosity ($\ln \eta_{eff}$) of Na-CMC:Chitosan gel (5 g/dL), which exhibits a viscous-flow state due to breakdown under shear flow, on the shear rate (γ) is presented in Figure 6. Such a relationship is characteristic of all selected temperatures, and it has been observed that an increase in temperature (i.e., a decrease in the $1/T$ value in the graph) leads to an exponential decrease in the effective viscosity.

The occurrence of an exponential relationship is primarily associated with changes in the activation energy of intermolecular interactions. The rheological method for determining this energy is based on the Frenkel–Eyring equation (see formula 6).

Based on the slope of the relationship graph shown in Figure 6, constructed according to the equation $\ln \eta = \ln A + (E_a/R) \cdot (1/T)$, the activation energy (E_a) was determined.

$$E_a/R = (\ln\eta_1 - \ln\eta_2)/[(1/T)_1 - (1/T)_2] = (4,7 - 3,5)/(3,3 - 2,9) * 10^3 = 3 * 10^3$$

$$E_a = 3 * 10^3 R = 3 * 10^3 * 8,31 = 24930 \text{ J/mol} = 24,93 \text{ kJ/mol}$$

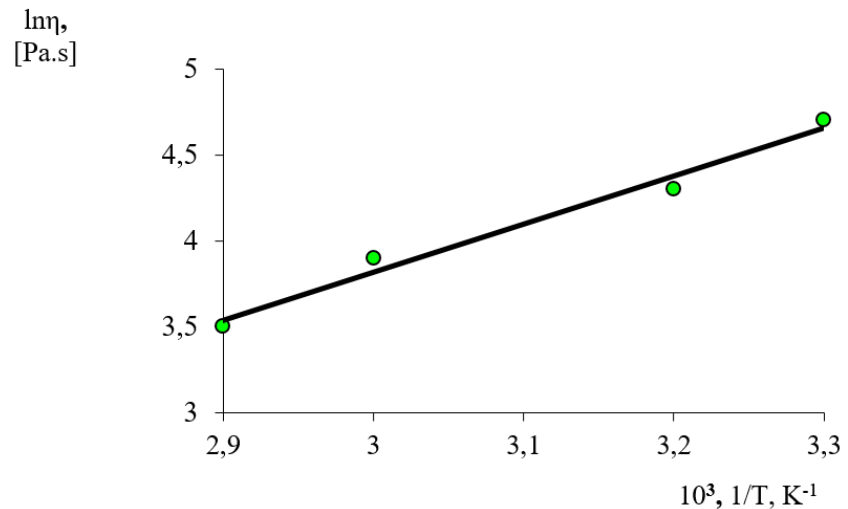


Figure 6. Dependence of the dynamic viscosity ($\ln \eta$) on inverse temperature ($1/T$) for Na-CMC:Chitosan gel

Conclusions

Rheological studies showed that Na-CMC and Na-CMC:Chitosan (90:10)-based gels undergo deformation-induced breakdown under shear field and transition into a viscous-flow state, exhibiting effective viscosity behavior characteristic of non-Newtonian fluids depending on the shear rate. It was also found that their viscosity values decrease with increasing temperature.

Comparative studies revealed that the activation energy of the viscous-flow process corresponds to the range of hydrogen bond energies and is higher in the Na-CMC:Chitosan (90:10) gel. This is attributed to strong interactions (ionic and hydrogen bonding) formed between carboxyl groups of Na-CMC and amino groups of chitosan.

Authors' contribution

Original draft preparation, rheological measurements, data curation, and formal analysis: J.Kh. and X.L.; laboratory research for molecular weight determination: M.F.

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Ethics approval.

Since this study does not involve human or animal subjects, ethical review and approval is not required. Therefore, ethical approval does not apply to research.

Consent for publication

Human participants were not involved in this study. For this reason, informed consent is not required.

Data Availability Statement

All experimental data confirming the results presented in this article are held by the authors and presented within the article in the form of the main text and image/table. Additional information can be obtained from the author(s) based on a reasoned query

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Conflict of interest

The authors declare that there is no conflict of interest in this study. There are no personal, financial or other interests that affect the presentation or interpretation of research results.

Abbreviations

| | |
|--------|--------------------------------|
| Na-CMC | sodium carboxymethyl cellulose |
| Ch | chitosan |

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