# Viscosity properties of sodium carboxymethylcellulose solutions

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Abstract Through viscosity measurements, concentration and temperature dependences of viscosity of sodium carboxymethylcellulose (CMC) solution were recorded. Effects of glycerin, mechanical shearing and several electrolytes on the CMC solution were also determined. Results showed that the viscosity dependence on concentration obeyed the Huggins and Kramer equation, the dependence on temperature complied with the Arrhenius equation. CMC chain could synergize with glycerin, konjac glucomannan (KGM), and aluminum sulfate 18 hydrate. Sodium chloride, hydrochloric acid, and calcium dichloride reduced the viscosity of the CMC solution. By suggesting the ion-binding and hydrogen bond as the major form of the electrostatic interaction in the CMC solution, the synergistic and pseudoplastic phenomena as well as the maximum over stirring time were reasonably explained.

**Keywords** Electrostatic interaction  $\cdot$  Ion-binding  $\cdot$ Hydrogen bond · Polyelectrolyte · Sodium carboxymethylcellulose · Viscosity

# Introduction

Sodium carboxymethylcellulose (CMC) is a derivative of cellulose formed by its reaction with alkali and chloroacetic acid. Purified CMC is a white to offwhite, non-toxic, odorless, biodegradable powder, which can be dissolved in hot or cold water. CMC is used for a variety of applications in a number of industries, including the food, personal care, pharmaceutical, oilfield and paper industries due to the superior properties as a binding, thickening, and stabilizing agent in these end uses. The most important character that makes them useful in these applications is high viscosity in low concentration. Literature has reported some progress in this basic concern about both CMC solution and mixture. After the apparent viscosity of CMC solutions as a function of shear rate, temperature, and concentration was modeled (Andriana et al. [2002](#page-7-0)), the steady shear viscosity of aqueous CMC solution was measured within the power-law range over temperature and concentration (Lin and Ko [1995](#page-7-0)). By studying the Newtonian behavior of aqueous CMC solution, effect of shear-induced recombination of CMC macromolecular crystallites was proposed (Jayabalan [1989](#page-7-0)). Recent years, rheological behavior of CMC in aqueous solution from non-wood pulps was synthesized and characterized (Barba et al. [2002](#page-7-0)). Mixtures of CMC with natural or synthesized polymers behave more complicated viscous properties and attract more attentions. In order to evaluate synergistic/non-syn-

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ergistic effects of mixed polysaccharide systems, rheological character of the mixture of CMC with xanthan was studied under destructive and nondestructive shear conditions (Florjancic et al. [2002](#page-7-0)). By means of viscosity measurement, the CMC interaction with mucin (Rosi et al. [1996\)](#page-8-0) and with 5-fluorouracil (Nishida et al. [1982\)](#page-8-0), an anticancer drug, were reported. Although these works extended the knowledge about the viscosity of the CMC solution, aspects of explanation are not sufficient to guide the CMC application.

The sodium CMC molecular structure is based on the  $\beta$ -(1 $\rightarrow$ 4)-D-glucopyranose polymer of cellulose. Different preparations may have different degrees of ionizable group substitution, but it is generally in the range 0.6–0.95 derivatives per monomer unit. When it dissolves in water, an electrolytic process takes place to separate a CMC molecule into sodium cations and a polymer anion. In this sense, the CMC belongs to polyelectrolyte. These ions in solution interact with each other through electrostatic forces. In addition to this, the water molecule and OH groups on the CMC molecule exhibit electric dipole that performs considerable electrostatic interacting force (the so called hydrogen bond). These electrostatic interactions play a key role to understand the viscosity of the CMC polyelectrolyte solution. Regarding this important issue, investigations have been carried out with experiment on the CMC solution filled with low molecular weight salt and with theoretical model for polyelectrolyte. The effect of polyion charge on specific viscosity of CMC (Trivedi et al. [1987\)](#page-8-0), and viscosity behavior of multivalent metal ion-containing carboxymethyl cellulose solutions (Thomas et al. [2003\)](#page-8-0) were studied. Rheological properties of CMC aqueous solution with sodium and chromium chloride salts were measured (Matsumoto and Mashiko [1988](#page-7-0); Andreeva et al. [1992\)](#page-7-0). Polyelectrolyte effects in the CMC water-cadoxene solution was studied by translational diffusion and viscometry methods (Okatova et al. [1990](#page-8-0)). Although several theoretical models (Markus et al. [1997;](#page-7-0) Kunimasa et al. [2004](#page-7-0); Dobrynin and Rubinstein [2005](#page-7-0)) for the reduced viscosity of polyelectrolyte solution may be suitable to the CMC solution, correlation between electrostatic interaction and the viscosity properties of the CMC solution is not understood well. For example, the influence of ion-binding on the viscosity properties of the CMC solution has seldom been reported. Because of the wide applications of CMC and its typical representation for polyelectrolyte, investigation into the electrostatic interaction by viscosity measurement has practical and theoretical values.

In this paper, the viscosities of CMC solutions will be measured over concentration and temperature. And then, the influences of several added salt ions, glycerin, konjac glucomannan (KGM) and shear rate will be determined. Recorded curves will be respectively fitted to Huggins and Kramer equation (Mothe´ and Rao [1999](#page-8-0)), Arrhenius equation (Rubinstein and Colby [2003\)](#page-8-0), the equation for polyelectrolyte solution and Williamson model. Obtained result will be discussed with electrostatic and dynamic interactions in the solution.

### Experimental

## Materials

Sodium CMC in this study was made by Shanghai Reagent Co., Ltd., of Sinopharm. It was a white to off-white, non-toxic, odorless, biodegradable powder, which obeyed the Q/CYDZ-03-92 specification. Sodium content located in 6.5–8.5%, chloride compound  $\leq 3.0\%$ , water  $\leq 1.0\%$  in weight. The CMC were directly dissolved in distilled hot water to obtain the solution samples. KGM sample used in our experiment was supplied by Hubei Enshi Hongye Konjac Development Co., Ltd., which had principal properties: granularity  $\leq 220$  µm, glucomannan content  $\geq$  95%, protein content <0.6%, ash content <3%. The other reagents were chemical grades.

#### Methods

For the samples, a NDJ-1 viscometer, equipped with column geometries of four scales numbered 1–4 and working in steady shear mode, was used to measure the viscosities. An electromantle water bath was used to control the temperature. To measure the shear rate dependence, a rheometer AR500 by TA instruments was set to steady state flow. A standard steel parallel plate with  $1,000 \mu m$  gap and 40 mm diameter was chosen for the steady flow measurement.

The fitting results were evaluated by a standard error defined in Eq. (1).

$$
\frac{\sqrt{\sum (x_m - x_c)^2}}{n}
$$
\n
$$
range
$$
\n(1)

where  $x_m$  was the measured value and  $x_c$  was the calculated value of  $x$  for each data point,  $n$  was the number of data points, and the range was the maximum value of  $x_m$  minus the minimum value. Generally speaking, a reasonable fit gives a standard  $error \leq 3\%$ .

## Results and discussion

1300 1400 1500

Dissolving process with mechanical stirring was the necessary step to make sodium CMC solution, therefore it was important to measure the viscosity during this process. The measured viscosity data at 50 rounds/min stirring angular velocity was presented in Fig. 1 as hollow diamonds with smoothly lined curve for clarity. It showed that viscosity initially increased to a maximum, and then decreased to a stable value from the moment when CMC was added in water to a sufficient long period. Firstly, a part of solid CMC dissolving in water resulted in a sharp viscosity increase, while the others remained as suspended powder, neither swelling nor dissolving. With continuous stirring, the majority of the solid CMC had dissolved into the water reaching a point of



Fig. 1 Viscosity variation during the dissolving process of CMC in water with a final concentration of 3% by mechanical stirring at 50 rounds/min angular velocity

maximum viscosity without complete disaggregation of CMC molecule. Finally, the CMC molecules reached maximum disaggregation corresponding to an unchangeable viscosity value. These phenomena in the dissolving process proposed that the CMC solution had its maximum viscosity at an intermediate state at which the CMC polymer remained some molecular binding.

In order to obtain a basic knowledge into the flow properties of the sodium CMC solution, the dependences of viscosity upon concentration in weight percent and temperature were showed in Figs. 2 and [3](#page-3-0) as smoothly lined points of various shape (The following figures mean the same). Viewing Fig. 2, it was clear that the viscosity exponentially increased with concentration. A lot of polymer solution had this tendency that was classically described by the Huggins and Kramer equation (Mothe´ and Rao [1999](#page-8-0)),

$$
\frac{\ln \eta_r}{c} = [\eta] + k_2 [\eta]^2 c \tag{2}
$$

where  $\eta_r = \eta/\eta_s$  was the relative viscosity,  $\eta$ ,  $\eta_s$  the viscosities of the solution and the solvent,  $\eta$  in a square bracket  $\lceil \eta \rceil$  was a variable traditionally denoting the intrinsic viscosity, c the concentration,  $k_2$  the constant. After curve in the Fig. 2 was fitted to Eq. (2), good agreement was obtained. The corresponding parameters were numerically determined as  $[\eta] = 115$ ,  $\eta_s = 31.6$  mPa.s,  $k_2 = 0$ . Standard error between the experimental data and the theoretical data calculated by Eq. (2) with this set of constants was 1.8%.



Fig. 2 Viscosity of CMC solution against concentration in weight percent

<span id="page-3-0"></span>

Fig. 3 Viscosities of CMC solutions at 1, 3, 5% concentrations versus temperature

Figure 3 showed that the viscosities of CMC solutions behaved like most water-soluble polymers, whose viscosity decreased when temperature increased. The measured data could be satisfactorily fitted to Arrhenius equation given by

$$
\eta(T) = A \exp\left(\frac{E}{RT}\right) \tag{3}
$$

where E was the activation energy and  $R = 8.31$  J/  $K$ .mole was the gas constant, T was absolute temperature, and A was a pre-exponential constant. The fitted parameters A and E for 1, 3, 5% CMC solutions were  $2.29 \times 10^{-4}$  mPa.s and  $3.17 \times 10^{4}$  J/ mole,  $6.50 \times 10^{-4}$  mPa.s and  $3.46 \times 10^{4}$  J/mole,  $3.72 \times 10^{-3}$  mPa.s and  $3.61 \times 10^{4}$  J/mole, respectively. Standard error between experimental and theoretical data for these sets of constants were within 2.1%.

A number of literatures have reported similar results for the viscosity of neutral hydrocarbon polymer solution varying with concentration and temperature. They were commonly interpreted with both the dynamics of the neutral polymer chains in solution and the stochastic effect of the temperature. In the case of sodium CMC solution, which belonged to a polyelectrolyte solution, electrostatic interaction among ions globally existed in the solution. Not only the polyions electrolysed from the CMC interacted with the small counterions which rendered the system electroneutral, but also the electric dipole of the OH group on the CMC molecule joined in the interaction. As a matter of fact, the CMC solution was a system full of electrostatic interactions, by which ion-binding and hydrogen bond might connect two or more ployions as an effective larger polyion. Just like the neutral polymer solution, the temperature stochastic process in the solution tended to separate some bound ions to an equilibrium state. There was thus a balance at which the entire interacting mechanism in the solution reached. Each balance corresponded to an activation energy value in Eq. (3), which determined the temperature dependence of viscosity.

Beyond these basic properties, sodium CMC solution performed much more interesting flow properties in the presence of low molecular weight electrolytes, which introduced cation and anion into the solution, such as NaCl. The properties of the CMC solution could be obviously altered by these ions. Figure 4 illustrated the influences of various ions from added salts on the viscosity of 1% CMC solution. It showed that sodium chloride, hydrochloric acid, calcium dichloride and ferric chloride decreased the viscosity when increasing their molar concentration. However, aluminum sulfate 18-hydrate increased the viscosity while increasing its molar concentration to reduce the pH value of solution from 7 to 6. For aluminum ion, a similar result was previously reported for  $Al^{3+}$  to form CMC solution as rigid elastic gel (Elliot and Ganz [1974](#page-7-0)).

Mechanically, viscosity is the ratio of stress and shear rate, which describes the ability for faster fluid layer to draw slower fluid layer. The more expanded polymer in solution can connect more distant layers, so enhances the ability to transfer drawing force from



Fig. 4 Effects of various salts on the viscosity of 1% CMC solution

faster to slower fluid layers, and results in higher viscosity in the solution. In the presence of low molecular weight ions, the ionized –COO groups on CMC chain was electrostatically shielded and the CMC chains then adapted a less expanded structure. The viscosities were thus reduced by the small ions, such as sodium cation, chloride anion, hydrogen cation, and so on. For gel formation, a higher concentration was thus required in the presence of salts than for a small ion free medium. However, when the aluminum sulfate dissolved in the CMC solution, hydrolyzing and polymerizing reactions occurred in the solution to produce colorless and viscous Al(OH)3 colloid. This colloid itself increased the viscosity of the CMC solution. Futhermore, the OH groups on the colloid had the steric capability of binding CMC polyions in solution through hydrogen bond. Therefore, the viscosity of CMC solution was actually increased by two mechanism. The hydrolyzing and polymerizing reactions are as follows,

Hydrolyzing reaction  $2Al_2(SO_4)_3 + 2n H_2O =$  $2\text{Al}_2(\text{OH})_{n} \cdot (\text{SO}_4)_{3-n/2} + n\text{H}_2\text{SO}_4$ 

Polymerizing reaction  $mAl_2(OH)_n$  (SO<sub>4</sub>)<sub>3-n/2</sub> =  $[Al_2(OH)_n \cdot (SO_4)_{3-n/2}]_m$ 

The electrostatic shielding effect commonly exits in polyelectrolyte solution. By considering the influence of charges on the single hydrodynamics of a polyelectrolyte and the cooperative coupling of all particles, a formula for viscosity of polyelectrolyte solution at low polymer concentration was calculated (Hess and Klein [1983](#page-7-0)). It was expressed as a reduced viscosity,

$$
\eta_{\text{red}} \propto \frac{c_p Z_{\text{eff}}^4}{\left(\frac{2M_s}{M_p}c_s + Z_{\text{eff}}c_p\right)^{3/2}}\tag{4}
$$

where  $Z_{\text{eff}}$  was the effective charge number per polymer,  $M_p$  and  $M_s$  the molecular weights of the polymer and low molecular weight salt in solution,  $c_p$  and  $c_s$  the concentrations of the polymer and salt. When considering the dependence of viscosity on the molar concentration of added salt, it was beneficial to assume this formula could be extended to the solution at high concentration of this paper by fixing the other variables at constant. On this assumption, a formula describing the effect of added salt on the CMC viscosity was proposed as,

$$
\eta = \eta_a \frac{c_a^{3/2}}{(c_s + c_a)^{3/2}} \tag{5}
$$

Table 1 Fitted parameters to Hess and Klein formula

Added salt	$\eta_0$ (mPa.s)	$c_0$ (mol/L)	Standard error $(\% )$	
<b>NaCl</b>	105	0.0958	3.2	
HCl	112	0.0358	3.9	
$CaCl2 \cdot 2H2O$	113	0.0453	4.2	
FeCl <sub>3</sub>	113	0.0309	4.6	

here,  $c_a$  was a constant, and  $\eta_a$  was the viscosity amount in the absence of added salt. It was clear that the variation trend of this equation qualitatively agreed with measured data in Fig. [4](#page-3-0) for sodium chloride, hydrochloric acid, calcium dichloride and ferric chloride. Quantitative numerical fitting results were listed in Table 1 for these salts. Standard error between the experimental data and the theoretical data calculated with Eq. (5) were within 5%.

As an example of the importance of order of addition for the small ions, Fig. 5 was a graph of viscosity versus salt concentration at 2% CMC concentration. In one case, the CMC was dissolved in the water before the sodium chloride salt, and the salt had a minimal effect on the viscosity of the solution. In the other case, the CMC was dissolved after the salt, and the resulting final viscosity was much lower, especially as the salt concentration increased. As pointed out above, if the salt was dissolved in water before CMC, the salt ion promptly shielded the dissolved CMC chains stopping the expansion. In contradiction, the salt ion could not depress all the expansion by shielding if the salt were



Fig. 5 The viscosities of CMC solution with sodium chloride addition when the salt added after and before CMC

dissolved after CMC. The higher ion concentration the larger stoppage in the solution leaded to more viscosity drops. This effect moderately reduced the viscosity when the molar concentration of cation increased. Furthermore, a fraction of the CMC chains might have bound with each other before the salt dissolved. This might be the other reason for the viscosity of the solution had much higher viscosity value when the salt was added after CMC than before CMC.

Figures 6 and 7 showed examples of the effect of water/non-solvent mixtures on the viscosity of CMC solution at various concentrations and temperatures. In this test, the non-solvent was glycerin. With increasing glycerin concentration, the viscosity went up. The maximum value was reached with a 30/70



Fig. 6 CMC viscosity in water–glycerin solution at 1, 3, 5% concentration



Fig. 7 CMC viscosity in water–glycerin solution at 20, 40,  $60^{\circ}$ C

mixture of water and glycerin. At higher than 70% concentrations of glycerin, the CMC was not fully dissolved in solution and thus did not give as much viscosity. There were three OH groups on a glycerin molecule. Unlike molecules prefer to interact with each other. So it was more reasonable to assume hydrogen-bonds on a glycerin molecule bound three CMC molecules as an effective larger molecule. According to Mark–Houwink equation, this effect (the so called synergy) increased the viscosity of the solution.

Other hydrocolloids can also give a synergistic viscosity increase with sodium CMC. Figure 8 presented an example. If one were to mix a 2% KGM solution of 340 mPa.s with a 2% CMC solution of 380 mPa.s, the net result was not the 360 mPa.s average of the two; it would be higher than the average value. With the ratio of CMC solution to total solution rising from 0 to 100%, the viscosity went up to a maximum, and then gradually dropped down to the average value. As is well known that KGM molecule contains glucose and mannose on which there are rich hydroxyls. The hydroxyl would form hydrogen bonding between the KGM and CMC molecules in the solution. There were more average electrostatic attractions (hydrogen bonding) between unlike molecules, which resulted in this synergistic viscosity increase. The occurrence of the interactions between KGM and CMC molecular chains through hydrogen bond was previously found in blend films of KGM and CMC by using FT-infrared, wide-angle X-ray diffraction, and differential thermal analysis (Xiao et al. [2001](#page-8-0)). The ratio of synergistic viscosity



Fig. 8 Synergistic viscosity with konjac glucomannan

to average value could be roughly estimated by using Mark–Houwink equation. Considering a simple binary interaction, a hydrogen bond connected a KGM and a CMC chain as an effective chain that had the molecular weight of their sum. Keeping concentrations of CMC and KGM at constant and ignoring the small effect of solvent viscosity, the ratio of synergistic viscosity to average value could be derived as

$$
ratio \approx \frac{(M_{\text{CMC}} + M_{\text{KGM}})^a}{\frac{1}{2}(M_{\text{CMC}}^a + M_{\text{KGM}}^a)}\tag{6}
$$

Here,  $M_{CMC}$  and  $M_{KGM}$  respectively denoted the molecular weight of the CMC and KGM polymers, a the Mark–Houwink constant that usually takes the amount in  $0.5 \sim 1$ . No matter what amounts the molecular weights took, the numerical value of Eq. (6) was bigger than 1 to show a viscosity synergy.

Figures 9, 10 demonstrated changes of viscosity with shear rate at different sodium CMC concentrations and temperatures. Measured results showed totally reversible pseudoplastic (or shear-thinning) properties, which meant that the viscosity decreased at increasing shear rate. As soon as the shear stopped, the viscosity returned to its original value. These pseudoplastic properties could be attributed to CMC's long chain molecules that tended to orient themselves in the direction of flow. As the shear stress was increased, the more chains rearranged themselves along the shear direction, then the shear resistance to flow (viscosity) decreased. When a lower stress was imposed on the same solution, the



Fig. 9 Dependence of the viscosity of CMC solution upon shear rate at 1, 3, 5% concentration and 20 $\degree$ C



Fig. 10 Dependence of viscosity of CMC solution upon shear rate at 20, 40, 60 $\degree$ C and 3% concentration

viscosity was higher because random molecular orientations exhibited increased resistance to flow. In addition to this orientation effect, there were enough OH groups on a CMC chain, which could cause hydrogen bonding among CMC polymers. The electrostatically bound state of the CMC chain in the solution was not stable. Strong mechanical shearing could break some hydrogen bonds in the solution leading to smaller effective molecular weight. According the Mark–Houwink equation, smaller molecule weight corresponded to lower viscosity. Due to the relatively high bonding energy, it suggested that this mechanism preferred to cause pseudoplastic property in higher shear rate regime. The reversible pseudoplastic property of CMC solution reflected the break and recover of both hydrogen bond and random molecular orientation.

Due to the behavior of the CMC solution was pseudoplastic, the most suitable model to fit the experimental curves of viscosity versus shear rate in Figs. 9 and 10 might be the Williamson model given by

$$
\eta = \frac{\eta_0}{1 + (k\dot{\gamma})^n}
$$

where  $k$  was the consistence index,  $n$  was the flow behavior index and  $\eta_0$  was the zero-rate viscosity. Numerically fitting the data in Figs. 9 and 10 to this equation, parameters were determined in Table [2.](#page-7-0) Standard errors between the experimental data and the theoretical data calculated with Eq. (7) were consistently within 2%. At the same temperature, all

<span id="page-7-0"></span>Table 2 Fitted parameters to Williamson model

Condition	$\eta_0$ (mPa.s)	K(s)	n	Standard error $(\% )$
5\% at 20 $^{\circ}$ C	$1.12 \times 10^{4}$	0.198	0.742	1.2.
3\% at 20 $^{\circ}$ C	$1.12 \times 10^{3}$	0.0879	0.593	1.6
1\% at 20 $^{\circ}$ C.	$1.20 \times 10^{2}$	0.0741	0.409	1.5
3\% at 40 $^{\circ}$ C	$5.00 \times 10^{2}$	0.0917	0.438	1.9
3\% at 60 $\degree$ C.	$1.66 \times 10^{2}$	0.0129	0.410	1.4

the three parameters increased when concentration rose. Among the three parameters the most sensitive one was the zero-rate viscosity  $\eta_0$ .

# Conclusion

In this paper, the viscosities of sodium CMC solutions over concentration, temperature, and the influences of several added salt ions, glycerin, KGM and shear rate were determined. Recorded curves were respectively fitted to Huggins and Kramer equation, Arrhenius equation, the equation for polyelectrolyte solution and the Williamson model. By suggesting the electrostatic and dynamic interactions as well as temperature stochastic process as the fundamental mechanism in the CMC solution, the measured data were reasonably interpreted. It was concluded that:

- 1. The concentration and temperature dependences of viscosity of CMC solution obeyed the Huggins and Kramer equation and the Arrhenius equation, respectively.
- 2. CMC chain could synergize with aluminum ion, glycerin and KGM. This character provided a useful method to make CMC product of special properties, which was required by the increasing industrials.
- 3. Electrostatic interaction globally existed in the CMC solution. It was the key factor to explain the viscous properties of CMC solution. Ion binding and hydrogen bonding were the most important interactive forms.
- 4. The balance of hydrogen bonding in CMC solution was at quasi-static, which could be broken by strong mechanical shearing. This mechanism might be one of the reasons of the

pseudoplastic property of the CMC solution in the high shear rate regime.

Investigation into the complicated interaction due to Coulomb force in the polyelectrolyte solution is one of the most important theoretical problems that interest the polymer chemist. CMC not only has the properties of both polymer and electrolyte, but also is a typical example of the polyelectrolyte. Appending the wide variety of application of CMC, the presented results in this article have theoretical and practical values.

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