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Rheological properties of hydroxypropylmethyl cellulose/sodium dodecylsulfate mixtures

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Abstract: The rheological properties of mixtures of hydroxypropylmethyl cellulose (HPMC), a nonionic associative cellulose ether, and sodium dodecylsulfate (SDS), an anionic surfactant, were investigated by viscosity measurements performed at different shear rates (0.1–6000 s^{-1}). HPMC/SDS mixtures containing different concentrations of SDS (c_{SDS}, 0.00–3.50 mass %) and HPMC concentrations, which corresponded to the overlap parameter c/c^{*} of 3, 6 and 12, were prepared. All HPMC/SDS mixtures were found to be shear-thinning when examined in the low-end to mid-range of the applied shear rates. The degree of shear-thinning, *n*, and the viscosity of the mixtures were influenced by composition of the HPMC/SDS mixtures and HPMC–SDS complex formation. The changes in *n* ranged from values typical for highly shear-thinning to almost perfectly Newtonian liquids, and were more pronounced as c/c^{*} was increased from 3 to 6 and 12. A change in the flow profile and a buildup of the first normal stress difference (N_1) was observed in HPMC/SDS mixtures with c/c^{*} = 6 and 12 and c_{SDS} 0.55–1.00 and 0.55–2.50 mass %, respectively, when a critical shear rate, $\dot{\gamma}_{crit}$, was exceeded, suggesting that a shear-induced structure formation in the mixtures occurred.

Keywords: polymer–surfactant interaction; HPMC–SDS interaction; shear-thinning; shear-thickening; shear-induced structure formation.

INTRODUCTION

Polymers and surfactants are common components of many products of the food, pharmaceutical and chemical industry. An interaction between a polymer and a surfactant often occurs when they are jointly found in a solution. Polymer–surfactant interaction influences the physicochemical properties of solutions and is often employed to achieve different effects, such as emulsification, colloidal

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stability, viscosity enhancement, gel formation, solubilization, phase separation *etc*. Details of polymer–surfactant interaction depend on the molecular characteristics and concentration of both polymer and surfactant.[1–5]

Hydroxypropylmethyl cellulose (HPMC) is a nonionic, water-soluble cellulose ether. It is obtained by partial substitution of hydroxyl groups of cellulose with hydrophobic hydroxypropyl and methyl groups. The substituents make HPMC a typical amphiphilic polymer with properties such as ability to adsorb at air–water and oil–water interfaces, emulsification, self-assembly and association with other amphiphilic molecules.[6–9] In this regard, addition of low molar mass surfactants, especially anionics such as sodium dodecylsulfate (SDS), to HPMC solution may result in a polymer–surfactant interaction. HPMC–SDS interaction takes place when the SDS concentration ($c_{\rm SDS}$) exceeds the critical association concentration (*CAC*), which is the minimal surfactant concentration required for the onset of association of a surfactant and a polymer.[1,10] HPMC–SDS interaction occurs *via* hydrophobic moieties of the components, where SDS binds to the HPMC chains and thereby brings about HPMC–SDS complex formation.[11–14] The binding and complex formation supports physical cross-links between the entangled HPMC chains, which result in an increase in the viscosity of HPMC/ /SDS mixtures.[11,15] Simultaneously, the formation of negatively charged SDS micelles along the HPMC chains progressively converts the non-ionic polymer into a polyanion. As the $c_{\rm SDS}$ is further increased, electrostatic repulsive forces between neighboring HPMC chains start to dominate, the network structure is gradually lost and consequently, the viscosity of the HPMC/SDS mixture falls. Individual HPMC chains become fully solubilized with SDS when the $c_{\rm SDS}$ reaches the polymer saturation point, *PSP*. Increasing the $c_{\rm SDS}$ above the *PSP* causes only a slight decrease in viscosity of HPMC/SDS mixtures because of the formation of free SDS micelles in the solution, which brings about slight conformational changes of the SDS-solubilized HPMC chains.[14]

Rheological measurements are often used to study interaction of an associative polymer and a surfactant in a solution in which polymer chains are entangled, *i.e.*, when the concentration of the polymer is above the overlap concentration (c^*).[12,16,17] The degree of entanglement is often expressed as the overlap parameter c/c^*, which is the ratio of the actual polymer concentration in a solution and its overlap concentration.[18] In rheological studies of polymer–surfactant interaction, a zero-shear viscosity, *i.e.*, the viscosity of a polymer/ /surfactant mixture at zero shear rate, is commonly used to quantify the changes in mixtures. The influence of the shear rate on the viscous properties of HPMC/ /SDS mixtures has rarely been reported.[15,19] The goal of the present work was to investigate the rheological properties of HPMC/SDS mixtures containing different concentrations of SDS ($c_{\rm SDS}$, 0.00–3.50 mass %) and different degrees of entanglements of the HPMC chains ($c/c^{\ast}$ of 3, 6 and 12) by measuring the viscosity of the mixtures performed at different shear rates (0–6000 s^{-1}).

EXPERIMENTAL

Materials

Hydroxypropylmethyl cellulose, HPMC (trade name Methocel K4M CR, methoxyl content 22.7 mass %, hydroxypropyl content 8.9 mass %) was obtained from Colorcon Ltd., England. The viscosity average molar mass ($\bar{M}_V$) determined at 20 °C was 91500 g mol^{-1} and the overlap concentration was $c^{\ast}$ = 0.126 %, *w*/*V*.[15] Sodium dodecylsulfate, SDS, purity >99 %, was obtained from Merck, Germany. The critical micelle concentration (*CMC*) determined at 20 °C by conductometric titration was 0.244 % *w*/*V*.[15] All samples were used without any further purification. Demineralized water was used as the solvent.

Preparation of the solutions

A stock solution of HPMC (2.6 mass %) was prepared by dispersing HPMC in water at 80–90 °C under gentle stirring. The stock solution was left for 24 h at room temperature before further use. Stock solutions of SDS (3.00 and 7.00 % *w*/*V*) were prepared by dissolving SDS in water at 20 °C.

The stock solution of HPMC, diluted stock solution of SDS and water were mixed together to obtain HPMC/SDS mixtures of the desired composition. The HPMC concentration was set to 0.37, 0.74 and 1.49 mass %, which was equivalent to three, six and twelve times higher concentration of HPMC than its overlap concentration, *i.e*. the overlap parameter $c/c^{\ast}$ was 3, 6, and 12. The SDS concentration was varied from 0.00 to 3.50 mass % for each of the three HPMC concentrations. The concentrations in the further text are expressed as mass %, unless otherwise noted.

Rheological measurements

Rheological measurements were performed using a RheoStress 600HP rheometer (Thermo HAAKE, Germany), at 20 °C. The cone and plate geometry was used (d = 60 mm, θ = 1 °). The steady-state method was employed to obtain viscosity curves in a shear rate range of 0.1–6000 s^{-1}, while the rheometer was operated in the controlled rate mode (CR mode).[20] The measurements were performed in triplicate and average values are reported. The measurement error was 2.5 %.

Shear-thinning regions of the experimental viscosity curves were fitted to the Ostwald de Waele Equation:

$$\eta = K\dot{\gamma}^{n-1} \text{ (Pa s)} \tag{1}$$

where: η is the viscosity, Pa s; $\dot{\gamma}$ the shear rate, s^{-1}; the K is the coefficient of consistency and n is the degree of shear-thinning. Fit quality was evaluated by the coefficient of correlation, which was always better than 0.995.

The rheometer was equipped with a sensor for measuring a force in a direction perpendicular to the shear flow, *i.e*., the normal force, F_n. The first normal stress difference, N_1 (*i.e*., the stress in a direction perpendicular to the shear flow), was calculated by the instrument software using the equation:

$$N_1 = \frac{2F_n}{\pi a^2} \text{ (Pa)} \tag{2}$$

where a is the cone radius.

RESULTS AND DISCUSSION

Low- to mid-shear rate range rheology of the HPMC/SDS mixtures

The influence of c_{SDS} on the rheological properties of the HPMC/SDS mixtures containing three different concentrations of HPMC was investigated. The HPMC concentrations in the mixtures were 0.37, 0.74 and 1.49 mass %, which corresponded to the overlap parameters, c/c^*, of 3, 6, and 12, respectively. The higher the c/c^* value was, the more entangled were the HPMC chains in the solution. The viscosity curves of the HPMC/SDS mixtures containing 0.00–0.35 mass % SDS and HPMC concentrations that correspond to the three values of the overlap parameter are shown in Fig. 1.

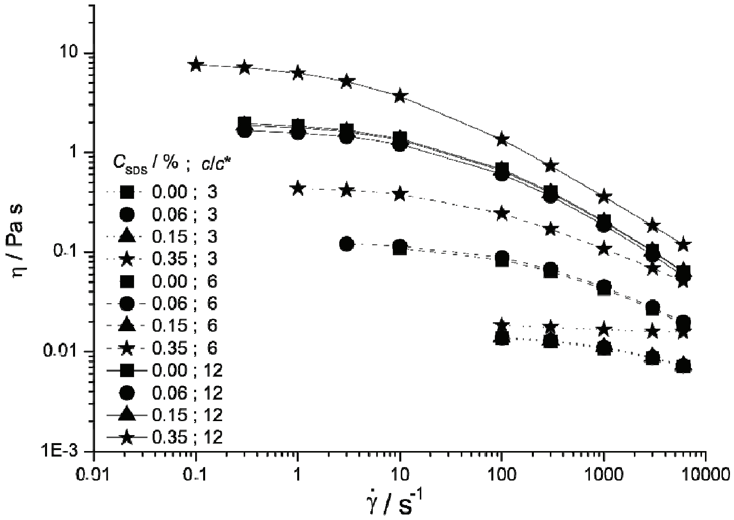

Fig. 1. Viscosity curves of HPMC/SDS mixtures containing 0.00–0.35 mass % SDS and HPMC concentrations that correspond to the overlap parameters of 3, 6 and 12.

The viscosity curves of the HPMC solutions containing no SDS showed typical shear-thinning behavior, *i.e.*, the viscosity of the solutions decreased with increasing shear rate. The decrease is due to disentanglement and orientation of HPMC macromolecules in the flow field.[18] The viscosity of the HPMC solutions increased as the number of HPMC chain entanglements increased, *i.e.*, as c/c^* increased from 3 to 6 and 12. In a like manner, the influence of the shear rate on viscosity was more pronounced and solutions become more shear-thinning as the value of c/c^* increased. This was confirmed by fitting the experimental data to the Ostwald de Waele Equation and determining the degree of shear-thinning, n, and the coefficient of consistency, K. The numerical values of the fitting parameters n and K are given in Table I. The influences of c_{SDS} on n and K are shown in Figs. 2 and 3, respectively.

TABLE I. The degree of shear-thinning, *n*, and the coefficient of consistency, *K*, for HPMC/SDS mixtures of different composition

c_{SDS} / mass %	c/c^* = 3		c/c^* = 6		c/c^* = 12	
	n	*K*	*n*	*K*	*n*	*K*
0.00	0.87	0.027	0.70	0.345	0.50	6.531
0.06	0.87	0.027	0.70	0.369	0.51	5.727
0.15	0.87	0.027	0.68	0.439	0.50	6.238
0.35	0.95	0.023	0.64	1.240	0.45	16.32
0.55	0.96	0.010	0.77	0.584	0.45	20.52
0.75	0.96	0.006	0.89	0.208	0.50	17.07
1.00	0.97	0.006	0.90	0.091	0.57	11.03
1.50	0.99	0.005	0.94	0.024	0.66	5.851
2.00	0.98	0.005	0.96	0.016	0.76	2.241
2.50	0.98	0.005	0.96	0.015	0.89	0.294
3.50	0.99	0.005	0.97	0.015	–	–

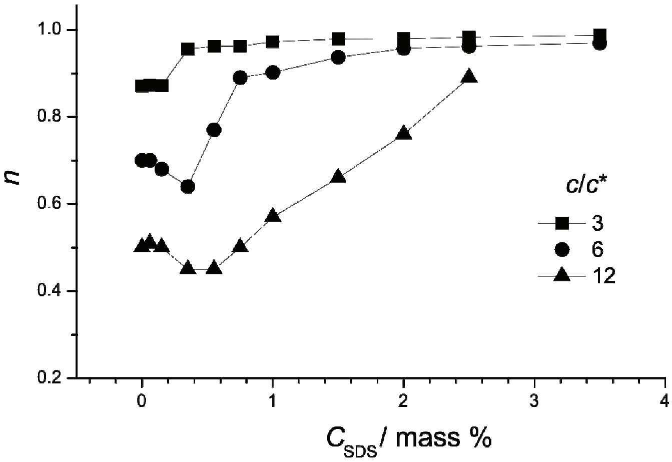

Fig. 2. The influence of the SDS concentration, c_{SDS}, on the degree of shear-thinning, *n*, of HPMC/SDS mixtures with the overlap parameter for HPMC of 3, 6 and 12.

As shown in Table I, the degree of shear-thinning decreased from 0.87 to 0.70 and 0.50, while coefficient of consistency increased from 0.027 to 0.345 and 6.531 as c/c^* was increased from 3 to 6 and 12, respectively. A decrease in *n* corresponds to a more pronounced shear-thinning flow (*i.e.*, more non-Newtonian flow), while an increase in *K* reflects an increase in the solution viscosity, and *vice versa*.[21] Such behavior is typical for polymer solutions with an increasing number of chain entanglements.[22]

The addition of SDS up to 0.15 mass % did not significantly influence the rheological properties of the HPMC solutions, as evidenced by the fact that viscosity curves of HPMC/SDS mixtures containing 0.00, 0.06 and 0.15 mass % SDS overlapped for a given value of c/c^*, Fig. 1. This indicates that there is no

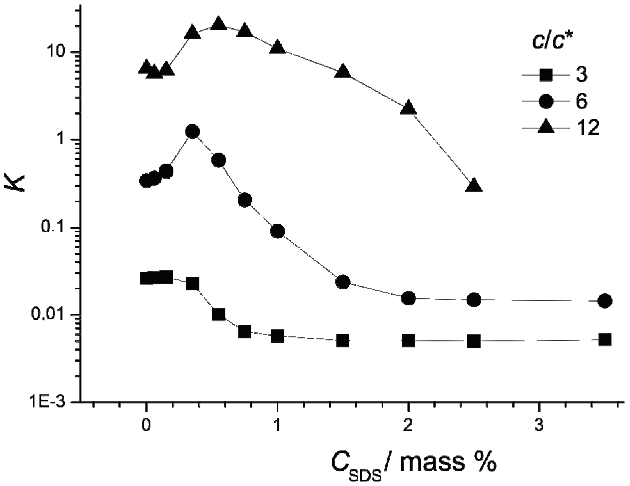

Fig. 3. The influence of the SDS concentration, c_{SDS}, on the coefficient of consistency, K, of HPMC/SDS mixtures with the overlap parameter for HPMC of 3, 6 and 12.

interaction between HPMC and SDS in the HPMC/SDS mixtures containing up to 0.15 mass % SDS. An increase in viscosity was observed when c_{SDS} exceeded 0.15 mass % for all the three HPMC/SDS mixtures (c/c^* of 3, 6, or 12), see the 0.35 mass % SDS viscosity curves in Fig. 1. The increase in viscosity was due to the onset of HPMC–SDS interactions and HPMC–SDS complex formation, which occurred when c_{SDS} was higher than the critical association concentration (*CAC*).[12,16] The complex formation occurred *via* hydrophobic interaction in which the SDS molecules bind to the HPMC chains. The SDS formed micelles around the hydrophobic moieties of the neighboring HPMC chains, which strengthens the network of entangled HPMC chains and resulted in an increase in viscosity.[13,17] The fact that the increase in viscosity occurred at c_{SDS} >0.15 mass % regardless of the c/c^* value showed that *CAC* value was independent of the HPMC concentration and was always 0.15 mass %. This is in line with previously reported results.[11,14] Increasing c_{SDS} further resulted in increase in the viscosity until a maximum in the viscosity is reached at a certain concentration (c_M). Maximum in the viscosity attained at c_M of 0.35, 0.35 and 0.55 % SDS for HPMC/SDS mixtures with c/c^* values of 3, 6, and 12, respectively. After reaching the maximum, the viscosity of the mixtures decreased on further increases in c_{SDS}, Figs. 1, 4 and 5.

The decrease in viscosity was due to more and more SDS anions becoming bound to the HPMC chains as c_{SDS} was increased. The binding of SDS caused an increase in the negative net charge on the HPMC chains and thus, electrostatic repulsion between neighboring HPMC chains occurred, which resulted in a gradual disentanglement of the chains, the network loosened and, consequently, the viscosity of the mixtures decreased.[12] Changes in the viscosity of HPMC/

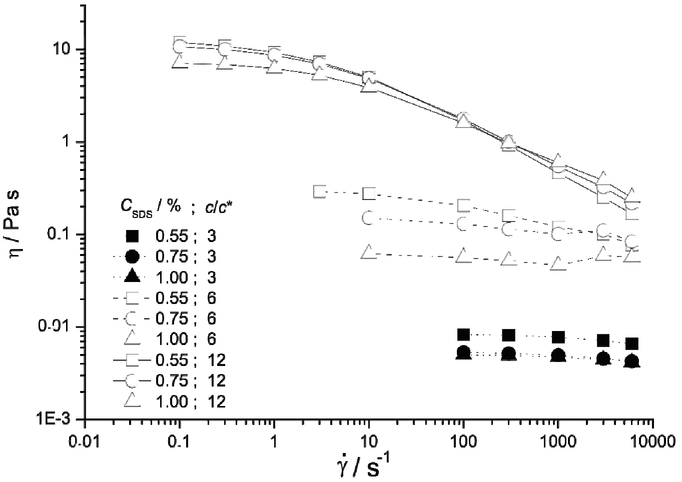

Fig. 4. Viscosity curves of HPMC/SDS mixtures containing 0.55–1.00 mass % SDS and HPMC concentrations that correspond to the overlap parameters of 3, 6 and 12. Viscosity curves of HPMC/SDS mixtures where a significant increase in the first normal stress difference (N_1) was observed are marked with open symbols.

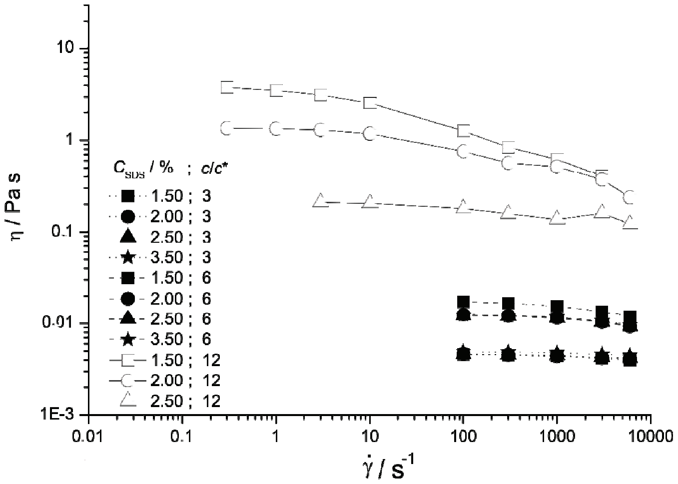

Fig. 5. Viscosity curves of HPMC/SDS mixtures containing 1.50–3.50 mass % SDS and HPMC concentrations that correspond to the overlap parameters of 3, 6 and 12. Viscosity curves of HPMC/SDS mixtures where a significant increase in the first normal stress difference (N_1) was observed are marked with open symbols.

/SDS mixtures no longer occurred when the c_{SDS} reached 0.75 and 2.00 mass % for the mixtures with c/c^* values of 3 and 6, respectively, Figs. 4 and 5. This indicates that the HPMC–SDS interaction was finished and that the c_{SDS} had reached the polymer saturation point (*PSP*). At the *PSP*, all the hydrophobic moieties of HPMC chains were fully solubilized with SDS micelles, the intermolecular links between neighboring HPMC chains were broken and the 3D network structure was lost.[15,17] Increasing the c_{SDS} above the polymer saturation point (*PSP*) brought about no significant changes in viscosity of the mixtures. The *PSP* increased with increasing c/c^{*} value (*i.e.*, increasing HPMC concentration) because more SDS is required to solubilize the greater amount of HPMC present in solution. The HPMC/SDS mixture with highest investigated HPMC concentration (*i.e.*, c_{HPMC} = 1.49 mass % and c/c^{*} = 12) would required the highest concentrations of SDS to fully solubilize the HPMC molecules and to reach the *PSP*. For this HPMC/SDS mixture, the *PSP* was not reached in the investigated range of c_{SDS} (0.00–2.50 mass %). In this case, the viscosity of the mixture changed on addition of SDS when $c_{\mathrm{SDS}} > CAC$, and it continued to change up to the highest investigated c_{SDS}, Fig. 5. The described changes in the viscosity of the HPMC/SDS mixtures nicely correlate with the changes in the coefficients of consistency, *K*, as a function of c_{SDS} in the mixtures, Fig. 3.

Apart from being influenced by the concentration of SDS and HPMC, the viscosities of the HPMC/SDS mixtures were also influenced by the applied shear. All of the examined HPMC/SDS mixtures were shear-thinning when examined at the low-end to mid-range of the applied shear rates, and their viscosities decreased on increasing the shear rate, Figs. 1, 4 and 5. The decrease in viscosity could be suitably described by the degree of shear-thinning *n*, Fig. 2. It could be seen, Fig. 2, that increasing c_{SDS} brings about characteristic changes in *n*, irrespective of the c/c^{*} value. For the lowest c_{SDS} value, below *CAC*, *n* does not change with increasing c_{SDS}, since there is no HPMC-SDS interaction. On further increase in c_{SDS}, the SDS binds to HPMC and supports a network of entangled HPMC chains, which results in more pronounced shear-thinning flow properties and thus a decrease in *n* occurred. The minimum value of *n* was attained at c_{M}, where the network was the stiffest. Further addition of SDS leads to an increase in *n* due to a gradual disentanglement of the HPMC chains because of the electrostatic repulsion. The network structure loosened and *n* increased until the PSP was reached. At the PSP, the network is completely broken and *n* reaches values close to 1, indicating almost perfect Newtonian flow and the absence of any structural rearrangements in the flow field.

The described changes in *n* with changing c_{SDS} were more pronounced in HPMC/SDS mixtures containing more entanglements of the HPMC chains, *i.e.*, in mixtures with a higher value of the c/c^{*} parameter. As c/c^{*} increases from 3 to 6 and 12, the difference between the lowest and the highest value of *n* determined for an HPMC/SDS mixture of a particular c/c^{*} increased, Fig. 2. Figure 2 shows rather small differences in flow profile over the whole range of c_{SDS} for HPMC/ /SDS mixtures with c/c^{*} = 3, and a flow profile ranging from highly shear-thinning to almost Newtonian for mixtures with c/c^{*} = 12. In addition, the changes in

n spanned over a broader range of c_{SDS} with increasing c/c^*, due to the fact that HPMC–SDS interaction occurred over a broader range of c_{SDS}.

Mid- to high-shear rate range rheology of HPMC/SDS mixtures

The HPMC–SDS interaction is determined by the molecular properties of HPMC and the composition of the HPMC/SDS mixtures. In addition, the shear rate can also influence the interaction, and this becomes especially true with increasing c/c^* parameter.

The HPMC/SDS mixtures with $c/c^* = 3$ were all shear-thinning throughout the employed shear rates (0.1–6000 s^{-1}), Figs. 1, 4 and 5. In addition, in these mixtures, no stresses perpendicular to the shear flow direction were detected (*i.e.*, $N_1 = 0$), irrespective of the composition of the HPMC/SDS mixture or the applied shear rate. However, this was not always the case in HPMC/SDS mixtures when c/c^* was increased from 3 to 6 and to 12. In the HPMC/SDS mixtures with $c/c^* = 6$ and 12 and c_{SDS} 0.55–1.00 and 0.55–2.50 mass %, respectively, a significant increase in N_1 was observed when a critical shear rate, $\dot{\gamma}_{crit}$, was exceeded, Figs. 6 and 7.

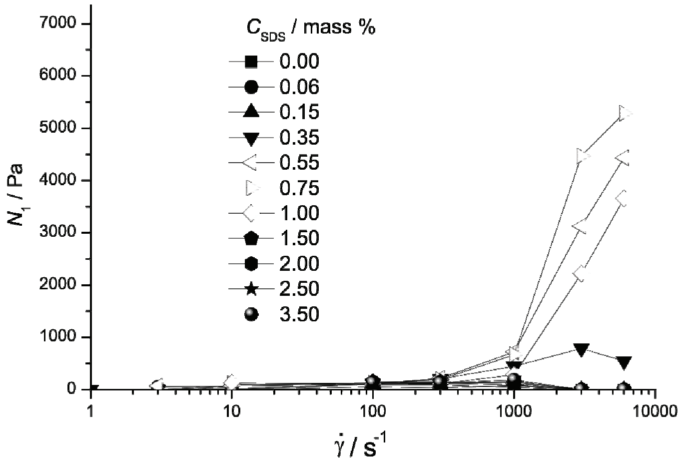

Fig. 6. Influence of the shear rate on the first normal stress difference (N_1) of HPMC/SDS mixtures with $c/c^* = 6$ and $c_{SDS} = 0.00$–3.50 mass %. HPMC/SDS mixtures where a significant increase in N_1 was observed are marked with open symbols.

A slight increase in N_1 was also observed in the $c/c^* = 6$ and 12 HPMC/SDS mixtures when $c_{SDS} = 0.35$ mass %. Below $\dot{\gamma}_{crit}$, N_1 was effectively zero. N_1 was also zero for the $c/c^* = 6$ and 12 HPMC/SDS mixtures when c_{SDS} was out of the above range for any of the employed shear rates (0.1–6000 s^{-1}). The observed N_1 values in the $c/c^* = 12$ mixtures were several times larger when compared to those for the $c/c^* = 6$ mixtures, Figs. 6 and 7. The development of a stress normal to the shear direction, N_1, is a typical manifestation of non-linear

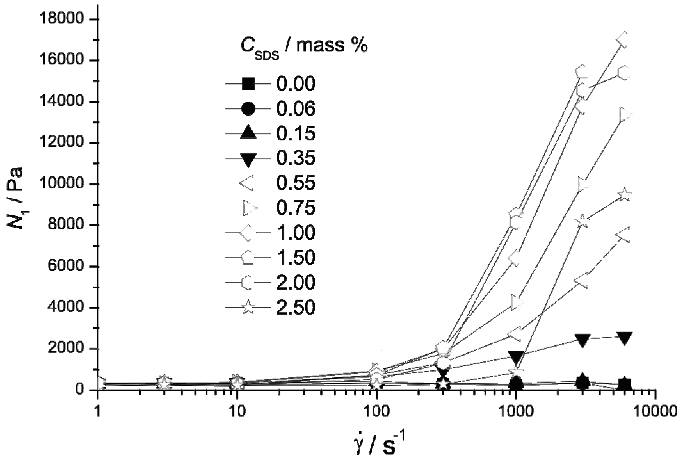

Fig. 7. Influence of shear rate on the first normal stress difference (N_1) of HPMC/SDS mixtures with $c/c^* = 12$ and c_{SDS} = 0.00–2.50 mass %. HPMC/SDS mixtures where a significant increase in N_1 was observed are marked with open symbols.

viscoelastic flow, and is indicative of an elastic response of a microstructure subjected to large-amplitude deformations.[22] In the HPMC/SDS mixtures in which an N_1 increase was observed, a change in flow profile of the viscosity curves also occurred when $\dot{\gamma}_{crit}$ was reached, Figs. 4 and 5 (viscosity curves marked with the open symbols). Namely, the decrease in viscosity with increasing shear rate became less steep than expected or even an increase in viscosity (*i.e.*, shear-thickening flow) occurred when $\dot{\gamma}_{crit}$ was reached. A break in a shear-thinning flow followed by a shear-thickening region in HPMC/SDS mixtures was previously reported for highly substituted HPMC macromolecules (degree of substitution for methoxyl group, DS_{Me}, and a molar substitution for hydroxylpropyl group MS_{HP},in ranges 2.2–3.0 and 0.48–1.88, respectively) of high molar mass, $\bar{M}_w$, between 2,200,000 and 3,800,000 g mol^{-1}, and was attributed to a shear-induced formation of HPMC–SDS complex.[19,24] The authors also reported on an increase in the first normal stress difference, and supported their findings on shear-induced structure formation with flow birefringence measurements. Similar results on shear-induced structure formation were also reported for mixtures of SDS and hydrophobically modified hydroxyl ethyl cellulose (hmHEC).[23] The results presented in this work (*i.e.*, an increase in N_1 and the change in the flow profile) thus suggest that a shear-induced structure formation may also occur in HPMC/SDS mixtures containing HPMC macromolecules of significantly lower molar mass ($\bar{M}_v$ = 91,500 g mol^{-1}) that contain less substituents (DS_{Me} = = 1.4 and MS_{HP} = 0.21) provided that the overlap parameter is sufficiently high.

CONCLUSIONS

Rheological investigation of HPMC/SDS mixtures containing different concentrations of SDS (c_{SDS}, 0.00–3.50 mass %) and HPMC concentrations, which corresponded to the overlap parameter, c/c^{*}, of 3, 6 and 12 were realized by viscosity measurements performed at different shear rates (0.1–6000 s^{-1}). All mixtures proved to be shear-thinning when examined in the low-end to mid-range of the applied shear rates. The degree of shear-thinning, n, and viscosity depended on the composition of the HPMC/SDS mixtures and were influenced by HPMC–SDS complex formation. Change in the c_{SDS} brings about characteristic changes in n, which were more pronounced as c/c^{*} was increased from 3 to 6 and to 12. A change in flow profile and a buildup of the first normal stress difference was observed in HPMC/SDS mixtures with c/c^{*} of 6 and 12 and c_{SDS} in ranges 0.55–1.00 and 0.55–2.50 mass %, respectively, when a critical shear rate, $\dot{\gamma}_{\mathrm{crit}}$, was exceeded, suggesting that a shear-induced structure formation in the mixtures occurred.

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ИЗВОД

РЕОЛОШКА СВОЈСТВА СМЕША ХИДРОКСИПРОПИЛМЕТИЛ-ЦЕЛУЛОЗЕ И НАТРИЈУМ-ДОДЕЦИЛСУЛФАТА

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Испитивана су реолошка својства смеша хидроксипропилметил-целулозе (HPMC), нејонског асоцијативног целулозног етра, и натријум додецилсулфата (SDS), анјонског сурфактанта, мерењем вискозитета при различитим брзинама смицања (0,1–6000 s^{-1}). Припремљене су HPMC/SDS смеше са различитим концентрацијама SDS (c_{SDS}, 0,00–3,50 мас. %) при чему је концентрација HPMC у смешама одговарала вредностима параметра преклапања c/c^{*} = 3, 6, и 12. Утврђено је да у опсегу нижих и средњих брзина смицања све HPMC/SDS смеше показују псеудопластичан тип протицања. Састав HPMC/SDS смеша и формирање HPMC/SDS комплекса утичу на вредност степена псеудопластичности, n. Вредности параметра n у смешама се крећу у опсегу вредности карактеристичних за изразито псеудопластичне флуиде до скоро потпуно Њутновских флуида, и више су изражене како се c/c^{*} повећава од 3 до 6 и 12. Код HPMC/SDS смеша са c/c^{*} вредношћу од 6 и 12 долази до промене у типу протицања као и до пораста прве разлике нормалних напона N_1 ($N_1 > 0$) уколико је c_{SDS} у опсезима 0,55–1,00 и 0,55–2,50 мас. %, редом, и уколико брзина смицања пређе критичну вредност, указујући на формирање смицањем индукованих структура у HPMC/SDS смешама.

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