Modeling of Floc Forming Suspensions Coupling the Population Balance Equation for Floc Aggregation-Breakage and the White-Metzner Model

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A viscoelastic model for floc-forming suspensions considering the aggregation and breakage of flocs during flows was developed based on our previous model, in which a population balance equation was adopted to simulate the floc aggregation-breakage. Similarly to the previous model, a Krieger-Dougherty model was used to describe the dependence of viscosity on the volume fraction of flocs, and a White-Metzner type model was used to represent the effect of viscoelasticity. In addition, the dependence of the relaxation time on the volume fraction of flocs was introduced to the present model using an elastic modulus function that depends on the effective volume fraction of flocs. The rheological behavior of the present model was examined by performing the simulation of startup flows of simple shear. The results of the simulation indicated that the temporal change in the distribution of floc size greatly depended on the elastic modulus function. The temporal behavior of the first normal stress difference therefore exhibits differences just after the startup of flow, depending on the elastic modulus function. The present model will be a simple viscoelastic constitutive model for floc-forming suspensions that represents the dependence of both viscosity and relaxation time on the floc size distribution.

Key Words: Fiber flocs / Population balance equation / Krieger-Dougherty model / White-Metzner model / Elastic modulus

1. INTRODUCTION

Cellulose nanofibers (CNFs) are bio-based novel materials and have been studied actively through various applications, such as nanocomposites, functional films, medical products, cosmetics, rheology modifiers, and food additives.1–6) In the manufacturing process of products using CNFs, flows of CNF suspensions often appear and their flow behavior affects the quality of products. Furthermore, the rheology of CNF suspensions is complicated because of the aggregation-breakage behavior of fiber flocs in flows, and hence the flow of CNF suspensions shows characteristic behavior. The flow of CNF suspensions is therefore an important issue from both engineering and rheological points of view.

Numerical simulation will be a strong tool for the analysis of CNF suspensions. However, few simulations have considered flow-induced changes in the microscopic structures of fiber flocs in CNF suspensions, although the characteristic rheological behavior of nanofiber suspensions is considered to be caused by structural changes in the microstructure of CNF suspensions in flows.7–14) Difficulty in coupling the macro flow computation of suspensions and the micro-simulation of flocs in flows is one reason for this. It is therefore necessary to develop a constitutive model for floc-forming suspensions, including CNF suspensions, that can describe the floc aggregation-breakage and is applicable to macro flow simulations.

In order to couple the simulation of flow-induced structures and the macro-flow analysis, Yearsley et al.15) developed a constitutive model for carbon black (CB) and carbon nanotube (CNT) suspensions, in which a Fokker-Planck equation for the orientation distribution of CBs or CNTs16–18) was coupled with a hybrid Maxwell-Voigt model. Using this model, they found that the rheology of CB and CBN suspensions was strongly affected by aggregate size. We developed a viscoelastic model for CNF suspensions19) based on a population balance equation (PBE) of fiber floc size proposed by Puisto et al.20) In this model, the dependence of viscosity of nanofiber suspensions on the volume fraction of nanofiber flocs is described using a Krieger-Dougherty model, and the viscosity function is combined with a White-Metzner type viscoelastic model. In the previous model, for simplicity, we did not consider the dependence of the relaxation time or the elastic modulus on the floc volume fraction. Many researchers, however, reported the floc volume fraction dependence of both the relaxation time and the elastic modulus for floc-forming suspensions.21–23) We therefore improved our previous constitutive model to include the volume fraction dependence of
the elastic modulus and examined the rheological properties predicted by the present model.

2. MODEL OF CELLULOSE NANOFIBER SUSPENSIONS

Here, we introduce a constitutive model for CNF suspensions based on PBEs. The model, which is the White-Metzner model, couples a PBE for fiber floc aggregation-breakage and a constitutive equation for viscoelastic fluids. In this model, the effect of the fractal dimension. The floc size should depend on the type of solvent similarly to polymers. In this model, the effect of the solvent on the floc size is expressed by the fractal dimension.

We applied a PBE-based model proposed by Puisto et al.\(^{(20)}\) to a model of suspensions of CNFs. In this model, the mass balance of fibers is described by the following equations:\(^{(20)}\)

\[
\frac{dn_i}{dt} = \frac{1}{2} \sum_{j=1}^{i} k^{(a)}(i-j,j)n_i n_j - \sum_{j=i+1}^{\infty} k^{(a)}(i,j)n_i n_j - k^{(b)}(i)n_i + \sum_{j=i+1}^{\infty} \beta(i,j) k^{(b)}(j)n_j,
\]

where \(i\) and \(j\) denote the numbers of monomers in different size classes of flocs, \(n_i\) is the number of flocs consisting of \(i\) monomers per unit volume, \(k^{(a)}\) and \(k^{(b)}\) are aggregation and fragmentation kernels, respectively, and \(\beta\) represents a fragmentation distribution function.

The aggregation and fragmentation kernels proposed by Vanni\(^{24}\) were used:

\[
k^{(a)}(i,j) = c_e \frac{4}{3} \gamma(r_i + r_j)^3,
\]

where \(c_e\) is a collision efficiency factor, which was set to 0.03,\(^{(25)}\) and \(\gamma\) is the shear rate, and

\[
k^{(b)}(i) = \gamma \exp \left( - \frac{2F_c}{5\pi \eta_i r_i^2 \eta \gamma} \right),
\]

where \(F_c\) is the aggregate effective internal bond force, and \(\eta\) is the viscosity of the suspension. Here, \(r_i\) indicates the radius of a floc consisting of \(i\) monomers defined by

\[
r_i = r_0 i^{1/d_f},
\]

where \(r_0\) is the length of a monomer, and \(d_f\) is the mass to radius fractal dimension. The floc size should depend on the type of solvent similarly to polymers. In this model, the effect of the solvent on the floc size is expressed by the fractal dimension.

We applied the discretization method proposed by Kumar and Ramkrishna\(^{(24,26)}\) and assumed binary fragmentation for \(\beta(i,j)\) to solve Eq. (1). The discretized equation was solved using the Euler forward differential method. In the present simulation, uniform breakage was assumed, and the function \(\beta\) was set as \(\beta = 2/(j-1).\)^{(24)} Furthermore, we assumed that the total number of monomers (\(\sum n_i\)) is conserved.

The number of monomers per unit volume can be estimated using the following relations:\(^{(19,20)}\)

\[
m_1 \approx \frac{2a_{\text{eff}}^3 \rho_s m_p 1}{3 \rho_l m_v v_1},
\]

where \(a_{\text{eff}}\) is the effective aspect ratio, \(\rho\) and \(m\) indicate the density and mass, respectively, and the indices \(s\) and \(p\) denote variables related to liquid and solid, respectively.

In the present model, the volume fraction dependence of viscosity is modeled using a Krieger-Dougherty model\(^{(27)}\) and that of the elastic modulus is described by a power-law model. Furthermore, these models are introduced in the White-Metzner model as a viscosity function and an elastic modulus function. As a result, the White-Metzner model is described by

\[
\tau + \frac{\eta(\varphi_{\text{eff}})}{G(\varphi_{\text{eff}})} \delta \tau = 2\eta(\varphi_{\text{eff}})D,
\]

where \(\tau\) is the extra stress tensor, and \(D\) is the rate-of-deformation tensor. The differential operator \(\delta/\delta t\) indicates the upper-convected derivative. Both the viscosity \(\eta\) and the elastic modulus \(G\) are represented by functions of the effective volume fraction \(\varphi_{\text{eff}}\), which can be calculated by\(^{(20)}\)

\[
\varphi_{\text{eff}} = \frac{\sum n_i v_i}{4} = \frac{4}{3} \pi r_0^3 \sum n_i r_i^3,
\]

where \(v_i\) is the volume of a floc consisting of \(i\) monomers.

Similarly to the previous model, the viscosity is evaluated using a Krieger-Dougherty model,\(^{(27)}\)

\[
\eta(\varphi_{\text{eff}}) = \eta_0 \left( 1 - \frac{\varphi_{\text{eff}}}{\varphi_m} \right)^{-k},
\]

where \(\eta_0\) is the solvent viscosity, \(\varphi_m\) is the maximum volume fraction, and \(k\) is a power index. Moreover, the elastic modulus function \(G(\varphi_{\text{eff}})\) is assumed to be described by a power-law model:

\[
G(\varphi_{\text{eff}}) = G_m \left( \frac{\varphi_{\text{eff}}}{\varphi_m} \right)^{m},
\]
where $G_m$ is an elastic constant, and $m$ is a power-index. Here, $G$ approaches $G_m$ and zero as the volume fraction ratio $\varphi_{\text{eff}}/\varphi_m$ approaches unity and zero, respectively. The power-law dependence of the elastic modulus on the volume fraction was reported for colloidal gels in which fractal flocs are formed and for CNF suspensions. The power-law model is therefore a reasonable choice as a first approximation of the dependence of the elastic modulus on the effective volume fraction. The relaxation time $\lambda$ is evaluated by

$$\lambda(\varphi_{\text{eff}}) = \frac{\eta(\varphi_{\text{eff}})}{G(\varphi_{\text{eff}})}.$$  

### 3. SIMPLE SHEAR FLOWS

We numerically simulated startup flows of simple shear at prescribed shear rates in order to evaluate the rheological properties of floc-forming suspensions. The equations of the White-Metzner model in simple shear flows at a shear rate of $\gamma$ are reduced to

$$\frac{\partial \tau_{xx}}{\partial t} = 2\tau_{xy} \dot{\gamma} - \frac{G}{\eta} \tau_{xx},$$  

$$\frac{\partial \tau_{xy}}{\partial t} = \frac{G}{\eta} (\eta \dot{\gamma} - \tau_{xy}),$$

where $x$ and $y$ indicate the flow and velocity-gradient directions, respectively, and $\tau_{xx}$ and $\tau_{xy}$ are the $xx$ and $yx$ components, respectively, of $\tau$. The viscosity $\eta$ and the elastic modulus $G$ were evaluated by Eqs. (8) and (9), respectively, using the effective volume fraction $\varphi_{\text{eff}}$ calculated from Eq. (7) based on the floc size distribution obtained by solving the PBE (1). We adopted the same values for the model parameters as those used in our previous study: $\rho_p=1.550$ kg/m$^3$, $\rho_s=1.000$ kg/m$^3$, $d_l=2.75$, $F_c=1.2$ nN, $a_{\text{eff}}=5.3$, $r_0=1.05$ $\mu$m, and $m_p/m_0=0.01$. Moreover, according to our previous simulation, the total number of monomers per unit volume was set to $2.5\times10^{16}$, the radius of the maximum floc was approximated as $1.000 r_0$, and the number of classes was set to 250. The radius $r_k$ of a floc in the $k$th class was decided as $r_{k+1} = \sqrt[N]{r_k}$, where the maximum floc size is defined as $N r_0$. Furthermore, we used the same values of the parameters of the viscosity function (8) as in the previous simulation: $\varphi_m=0.64$, $k=2.0$, and $\mu_0=1\times10^{-3}$ Pa-s, which were determined by referring to the experimental measurements of Bercaw and Navard.

We numerically simulated startup shear flows at constant shear rates. Fluid is at rest, and simple shear is imposed on the fluid at time $t=0$. The initial number density distribution of flocs was assumed to be uniform. Equations (1), (11), and (12) were numerically solved with a finite difference method. We performed the simulation by changing $G_m$ and $m$ while the values of other model parameters were fixed in order to investigate the effect of the volume-fraction dependency of the elastic modulus on the shear flow behavior of the present model. Referring the experimental results by Shih et al., we chose $m=4$ as a reference value and varied $m$ as 2, 4, and 6. Furthermore, we chose $G_m=0.05$ Pa as a reference to make the order of the relaxation time defined by $\lambda = \eta / G$ approximately $1$ s.

### 4. RESULTS AND DISCUSSION

#### 4.1 Steady shear properties

The relaxation time $\lambda$ is plotted in Figs.1 and 2 as a function of the relative volume fraction $\varphi_{\text{eff}}/\varphi_m$. The values of $G_m$ and $m$ are varied in Figs.1 and 2, respectively. As shown in Fig. 1, $\lambda$ increases with increasing $G_m$, under the numerical conditions of the present simulation, $\varphi_{\text{eff}}/\varphi_m$ varies approximately from 0.56 to 0.8, and $\lambda$ at $m=4$ changes slightly in this regime. The relaxation time rapidly increases as $\varphi_{\text{eff}}/\varphi_m$ approaches to zero, and this behavior seems to be unnatural. We therefore need to use the present model considering the range of application of the model. That is, we should be aware that this model assumes the nanofiber concentration that nanofibers form flocs and hence $\varphi_{\text{eff}}/\varphi_m$ is not small. The relaxation time is larger for larger $m$ in the entire region of $\varphi_{\text{eff}}/\varphi_m$, as shown in Fig. 2. Moreover, the relaxation time changes with $\varphi_{\text{eff}}/\varphi_m$ more remarkably for larger $m$ in the approximate range of from 0.56 to 0.8.

Figure 3 shows the relative viscosity $\eta/\eta_0$ as a function of shear rate $\dot{\gamma}$ for $m=4$. The steady shear viscosity is independent of $G_m$, as is known from Eq. (8). The viscosity exhibits a shear-thinning property, which agrees qualitatively with the

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**Fig. 1** Relaxation time $\lambda$ as a function of volume fraction ratio $\varphi_{\text{eff}}/\varphi_m$ for $m=4$ and $G_m=0.01$ Pa, 0.05 Pa, 0.1 Pa, and 0.5 Pa.
The results of our previous study indicated that the occurrence of shear-thinning in viscosity was relevant to the shear-thinning in the effective volume fraction $\phi_{eff}$ on $\gamma$, which is derived from the change in the floc-size distribution with $\gamma$.

Next, we analyze the elastic properties of the present model. Figure 4 plots the first normal stress difference $N_1 = \tau_{xx} - \tau_{yy}$ as a function of shear rate $\dot{\gamma}$ for $m=2$, 4, and 6. Here, $N_1$ increases with increasing $\dot{\gamma}$, which is a typical property of viscoelastic fluids. The dependence of $N_1$ on $m$ is shown in the figure. Here, $N_1$ is larger for larger $m$. Since the stress tensor is represented by a White-Metzner type model, the steady state values of $\tau_{xy} = \eta \dot{\gamma}$ are independent of $\lambda$. The first normal stress difference $N_1$ is described by $N_1 = 2\eta \lambda \dot{\gamma}^2$, where $\lambda$ is an increasing function of $m$, and hence $N_1$ is larger for larger $m$.

The dependence of the relaxation time $\lambda$ on the shear rate $\gamma$ for $m=2$, 4, and 5 is plotted in Fig. 5. The relaxation time decreases for larger $m$ because the elastic modulus $G$ at the same volume fraction increases with increasing $m$. For $m=2$, the relaxation time decreases monotonically with increasing $\dot{\gamma}$. For $m=4$ and 6, $\lambda$ decreases with increasing $\dot{\gamma}$ in the small-shear-rate regime and increases to approach constant values.

### 4.2 Transient shear properties

Next, we analyze the transient shear properties of the present model. Figure 6 shows temporal changes in the volume fraction ratio $\phi_{eff}/\phi_m$ at $\dot{\gamma}=1 \text{ s}^{-1}$ and $5 \text{ s}^{-1}$ for $m=2$, 4, and 6. The volume fraction quickly decreases to reach a steady state, and the rate of decrease is faster for smaller $m$. According to the temporal change in the effective volume fraction, both the viscosity and the elastic module change with time. Consequently, the relaxation time $\lambda$ also changes with time, as shown in Fig. 7. For every $m$, the relaxation time decreases from an initial large value to a steady state value with time. Undershoots are observed in the cases of $m=6$ at $\dot{\gamma}=1 \text{ s}^{-1}$ and $m=4$ and 6 at $\dot{\gamma}=5 \text{ s}^{-1}$. Based on the results of other simulations (not indicated here), undershoots in the relaxation-time response were confirmed to appear in cases of large $m$. The growth of the relaxation time tends to require a longer time to reach a steady state at larger shear rate.

Figure 8 shows the distributions of floc size at $\dot{\gamma}=0.5 \text{ s}^{-1}$, $1 \text{ s}^{-1}$, and $5 \text{ s}^{-1}$ at steady states. This figure plots the existing probabilities of flocs belonging to each class, $P$, scaled by that...
when the floc size is uniformly distributed, $P_0$. The floc size distribution is independent of $m$ at a steady state. As the shear rate increases, the peak position shifts to the small-floc-size side, which indicates that large flocs are broken by shear, and the distribution narrows. As reported in our previous paper, this tendency of the floc size distribution qualitatively agrees with numerical simulations by Puisto et al. and the shear thinning in viscosity originates from this behavior.

In Fig. 9, temporal changes in the floc size distribution at $\dot{\gamma}=5$ s$^{-1}$ for $m=2$, 4, and 6 are indicated. In the figure, the distributions at 0.5 s, 1 s, and 3 s after the startup of flow are plotted. For every condition of $m$, the peak position moves towards the small-floc-size side with time, and the temporal change in the floc size distribution takes a longer time for larger $m$. Although the floc size distribution is independent of $m$ at steady states, its temporal behavior highly depends on $m$. As shown in Fig. 8, the relaxation time $\lambda$ is larger, and a longer time is necessary to reach a steady state for larger $m$. These time responses are caused by temporal changes in the floc size distribution.

Figures 10, 11, and 12 show temporal changes in the shear stress $\tau_{yx}$ and the first normal stress difference $\tau_{xx} - \tau_{yy}$ for $\lambda=1$ s at $\dot{\gamma}=1$ s$^{-1}$ and 5 s$^{-1}$, respectively. In Figs. 11(b)
and 12(b), enlargements of the stress growth at early stages are indicated. In these figures, we plotted data for fixed-\(\lambda\) cases, which are discussed later.

Both \(\tau_{xy}\) and \(\tau_{xx} - \tau_{yy}\) develop over time to approach a constant value, and a longer time is necessary to reach a steady state for larger \(m\). These time responses are the results of the dependence of \(\lambda\) on \(m\) whereby \(\lambda\) is larger for larger \(m\). In other words, it takes a longer time for both \(\tau_{xy}\) and \(\tau_{xx} - \tau_{yy}\) to fully develop for a longer relaxation time. These results qualitatively agree with our previous predictions,\(^{19}\) in which \(\lambda\) was fixed. In addition, the growth of the stresses is delayed at a higher shear rate, especially for large \(m\). Furthermore, Figs. 11(b) and 12(b) show that the growth rate is faster for smaller \(m\) just after the startup of flow. This phenomenon is a result of the relaxation time being shorter for larger \(m\). The behavior of stress growth just after the startup of flow is discussed below.

Here, we compare these predictions for stress growth to those when the relaxation time is fixed to a steady state value. In Figs. 11(b) and 12(b), the growth of \(\tau_{xx} - \tau_{yy}\) for \(m=2, 4,\) and 6 is compared with the corresponding results for fixed values of \(\lambda\). We set \(\lambda=0.39\) s, 0.92 s, and 2.2 s for \(m=2, 4,\) and 6, respectively, at \(\dot{\gamma}=1\) s\(^{-1}\) and \(\lambda=0.35\) s, 0.97 s, and 2.7 s for \(m=2, 4,\) and 6, respectively, at \(\dot{\gamma}=5\) s\(^{-1}\). At both shear rates, the stress growth in fixed-\(\lambda\) cases is fast for \(m=2\) and 4 and slow for \(m=6\), as compared to the predictions of the present model. For \(m=2\) and 4, \(\lambda\) decreases monotonically from a large value to a steady state value with time and is larger than the corresponding fixed value before reaching a steady state value, as shown in Fig. 7. The response of stress growth is therefore delayed, as compared to fixed-\(\lambda\) cases. For \(m=6\), the response of \(\lambda\) exhibits an undershoot, as shown in Fig. 7, and \(\lambda\) is smaller than the fixed value until approximately \(t=5\) s. Consequently, the growth rate is fast compared to the corresponding fixed-\(\lambda\) cases. These phenomena are also observed for cases of \(\dot{\gamma}=5\) s\(^{-1}\). The difference in the stress response appears more remarkably at \(\dot{\gamma}=5\) s\(^{-1}\) because the relaxation time undershoots more greatly, needs a longer time to reach a steady state, and hence is below the fixed value for a long time period, as shown in Fig. 7(b).

As indicated in the above results, since the relaxation time depends on the effective volume fraction in this model, the differences between the present model and a model with a constant relaxation time are observed in temporal changes in the first normal stress difference.

Fig. 10 Temporal changes in the shear stress \(\tau_{xy}\) at \(\dot{\gamma}=1\) s\(^{-1}\) and \(\dot{\gamma}=5\) s\(^{-1}\) for \(m=2, 4,\) and 6.

Fig. 11 Temporal changes in the first normal stress difference \(\tau_{xx} - \tau_{yy}\) at \(\dot{\gamma}=1\) s\(^{-1}\) for \(m=2, 4,\) and 6. Enlargement is indicated in (b). Plots of triangles, diamonds, and circles indicate cases in which the values of \(\lambda\) are fixed to 0.39 s, 0.92 s, and 2.2 s, respectively.
5. CONCLUSION

We improved our previous model for CNF suspensions based on a PBE for floc aggregation and breakage to consider the effect of the volume fraction of flocs on the relaxation time. Similarly to the previous model, we used a Krieger-Dougherty model to describe the dependence of viscosity on the volume fraction of flocs and used a White-Metzner type model to represent the effect of viscoelasticity. Furthermore, the dependence of the relaxation time on the volume fraction of flocs was introduced to the present model using a power-law model for the elastic modulus.

The rheological behavior of the present model was examined by performing a simulation of startup flows of simple shear. The results of the simulation indicated that the distribution of floc size changed greatly, depending on the elastic modulus function. The relaxation time is long, and it takes more time to reach a steady state value when the power-index \( m \) of the elastic modulus function is large. The growth of the first normal stress difference is therefore delayed at large \( m \).

The time response of the first normal stress difference of the present model is different from that for a constant-relaxation case.

The computation of the PBE for the aggregation and breakage of flocs has a lower computational cost, as compared to the micro-simulation of the floc behavior. Consequently, the present model, in which the PBE is coupled with the White-Metzner model, is applicable for numerical simulations of complex flows, such as flows in channels. This model will be a simple viscoelastic constitutive model for floc-forming suspensions that represent the dependence of both the viscosity and relaxation time on the floc size distribution. Applications of the present model to the simulation of complicated flows are issues for future research.

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