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Fractal Nature of Magnetic Colloidal Dispersion with Cobalt Iron Oxide and Metal Iron Particles

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Abstract – The microstructure of highly aggregated colloidal dispersions was investigated by probing the rheological behavior of magnetic suspensions. The dynamic moduli as functions of frequency and strain amplitude are shown to closely resemble that of colloidal gels indicating the formation of network structure. The two types of characteristic critical strain amplitudes, γ_c and γ_y, were characterized in terms of the changing microstructure. The amplitude of γ_c indicates the transition from linear to nonlinear viscoelasticity and depends only on particle volume fraction not magnetic interactions. The study of scaling behavior suggests that it is related to the breakage of interfloc, i.e., floc-floc structure. However, yielding strain, γ_y, was found to be independent of particle volume fraction as well as magnetic interaction. It relates to extensive deformation resulting in yielding behavior. The scaling of elastic constant, G_e, implies that this yielding behavior and hence γ_y is due to the breakage of long-range interfloc interactions. Also, the deformation of flocs due to increase strain was indicated from the investigation of the fractal nature.

Key words: Colloidal dispersion, Rheological behavior, Magnetic suspension, Network structure, Floc

1. Introduction

Magnetic suspensions are colloidal dispersions of magnetic particles in a polymer solution and used in the production of flexible magnetic recording media as coatings for polymer films. The use of advanced metal particle (MP) pigments for high density digital storage application on flexible substrates has been expanded dramatically.

Concentrated suspensions form a complex microstructure; an association of particles to form an elastic network. A significant amount of research has been performed to study the rheological properties of gel-like suspensions formed by fractal aggregation with spherical particles [1-4], which result in non-Newtonian or viscoelastic behavior.

Since it possesses a highly aggregated structure, the magnetic suspensions are compared with other gel structures and/or other aggregated colloidal suspensions, but anisotropy of magnetic particle causes more complicate rheological behavior. Magnetic interactions between particles, which determine the dispersion stability can be strong and long-range. The particles agglomerate and flocculate which affects not only the recording performance of the final media product but also the application of coating [5] as shown in Fig. 1. A network structure is formed in magnetic dispersions without agitation. The network seriously affects the rheological properties since it strongly influences the overall microstructure of a magnetic dispersion. Even at relatively low concentrations, magnetic dispersion can possess a great shear rigidity and plateau elastic modulus. The elasticity results from networks between magnetic particles and/or aggregates with strong magnetic forces.

Kanai et al. [6] studied the elastic behavior of magnetic dispersion and found that interfloc structure is rigid at high volume fraction while it is flexible at low volume fraction. The yielding behavior of dispersion results from the network structure which is broken up above yield stress [7]. Potanin et al. [8] studied the effect of network structure on elastic properties for a model magnetic dispersion. They found that the structure of the magnetic suspension depends on milling time and polymer concentration, which adsorbs on magnetic particles.

The scaling behavior of network for colloidal suspension can explain the nature of structure [6,9-13]. Usually, the gel network is considered as closely packed flocs. Kanai et al. [6] showed that the structure could be explained by percolation exponent for a flocculated suspension. They found different exponent as the volume fraction increased indicating change of the structure. Shih et al. [11] proposed another scaling behavior of flocculated aggregation. They proposed the elasticity of dispersion was due to either flocs or interlink between flocs. The elasticity was proportional to the particle volume fraction with different exponent in the two cases.

In this paper, we studied the viscoelasticity of magnetic particle

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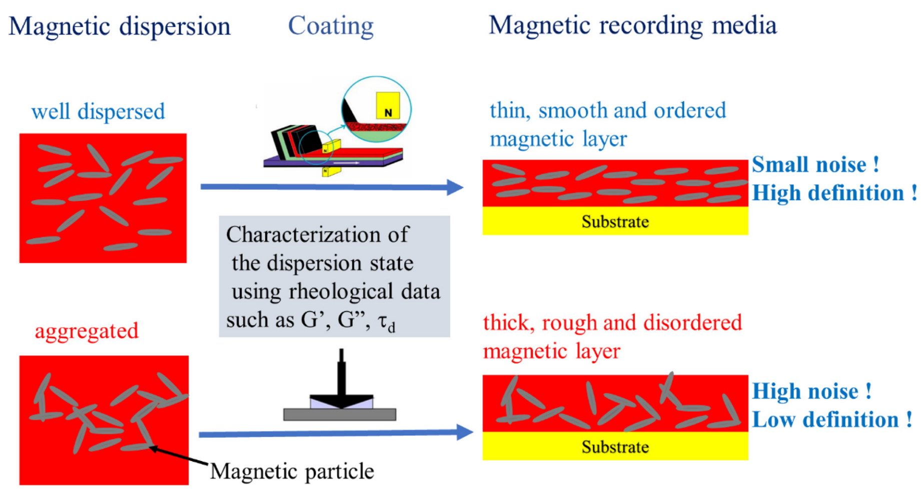

Fig. 1. The production of flexible magnetic recording media as coatings for polymer substrates and the role of the rheological characterization.

dispersions. The investigation was done in terms of volume fraction as well as interaction effect. Metal particles were used for the study on volume fraction while Co-γ-Fe_2O_3 particles were used for the study on interaction effect because of the convenience to control the interaction between particles. To explain the effect of structure on rheological behavior, the scaling behavior was also investigated.

2. Materials and Methods

The magnetic suspensions were prepared by dispersing magnetic particles in a polymer solution by milling. Polyvinylchloride copolymer (MR 110, Mn=12000, Nippon Zeon) containing 0.7 wt% SO_4 and 0.5 wt% OH functional groups was used as a wetting resin. It adsorbs on the surface of magnetic particles to provide steric stabilization. The solvent was cyclohexanone, chosen for its low volatility. Two types of magnetic particles were used: cobalt-modified γ-Fe_2O_3 (Co-γ-Fe_2O_3) and iron (Fe) particles (MP). Co-γ-Fe_2O_3 particles were provided from JVC Co. LTD and typically used in magnetic tape.

Fig. 2. Cryo-TEM image of Co-γ-Fe_2O_3 particle in the magnetic dispersion.

Fig. 2 shows a Cryo-TEM image of a magnetic dispersion made with Co-γ-Fe_2O_3 particles at 5 vol%. MP is metallic iron with a passive outer shell and used for high-density recording media and were provided by Quantage Co. LTD. MP has long axis-length of 150 nm with aspect ratio of 8. Co-γ-Fe_2O_3 has long axial length of 300 nm with aspect ratio of 6. The magnetization of MP is almost twice of that of Co-γ-Fe_2O_3. Co-γ-Fe_2O_3 particles are stable in the air and turn nonmagnetic, Co-α-Fe_2O_3, at very high temperature while MP is easy to burn on the contact with air. To study the effect of the interactions we used varied ratio of Co-γ-Fe_2O_3 and Co-α-Fe_2O_3 mixture. For the convenience, we refer to the mixtures as Co-γ-Fe_2O_3 suspensions. All the suspensions were simplified versions of commercial formulations. The weight ratio of polymer to metal particle was fixed at 0.175 for all MP suspension samples. All Co-γ-Fe_2O_3 samples contained 2.7 wt% polymer binder and 34.2 wt% particles with the balance being solvent, corresponding to a volume fraction of 0.098 particles. The suspensions ranged in concentration from all magnetic particles ($\phi_m/\phi = 1$) to all non-magnetic particles ($\phi_m/\phi = 0$). Here the descriptor is either volume fraction ϕ or fraction of magnetic particles ϕ_m/ϕ. The non-magnetic Co-α-Fe_2O_3 particles were prepared by heating Co-γ-Fe_2O_3 particles at 700 ^oC for 12 hrs. We confirmed that these particles had the same size and shape as the original particles with TEM (Transmission electron microscopy) measurements. Magnetic measurements indicated that less than 5% of the original magnetic moment remained in the Co-γ-Fe_2O_3 particles.

The rheological properties were measured using dynamic oscillatory shear and steady shear experiments. HAAKE RS100, which was stress controlled rheometer, was used to perform the experiments. For dynamic oscillatory shear, a sinusoidal stress $\tau = \tau_0 \sin(\omega t)$ was imposed on the sample. Dynamic moduli can be calculated from measured strain and shifted phase angle. All tests were done with 35 mm diameter 4^o cone and 35 mm diameter plate with 0.135 mm gap. The gap size was varied to check for slip. The temperature was fixed at 20 ^oC during the measurement.

3. Results and Discussion

Diluting the ink with solvent controlled the interactions between particles in the ink. A low volume fraction should increase the separation distance between particles and reduce the long-range magnetic attractive forces. However, the magnetic particles tend to form small primary aggregates even at low volume fractions. These primary aggregates are the basic units of the network structure.

The storage, G', and loss, G'', moduli as a function of frequency at $\tau = 1$ Pa which is within the linear viscoelastic region are shown in Fig. 3 for MP dispersions with different particle concentrations. These samples exhibit gel-like behavior; G' is only weakly dependent on frequency and is larger than G'' by an order of magnitude.

The loss modulus exhibits a minimum as a function of frequency. Mason et al. [14] observed such a minimum for concentrated, monodisperse emulsions stabilized by surfactant while the storage modulus shows plateau value. Potanin et al. [8] also found a minimum for magnetic particle suspensions. At low frequencies, energy dissipation occurs by network configurational rearrangement.

The rise of G'' at high frequencies reflects the increasing importance of the suspending medium viscosity. Energy dissipation occurs by hydrodynamic interactions between the individual particles and polymer solution [15]. At low frequency, the magnetic particles are trapped in the network by the magnetic interaction. Changes in the configuration of the network cause dissipation of energy resulting in an increase in the loss modulus at low frequency.

To study the effect of interactions between particles on the structure we examined Co-γ-Fe_2O_3 suspensions as we described above. The concentrations of magnetic particles to total amount of particles ranged from 0 to 100 vol% for the experiments. The frequency dependence of the storage and loss moduli for the Co-γ-Fe_2O_3 dispersions at $\tau = 1$ Pa is shown in Fig. 4. This measurement has been done at fixed total

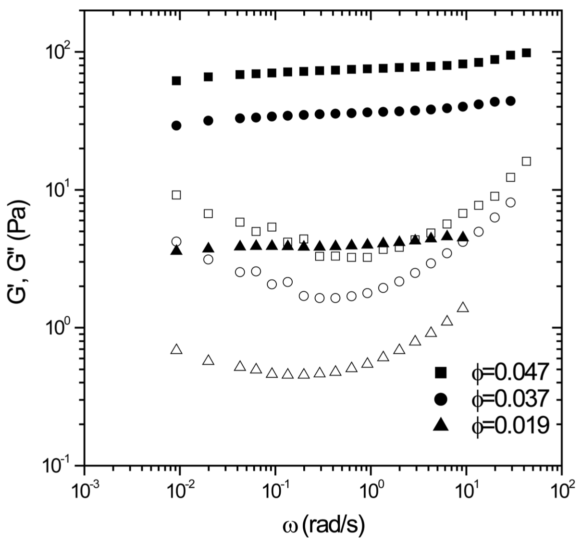

Fig. 3. Frequency dependence of storage modulus G' (filled) and loss modulus G'' (open) for the ϕ = 0.047 (square), 0.037 (circle), and 0.019 (triangle) MP suspensions.

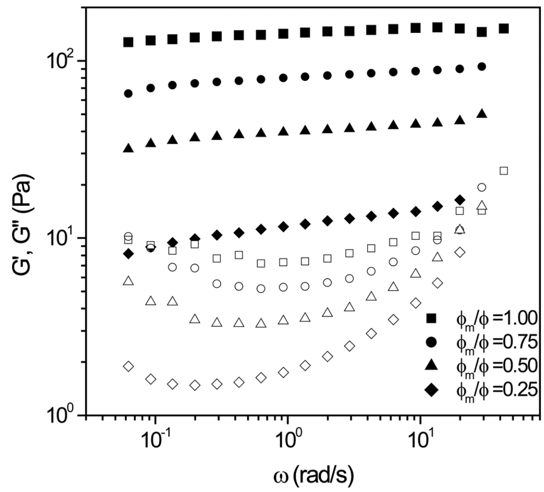

Fig. 4. Frequency dependence of G' (filled) and G'' (open) for the ϕ_m/ϕ = 1 (square), 0.75 (circle), 0.5 (triangle) and 0.25 (diamond) of Co-γ-Fe_2O_3 inks at fixed ϕ = 0.098.

particle volume fraction of 0.098 varying the fraction of magnetic particles as described above. G' decreases as the magnetic interaction between the particles decreases (*i.e.*, ϕ_m/ϕ decreases), showing, that the network structure is stronger at higher ϕ_m/ϕ as we can expect. G'' shows a minimum, just like MP inks.

In non-magnetic ink, $\phi_m/\phi = 0$, cross-over of the viscoelastic moduli is observed in Fig. 5, indicating there is no network structure. It appears that there exists a critical threshold, for formation of a network structure that depends on particle concentration as well as the interactions between particles. In same graph it is also shown that at the lowest particle volume fraction, $\phi = 0.007$, the MP dispersion exhibits rheological behavior intermediate between a viscous fluid and a gel. The moduli were measured at $\tau = 0.2$ Pa. The results show that there is no gel like structure at this volume fraction. Kanai et al. [6] reported that particles do not form a network structure below a critical volume fraction.

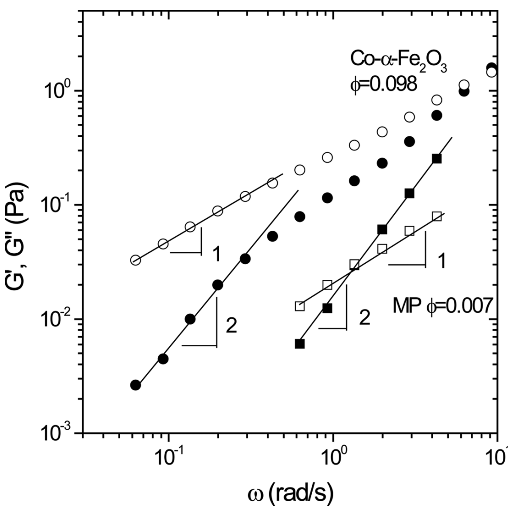

Fig. 5. G' (filled) and G'' (open) for ϕ_m/ϕ = 0 of Co-γ-Fe_2O_3 suspension (circle) at fixed ϕ = 0.098 and MP suspension (square) at ϕ = 0.007.

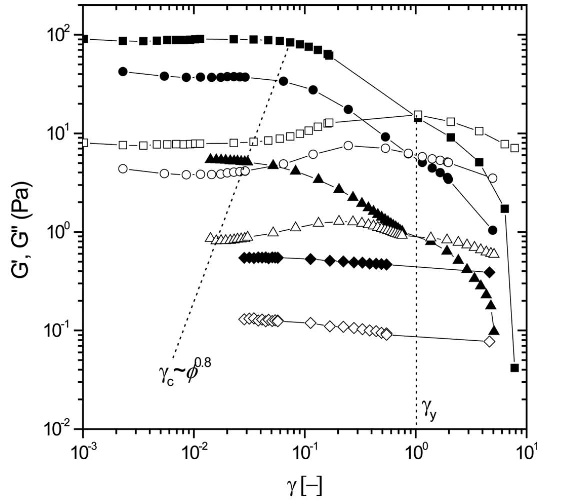

Fig. 6. Strain amplitude dependence of *G′* (filled) and *G″* (open) for ϕ = 0.047 (square), 0.037 (circle), 0.019 (triangle), and 0.007 (diamond) MP suspensions.

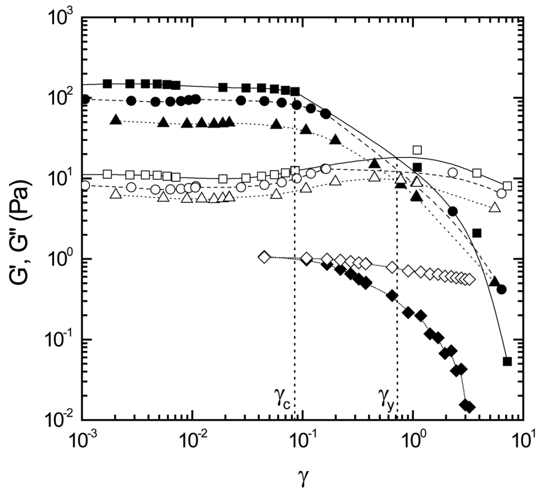

Fig. 7. Strain amplitude dependence of *G′* (filled) and *G″* (open) for ϕ_m/ϕ = 1 (square), 0.75 (circle), 0.5 (triangle) and 0 (diamond) of Co-γ-Fe_2O_3 suspensions at fixed ϕ = 0.098.

The dependence of the viscoelastic moduli on strain amplitude is shown in Fig. 6 for MP inks at a frequency of 6.28 rad/s. At strain amplitudes, γ, below the limit of linearity, γ_c, the storage modulus is much higher than the loss modulus indicating a gel structure. At large strains ($\gamma > \gamma_c$), the apparent G' decreases rapidly with increasing strain and the apparent G'' increases to a peak value before slowly decreasing. Note that in this nonlinear region the moduli are only apparent properties that reflect the peak stress-to-strain ratio and the phase lag.

The limit of linearity, γ_c, is a function of volume fraction indicating that micro-structural elements are destroyed at a smaller strain at lower concentrations. While the yield strain, γ_y at which the crossover between G' and G'' occurs, was found for all inks with $\phi > \phi_c$, marks the transition from solid-like behavior to liquid-like behavior [16]. This crossover occurs at almost the same strain amplitude of 1% ($\equiv \gamma_y$) showing that the topology of network structure is almost same and not depending on particle volume fraction.

The dependence of the viscoelastic moduli on strain amplitude is shown in Fig. 7 for Co-γ-Fe_2O_3 suspensions. This result shows the effect of interaction on the γ_c and γ_y at fixed particle volume fraction. In contrast of the volume fraction effect, both γ_c and γ_y are independent of ϕ_m/ϕ indicating insensitivity to interactions between particles. From Figs. 6 and 7, it is shown that γ_y is independent on volume fraction and magnetic interactions (ϕ_m/ϕ) while γ_c depends only on particle volume fraction not on magnetic interactions. It tells us that the nature of the structures formed by different particle volume fractions differ from those formed by different particle interactions.

The weakest link which is broken at γ_c, is not dependent on particle interactions but volume fraction which determines the size of network [11]. We propose that the unit of network structure is the floc which is formed by aggregated particles. Hence γ_c is determined by size of floc not the interaction between particles. The γ_y is independent on particle interaction as well as volume fraction. As we show below, γ_y is related to yielding behavior.

The extensive breakage of bonds should be independent of volume fraction [6]. The strain corresponding the yield stress is γ_y in Figs. 6 and 7. The yield stress (τ_y) obtained from γ_y ($\tau_y = G\gamma_y$) is shown in Table 1. A fit of a steady shear experiment to the Casson equation is shown in Fig. 8 and can be extrapolated to find the dynamic yield stress, τ_d.

$$\tau^{1/2} = \tau_d^{1/2} + \eta_\infty^{1/2}\dot{\gamma}^{1/2} \qquad (1)$$

The Casson yield value is additional important evidence of a critical volume fraction. The yield value is very small at a low volume faction and rises at the volume fraction of 0.019, indicating that the network formation begins to be noticeable at around this point.

The shear yield stress, τ_y was compared to the dynamic yield stress, τ_d in Table 1. The magnitude for both is very close implying that γ_y is related to dynamic yield stress and hence broken of a structure: the extensive breakage of bonds as we described above.

Shih et al. [18] showed that the limit of linearity involves the first few bonds breakages between flocs and hence depends on particle volume fraction. Since the γ_c represents the limit of linearity, the structure which is weakest bond is broken at this point and has different nature with one at γ_y. The characteristics of those structures were studied by investigating scaling behavior of the suspensions. The extensive deformation to break network structure resulting in yielding behavior is independent on particle volume fraction as well as magnetic interactions between particles. However, the dependency of amplitude of storage modulus at γ_y on volume fraction is much

Table 1. Comparison of the yield stresses obtained from dynamic and steady shear experiments for MP suspensions

Particle volume fraction	τ_y (Pa)	τ_d (Casson fitting) (Pa)
0.0470	19.0	20.00
0.0366	8.3	6.09
0.0190	1.2	0.90

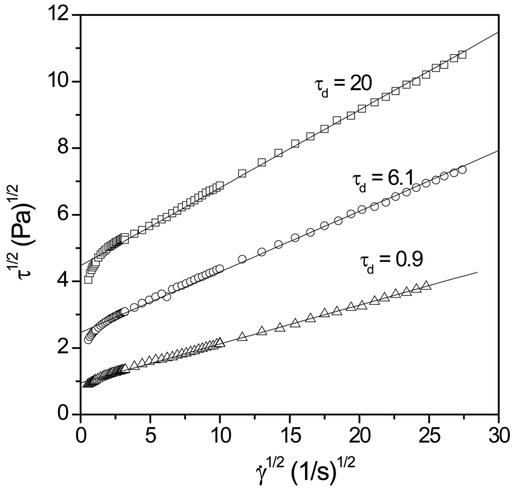

Fig. 8. Casson fitting from steady shear data for the ϕ = 0.047 (square), 0.037 (circle), and 0.019 (triangle) for MP suspensions.

stronger than on magnetic interactions. It implies that the yielding behavior occurs on interfloc structures rather than intrafloc (the microstructure between particles) interactions.

Percolation theory predicts that an unlimited network is built up above the critical concentration. Above this critical concentration, properties of the suspension such as the elastic modulus G or the critical strain amplitude γ_c follow a scaling law of the type $G \sim (P_p - P_c)^f$, where P_p is a descriptor of the particulate suspension. P_c is the threshold value and f is the fractal dimension of network structure. For our system P_p is corresponding to ϕ and/or ϕ_m/ϕ.

When $\phi >> \phi_c$ the scaling can be approximated as the plateau modulus $G_e \sim \phi^f$. The plateau modulus, G_e, as functions of ϕ and ϕ_m/ϕ are plotted in Fig. 9(a) and Fig. 9(b) respectively. The plateau moduli were obtained from Fig. 6 and Fig. 7 for MP and Co-γ-Fe_2O_3 suspensions, respectively. An exponent f = 3.4 was found by fitting the MP data as shown in Fig. 7(a). Theoretical studies of aggregates describe their internal structure in terms of interconnected fractal clusters [6,11]. Using a diffusion-limited, cluster-cluster aggregation model, Buscall et al. [2] predicted $G' \sim \phi^n$, with $n = 4 \pm 0.5$. This is very close to the value n = 3.5 obtained by Chen et al. [19] for flocculated silica dispersions. Although the particle concentration of our samples is relatively low, the value of n = 3.4 obtained from Fig. 9(a) indicates these particles tend to be highly aggregated.

Figure 9(b) shows that G_e increases monotonically with ϕ_m/ϕ in the Co-γ-Fe_2O_3 suspensions, so that $G_e \sim \phi_m/\phi$. G_e increases linearly with interactions between particles and exponentially with the particle volume fraction.

de Gennes [20] showed scaling concepts which can be successfully sued for polymer gels. Since the colloidal gels are very similar to polymer gels in that both are viscoelastic and have similar aggregation structure, Shih et al. [11] developed this concept to apply to flocculated aggregation. They considered the elastic constant of a dispersion to result from either link between or within flocs. The elastic constant was proportional to the particle volume fraction with different exponents in the two cases. Since the viscoelastic properties of magnetic ink show gel like behavior and the micro-structure of magnetic dispersion is formed by particle aggregation, we used Shih's scaling concepts for magnetic inks.

The scaling behavior of γ_c and plateau modulus, G_e, for colloidal gels depend on the strength of interactions between the flocs compared to within the flocs. Interfloc is the bond between flocs while intrafloc describes internal bond in a floc. The "weak link" regime is defined as the rigidity of intrafloc links being much larger than that of interfloc links, $G_{intra} >> G_{inter}$ where subscriber intra means intrafloc and inter means interfloc respectively. This is similar to a dispersion of spheres and the properties scale according to

$$\gamma_c \propto \varphi^{1/(3-d_f)} \quad (2)$$

$$G_e \propto \varphi^{1/(3-d_f)} \quad (3)$$

where d_f is the fractal dimension of the flocs. In the weak-link regime, the weakest interfloc bond is broken at γ_c and G_e is dominated by the behavior of G_{inter} since the flocs are now more rigid.

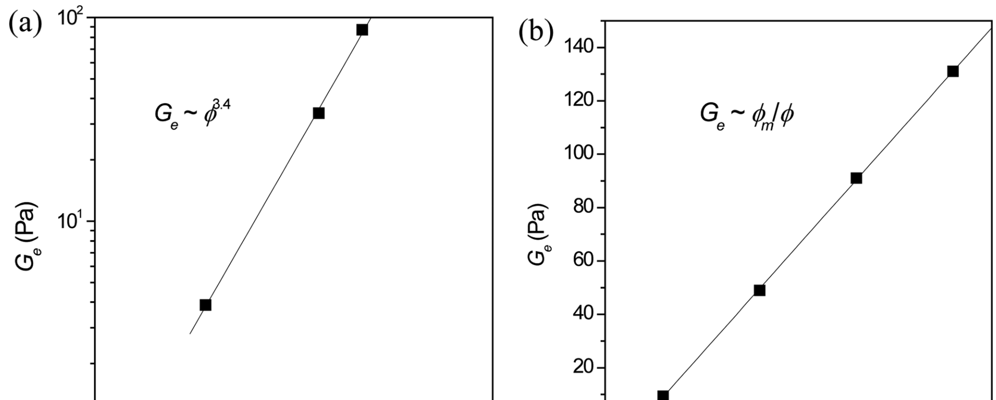

Fig. 9. The plateau modulus obtained from strain sweep as function of volume fraction for (a) MP suspension and as function of ϕ_m/ϕ for (b) Co-γ-Fe_2O_3 suspension.

The “strong link” regime is defined as the rigidity of interfloc links being much larger than that of intrafloc links, $G_{inter} >> G_{intra}$. In this case, the links between flocs are completely rigid. The bonds within a floc are broken at γ_c and G_e is dominated by G_{intra}. The properties scale according to

$$\gamma_c \propto \varphi^{-(1+x)/(3-d_f)} \tag{4}$$

$$G_e \propto \varphi^{(3+x)/(3-d_f)} \tag{5}$$

where x is the backbone fractal dimension. It should have a value between unity and the fractal dimension of the floc.

In the weak-link regime, γ_c increases with particle concentration while it decreases in the strong-link regime. As shown in Fig. 6, γ_c increases as particle volume fraction increases. The dependence is positive, identifying the behavior as conforming to the weak link regime. We found that the exponent of ϕ in Eq. (3) is 0.80 and the fractal dimension of the flocs is d_f=1.74.

G_e should depend on ϕ by the same exponent according to Eq. (4). However, Fig. 9(a) shows that the dependence is much stronger with an exponent of 3.4. This implies that G_e depends on ϕ according to the strong-link regime. Assuming that the same fractal dimension can be used, the backbone fractal dimension is calculated to be $x = 1.3$, which falls between the expected limits. The floc fractal dimension of 1.74 is an indication of highly aggregated structures implying diffusion-limited aggregation, wherein the probability of contact of diffusing particles to one another is very high. In contrast, this probability is very low in reaction-limited aggregation processes which occur more frequently at very low volume fraction.

According to the model of Shih et al. [18], our results show that the γ_c is a function of interfloc links and the plateau modulus, G_e, is from the floc itself. Fig. 10 shows a chain of elastic flocs in which the link between flocs can also be elastic. The “weak link” regime is defined as the rigidity of intrafloc links being much larger than that of interfloc links, $G_{intra} >> G_{inter}$. This indicates that the interfloc links are weaker but more rigid and brittle than the intrafloc links. Since the weakest bonds are between flocs, the limit of linearity, γ_c, corresponds to disruption of the interfloc links, not the breakage of flocs themselves. One possible explanation of this discrepancy between the model and experimental result is that G_e is dominated by soft floc structure. The flocs have stronger internal bonds and behave like soft spheres (*i.e.*, deformable). The shape of the data in Figs. 3 and 4 is similar to that of soft glassy materials [21,22]. The soft glassy materials such as foams, dense emulsions, and pastes show qualitative similarities to a glass transition with the possible deformation and flow.

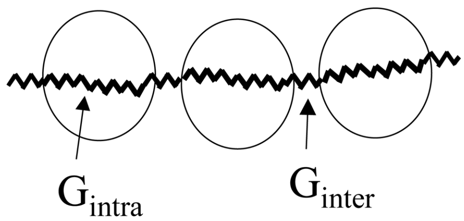

Fig. 10. Illustration of sources of G_{intra} and G_{inter} in terms of floc-floc model.

From our results, γ_c is dependent on the particle volume fraction not on the particle interactions. In other words, the strength of interfloc link strongly depends on particle volume fraction and hence the size of floc as $\xi \sim \phi^{1/(d_f-3)}$ where x is floc size. We showed that G_e increases exponentially with particle volume fraction and linearly with particle interactions in Fig. 9. This is consistent with the relationship

$$G_e \sim \frac{K_0}{\xi^{2+x}} \tag{6}$$

where K_0 is a bending constant between two particle bonds which depends on particle interaction and x is a backbone fractal dimension as we described above. The scaling from Eq. (6) is a little less than experimental result, i.e., 2.6 vs 3.4. The result shows that plateau modulus is dominated by volume fraction rather than by magnetic interactions between particles. It implies that the effect of interfloc structure is much bigger than intrafloc structure.

While linearity in dynamic viscoelastic property is broken above γ_c, the shear yield stress is found at γ_y. Since γ_y is independent on volume fraction as well as particle interactions it is rather related to extensive deformation of the structure to be broken. Even though the breakage of the weakest bond in interfloc occurs at γ_c, there are still significant long-range magnetic interactions holding those floc structures. This long-range interaction is not significant at γ_y and the floc structures begin to be broken.

4. Conclusions

The magnetic suspensions have a network structure formed by the magnetic particles has been investigated. By examining volume fraction effect and particle interaction effect, we showed that our suspensions exhibit two mechanisms for network breaking. First, interfloc bond is broken at γ_c. This mechanism is dependent on volume fraction not on particle interactions. However, this breakage does not result yielding behavior. Since the magnetic interaction is strong the interactions between flocs remain significant after the breakage of interfloc. This interfloc interaction decreases until it is minimal at higher strain γ_y. The scaling of γ_c shows that it is a consequence of breakage of interfloc links. In contrast the scaling model we compared, plateau modulus, G_e, comes from different structure so called intrafloc links. We explained this discrepancy by proposing that the flocs are deformable and formed by strongly aggregated particles while the interfloc link is relatively weak and brittle.

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