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Behavior of carbon cone particle dispersions in electric and magnetic fields

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ABSTRACT

The behavior of carbon cone (CC) particle dispersions in electric and magnetic fields is presented. The behavior of CC dispersions in an ac electric field was studied by the use of electrorheological (ER) measurements. Low ac electric fields were sufficient for fibrilar structures to form. However the structures formed were relatively weak as determined by ER measurements and this was attributed to the high conductivity of the particles. This is also consistent with the low relaxation frequency found by impedance spectroscopy. The behavior of a dispersion of CC added ferrofluid was studied using magnetorheological measurements. The relative increase in viscosity was found to be around 1.2–1.5 for low magnetic fields and low shear rates. This was attributed to purely hydrodynamic effects caused by CC particle–particle interactions.

1. Introduction

In some particle suspensions subjected to an electric or magnetic field, the particles form fibrilar structures which may change the properties of the suspension from liquid-like to solid-like. These kinds of suspensions are called electrorheological (ER) and magnetorheological (MR) fluids and they are used in a wide range of applications [1,2]. By measuring the rheological properties of these fluids the strength or rigidity of the fibrilar structures may be studied. The solid phase of such suspensions may be composed of various materials such as silicate ceramics, conductive organics and polymers. Numerous articles also report the use of carbonaceous particles of various kinds [3–8]. Some of these suspensions function as ER fluids. In other cases it is the resulting fibrilar structures that are interesting as they may be used for conductive paths in composites [4,7]. In this work a unique material called carbon cone material is used as the solid phase of various suspensions. Little experimental work has been done regarding this material, hence the basic physical properties are mostly unknown. The material is produced in an industrial scale process named Kværner Carbon Black & Hydrogen process [9]. Particles with conical and disk shape result from the process. The large scale process greatly reduces the material cost making the investigation of this materials interesting for possible future applications. Compared to carbon black, also produced in industrial scale processes, the carbon cone material contain particles with semicrystalline structure. In addition the conical particles represent perfect structures with a closed tip, creating unique electronic properties in the tip area [10]. At the present, pure samples representing only one geometry does not exist and finding an efficient purification process has proven difficult. Hence this study investigates the behavior of the mixed carbon particles in a dispersion in an electric and magnetic field. The influence of the particle concentration and the electric/magnetic field on the apparent viscosity is investigated. The ER effect is discussed with regard to the particle conductivity and the relaxation frequency of the dispersion. Possible applications are also discussed.

2. Experimental

2.1. Materials

The carbon cone (CC) particles provided by n-Tec AS [11], were used as the dispersant phase in both ER and MR fluids. The particles were used as produced. The particles have various geometries consisting of approximately 20% cones, 70% disks and 10% amorphous carbon. The size distribution typically varies from a few hundred nanometers for the amorphous carbon to about 5 μm for the largest disks, with most of the particles having dimensions of 1–3 μm, as determined by SEM analysis, Fig. 1.

For electrorheological studies the dispersive phase used was polydimethylsiloxane (silicon oil) (Dow Corning, density $\rho =$

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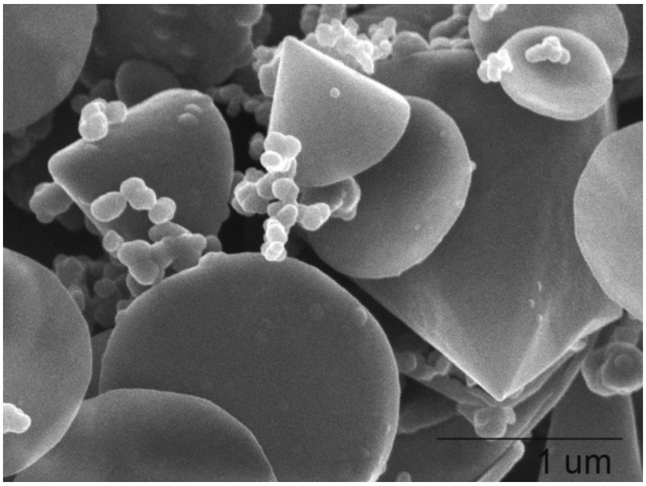

Fig. 1. The dispersant phase consisting of carbon cones, disks and amorphous carbon particles.

0.976 g/ml, viscosity $\eta = 100$ mPa s at 25 °C, dc conductivity $\Sigma_s \sim 10^{-12}$ S/m [12], permittivity $\varepsilon_s = 2.8$). The ER samples studied were prepared by mixing the carbon particles with the silicon oil. Four samples were prepared with concentrations 0.2, 0.5, 1.1 and 2.5 wt%. After mixing of the particles with the fluid, using ultrasonication (20 min at 30 W), the CC particles were well dispersed in the silicon oil. The dispersions were stable for days depending on the volume fraction.

For producing a magnetorheological fluid we used ferrofluid (FF) EMG901 from FerroTec with bulk viscosity 10 mPa s at 27 °C, density 1.53 g/cm^3 at 25 °C, and saturation magnetization 60 mT [13]. The carrier liquid of this FF is a synthetic isoparaffinic solvent with an anionic surfactant (oleic acid). The magnetite particles (Fe_3O_4) are predominantly spherical with diameter 10 nm with log-normal distribution. The MR sample was prepared mixing 1.5 wt% CC with the ferrofluid using vigorous shaking and then sonicating for 1 h. The dispersion was stable for a couple of hours.

2.2. Electrorheological measurements

ER properties of the samples were measured at 25 ° C using a rotation type rheometer (Physica MCR 300) with an ER cell with concentric cylinder geometry. The gap between the cylinder and the cup was 1.13 mm. A high voltage ac generator with a fixed frequency of 50 Hz was used to create the electric field perpendicular to the flow direction. The fields used were 100, 200 and 300 V/mm. The voltage was controlled manually, which introduces an uncertainty of approximately ±10 V/mm in the electric field. Due to friction from the electrodes on the rotating cylinder, all data were corrected accordingly by subtracting the values recorded for the empty cell.

All samples where dehydrated at 100 ° C for 20 h. Prior to each ER measurements all samples where sonicated for 20 min. A pre-shear experiment was carried out to measure the rheological properties at zero field. Then ac electric field was applied for 3 min, to allow structure buildup, before the next measurement. Flow curves were obtained with the shear rate starting at 1×10^{-3} Hz and increasing in a ramp logarithmic manner to 1×10^{3} Hz. As previous work showed that chains formed by carbon cone particles do not dissolve after the electric field is turned off [8], the samples were sonicated between each measurement to assure similar conditions for all measurements.

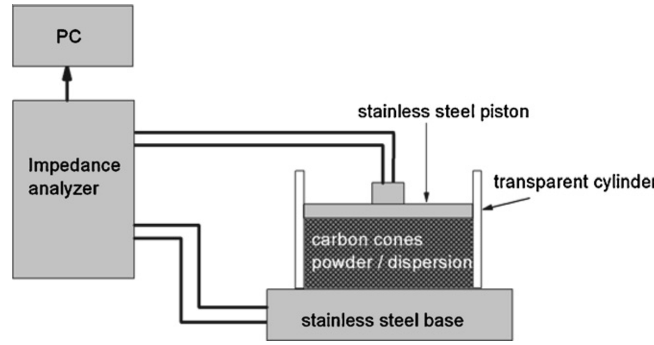

Fig. 2. The experimental set-up for impedance measurements, using a cell with two-electrode configuration.

2.3. Magnetorheological measurement

For magnetorheological measurements the same rheometer as described in Section 2.3 was used with a MR cell using a parallel plate geometry. This setup gives average higher radial shear than a cone/plate geometry. The experiments were run at a constant temperature of 20 ° C and with 0.20 mm between the plates. The shear rates used were between 20 and 1×10^3 Hz. The magnetic fields varied between B = 0–1085 mT.

2.4. Conductivity measurements

To measure the dc electrical conductivity of the dry carbon cone powder a two-electrode configuration was used. The measurement cell (shown in Fig. 2) consisted of a cylindrical tube placed on a stainless steel support plate which served as one of the electrodes. A stainless steel piston with diameter equal to the inner diameter of the cylinder was used as the second electrode ($A = 28.3$ cm^2). Carbon cone powder was poured into the cylinder, the cylindrical electrode was placed carefully on top and the height of the resulting powder column, typically ~ 2 cm, was measured manually. This was repeated for pressures varying from 0.87 to 9.98 kPa using additional weights. A dc voltage between 0.5 and 3.0 V was applied to the circuit and the resulting current through the circuit was recorded by a multimeter (HP 3478A) connected to a PC via a GPIB connection. For as-produced carbon cone particles the dc conductivity was found to be amplitude dependent as seen in Fig. 3. This might be due to a contact potential between the carbon particles. As expected

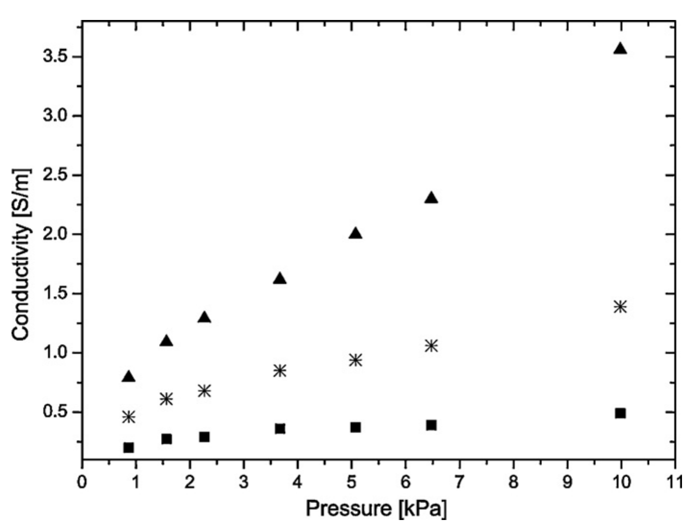

Fig. 3. DC conductivity of dry CC powder at various voltages as a function of packing pressure. Amplitudes used were; 1 V (square) 2 V (star) and 3 V (triangle).

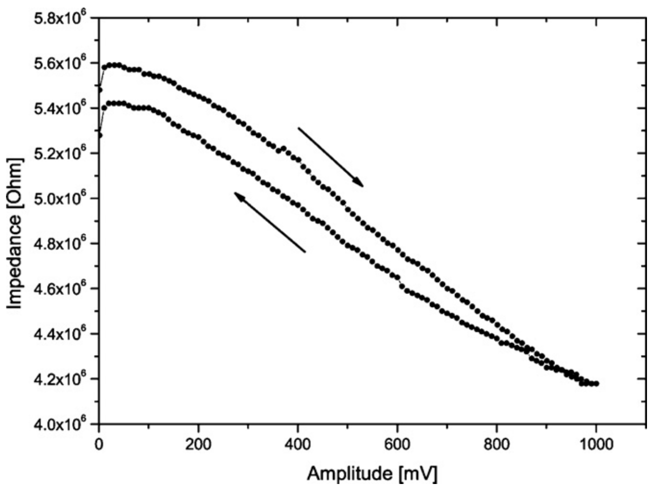

Fig. 4. Impedance vs amplitude for a CC dispersion of 1.1 wt% in silicon oil measured at 10 Hz. Hysteresis is observed resulting in lower impedance when returning from high to low amplitudes. The distance between the electrodes was 4 mm.

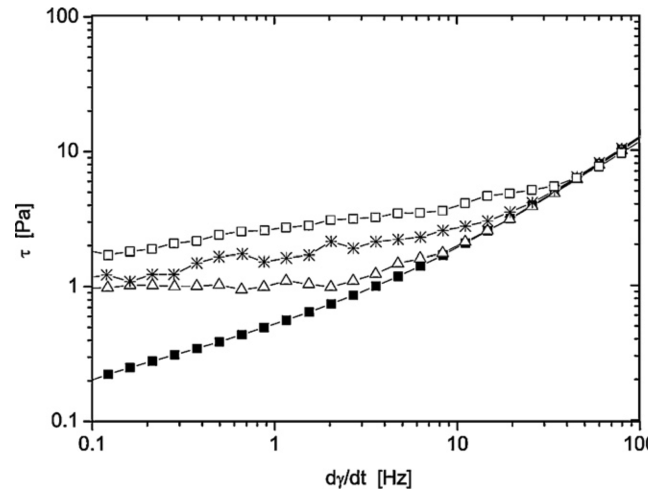

Fig. 5. Shear stress vs shear rate at various electric field strengths for a dispersion with 1.1 wt% CC (filled square 0 V/mm, triangle 100 V/mm, star 200 V/mm and open square 300 V/mm).

the conductivity depends strongly on the packing (pressure) of the powder.

The conductivity of the carbon cone powder was taken to be $\Sigma_p = 0.2$ S/m (measured at 1 V rms and at a pressure of 0.87 kPa) using the relation $\Sigma_p = h/RA$, where h is the distance between the electrodes, A is the area of the electrode and R is the measured resistance of the material.

2.5. Impedance measurements

Impedance spectroscopy was carried out for a CC silicon oil dispersion with 1.1 wt% using an Solartron 1260 + 1294 impedance analyzer and the same cell configuration which was used for conductivity measurements of dry CC particles. The current injecting and potential pick-up terminal leads were connected together at the electrodes, hence enabling two-electrode measurements. The analysis was carried out using frequency sweep from 0.1 Hz to 1 MHz, with an applied voltage in the range 50–3000 mV rms. The distance between the electrodes was 4 mm giving an ac electric field varying between 12.5 and 750 V/mm. The measurements showed that the impedance of the CC dispersion was strongly amplitude dependent (Fig. 4). The dielectric constant ε' for the dispersion was found to be ~3.8 at 1×10^5 Hz, calculated from the relation $C = \varepsilon A/\varepsilon_0 h$, with $C = b/\omega$ where b is the susceptance and ε_0 is the permittivity of vacuum. The dielectric loss ε'' was calculated from the conductance Y' by subtracting the DC conductance from values measured at 1 V rms.

3. Results and discussion

3.1. Electrorheological measurements

The zero field flow curves (not shown) showed that the samples with concentration 0.2 and 0.5 wt% were approximately Newtonian while the samples with 1.1 and 2.5 wt% showed shear thinning behavior. In Fig. 5 the stress vs shear rate at an electric field of 100, 200 or 300 V/mm is shown for a sample of 1.1 wt%. As expected the shear stress increases with increasing field strength. A maximum increase in the yield stress of a factor 10 is observed for this sample. This relatively weak increase in yield stress reflects the low particle concentration. This also caused a response time of the order of minutes. Such a long response time was also reported for a ER fluid composed of SWNT at low concentration [15]. The reason the electric field did not exceed 300 V/mm in the experiment was due to observed electric break down of the system at this field strength. This was seen as a rapid decrease in voltage output after about 1 min at an applied field of 300 V/mm. This was attributed to the formation of a conductive path through the host liquid. The break down lead to unstable electric fields at low shear rates. At higher shear rates, the chain structures broke and the conduction was reduced, thus restoring the electric field. Concerning low electric fields, previous experiments investigating the structure formation of carbon cone particles in ac electric fields [8], showed that the carbon particle dispersions form chains at electric fields as low as 50 V/mm. The response time at such a field strength is slow, on the scale of tens of minutes but decreases rapidly to seconds as the field is increased and with increasing volume fraction. Hence the chosen range of electric field strengths.

Fig. 6 shows the flow curves for samples of various volume fractions at an ac electric field of 100 V/mm. The flow curves may be fairly well described by the Bingham model given by

$$\tau = \tau_y + \eta_{pl}\dot{\gamma}, \tag{1}$$

where τ is the shear stress, $\dot{\gamma}$ the shear rate and η_{pl} the plastic viscosity. The yield stress τ_y was here taken to be the dynamic yield stress at $\dot{\gamma} = 1$ Hz. It should be noted that the dynamic yield stress is in

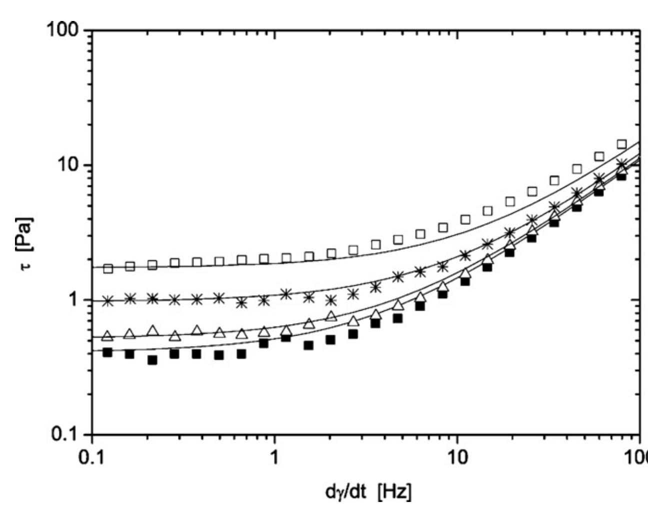

Fig. 6. Shear stress vs shear rate for dispersions with concentrations 0.2 wt% (filled square), 0.5 wt% (triangle), 1.1 wt% (star) and 2.5 wt% (open square). The solid line is the fitted Bingham model. The electric field used was 100 V/mm.

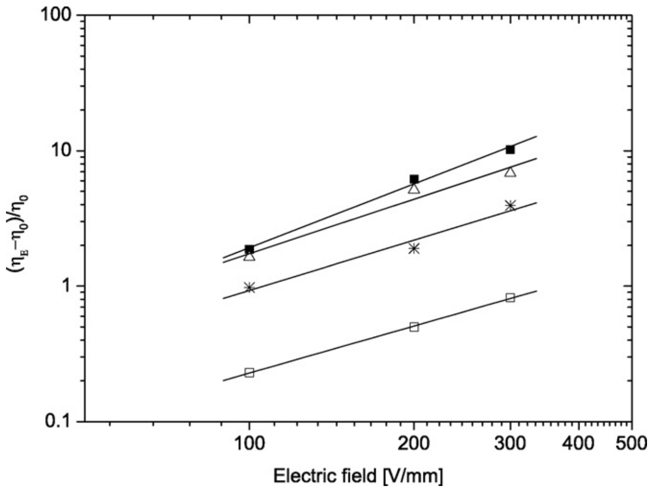

Fig. 7. The effective viscosity $(\eta_E - \eta_0)/\eta_0$ vs electric field for CC dispersions with concentrations 0.2 wt% (filled square), 0.5 wt% (triangle), 1.1 wt% (star) and 2.5 wt% (empty square). The solid lines represent linear fits with the slope of the line equal to α.

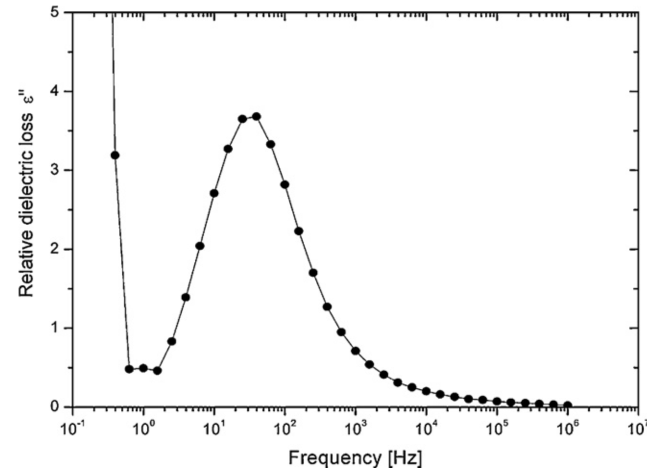

Fig. 8. The relative dielectric loss for a dispersion of 1.1 wt% CC in silicon oil. The main relaxation peak is seen at around 30 Hz. A small peak may be observed at 1 Hz and may be due to electrode polarization.

general not equal to the static yield stress [14]. As expected the yield stress τ_y increases with increasing volume fraction of particles.

The ER efficiency may be expressed by the effective viscosity given by

$$\eta_{eff}^E = \frac{\eta_E - \eta_0}{\eta_0}, \quad (2)$$

where η_{eff}^E is the viscosity measured at an electric field E and η_0 is the zero field viscosity. The effective viscosity scales as $\eta_{eff}^E \sim E^\alpha$ where the exponent α depends on the response of particle organization. It may be seen from Fig. 7 that the effective viscosity is inversely proportional to the volume fraction of the suspensions, decreasing as the volume fraction increases.

The fitted values of α are in the range of 1.15–1.56 for all of the suspensions. The highest α value 1.56 correspond to the lowest CC concentration. According to polarization theory $\alpha = 2$ when a pure dipole interaction is considered. However, the conduction model is appropriate for our system due to the low frequency fields used. Particle polarization and particle interactions are controlled by the particle and fluid conductivities [16]. In the conduction theory α is expected to be $1 < \alpha < 2$ which is in good agreement with the results.

According to Lan et al. [17] there exist a critical conductivity ratio Γ_c where $\Gamma = \Sigma_p/\Sigma_s$, which gives the maximum shear stress. Here Σ_p and Σ_s are the conductivities of the particles and dispersant respectively. For $\Gamma > \Gamma_c$ the ER effect decreases again. According to Lan the ER effect occurs when Γ varies in the $1 \times 10^3 - 4 \times 10^5$ range. Block et al. [18] also reported a maximum ER effect at a particle conductivity of around 10^{-7} S/m, the reason being the strength of the interfacial polarization reaching a maximum at this value. In our system $\Sigma_p = 0.2$ S/m and $\Sigma_s \sim 10^{-12}$ giving $\Gamma \sim 10^{11}$, and despite this high value we still observed the ER effect, in contradiction to Lan's conclusion.

3.2. ER and relaxation frequency

Another important factor which influences the ER effect is the relaxation frequency. Ikazaki et al. [20] found that ER fluids with relaxation frequencies between 1×10^2 and 1×10^5 Hz showed a strong ER effect. They also showed that the ER effect increased with increasing current densities up to a certain level. A high current density giving a relaxation frequency above 10^5 Hz, resulted in a decrease in the ER effect. The same result was reported by Hao et al. [21]. As seen from the dielectric loss factor shown in Fig. 8, the relaxation frequency is around 30 Hz, well below the lower limit of 100 Hz given for a good ER fluid. The low relaxation value may indicate that orientation polarization plays and important role in our system. In agreement with the findings of Block et al. [18] one would expect our system with high particle conductivity to result in a weak interfacial polarization. Hence the shape and size of the particles makes orientation polarization dominate. In Fig. 8 a small peak may be observed at around 1 Hz and this may be due to electrode polarization due to minute amounts of water in the system [22]. The increase seen at the lowest frequency is a result of the uncertainty in the DC conductance and should be disregarded.

Regarding the influence of the particle shape on the ER effect there are various studies in the literature. Lengalova et al. [23] studied the ER effect of various dispersions with solid phase of the different particle sizes and shapes. They found that the ER effect clearly depended on the particle size, shape and on the aggregation abilities of the particles. They noted that at low shear velocities the apparent viscosity did not correlate well with the measured dielectric loss, which was attributed to flaky, asymmetric particles giving rise to increased hydrodynamic interactions at low shear rates. In the present work we would expect a similar behavior due to the abundance of disk shaped particles. In a study by Qi and Wen [24] it was concluded that the ER effect decreases when the aspect ratio of the particles increases. However, Lin and Shan [15] reported a large ER response in a system with SWNT at low concentrations. They suggested that the high aspect ratio of the particles lead to a large viscous force, leading to a greater energy dissipation which resulted in a higher apperant viscosity than compared to low-aspect ratio particles at the same concentration. In this work the majority of the particle have a large aspect ratio, but the area of the particles is large, something which could make the turning of the particles slow due to large viscous forces. This may give rise to the low relaxation frequency found. In the end it is difficult to conclude anything with respect to which of the parameters including, size, shape, particle concentration, dielectric constant or conductivity that influence the ER effect the most in our system.

3.3. Magnetorheological measurements

Fig. 9 shows the relative viscosity η_{rel}^M for 1.5 wt% CC in ferrofluid EMG 901, as a function of shear rate at various magnetic

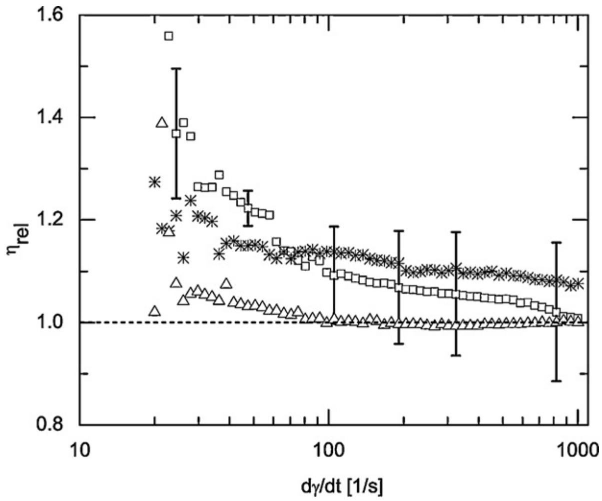

Fig. 9. The relative viscosity η^M_{rel} vs shear rate for magnetic fields $B = 0$ mT (square), 98 mT (star) and 844 mT (triangle) for 1.5 wt% CC in ferrofluid.

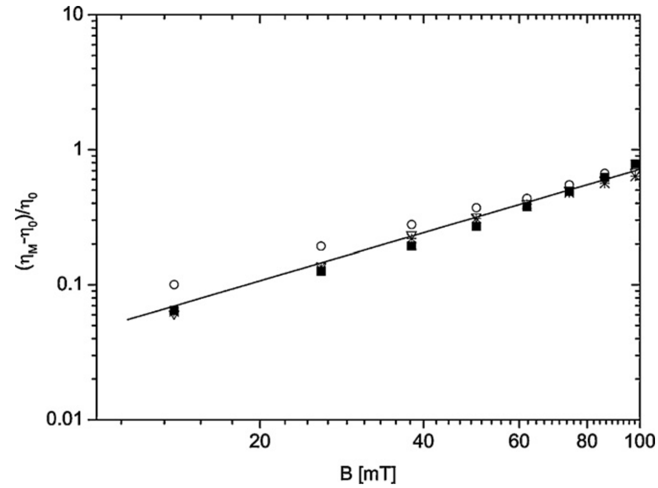

Fig. 10. The effective viscosity for 1.5 wt% CC calculated for various frequencies and magnetic fields B between 0 and 100 mT. Square is for 50 Hz, circle 204 Hz, triangle 451 Hz and star 1000 Hz. The linear fit line has a slope equal to 1.2.

field strengths. η^M_{rel} is given as

$$\eta^M_{rel} = \frac{\eta_{CC}}{\eta_{FF}}, \quad (3)$$

where η_{CC} and η_{FF} is the viscosity of the ferrofluid containing carbon cone particles and the pure ferrofluid, respectively. As expected shear thinning is observed at high shear rates in the whole magnetic field range. This is due to hydrodynamic forces which break up any chains or clusters formed by the magnetic field. The viscosities are also monotonically increasing as function of the magnetic field, however not linear above 100 mT, just above the FF saturation magnetization of 60 mT [13]. At low shear rates and low fields, $\eta^M_{rel} > 1$ indicating that the carbon cone particles increase the viscosity due to their particle–particle interactions and due to the bounded layer of liquid and magnetic particles around each particles giving rise to a larger effective volume. For low shear values and an applied field $B > 200$ mT, η^M_{rel} is 1, indicating that magnetic forces in the ferrofluid dominates over CC particle–particle interactions.

As for ER fluids, an effective viscosity may be defined for MR fluids;

$$\eta^M_{eff} = \frac{\eta_M - \eta_0}{\eta_0}, \quad (4)$$

where η_M is the viscosity at an applied magnetic field, and η_0 is the viscosity at zero field. The effective viscosity η^M_{eff} is shown in Fig. 10 for magnetic fields $B < 100$ mT where the system showed a linear behavior. In this region η^M_{eff} was found to scale as $\eta^M_{eff} \sim B^\beta$ with β=1.2. For pure FF (not shown) β=1.5 which means that the response due to a change in magnetic field is stronger for pure FF compared to the CC dispersion. The reason for this, as for the η^M_{rel}, is that the CC particle–particle interaction reduces the solutions response to the magnetic field. The influence of the frequency on the viscosity is small, as seen in Fig. 10.

As reported by Samouhos and McKinley [25] an MR fluid consisting of carbon nanotubes (CNT) and magnetite particles in an organic ferrofluid has successfully been made. Other CNT–magnetite composites have also been reported [26,27]. Samouhos and McKinley describe a non-covalent surface coating of CNTs with magnetite nanoparticles. It could be possible to adapt this method for the carbon cone particles in order to produce a better magnetorheological fluid.

4. Conclusion

Carbon cone particles in dispersions have been subjected to electric and magnetic fields. In electric fields the dispersions of CC in silicon oil show a typical Bingham fluid behavior with a maximum increase in yield stress of a factor 10. The relatively low ER effect as compared to commercial ER fluids is attributed to the high particle conductivity and the low relaxation frequency as determined by impedance spectroscopy.

In a magnetic field the addition of carbon cone particles to an organic based ferrofluid gave a small increase in the viscosity at low shear rates and low fields. This is attributed to purely hydrodynamic effect of increased binding of the fluid by the particles. At higher fields the dispersion show shear thinning behavior.

The study shows that the carbon cone material is not adapt for use as solid phase in ER/MR fluids. However, the possibility of easy alignment of carbon cones particles using AC electric field with low voltages motivates the use of this material in composites to create anisotropic conductive paths. This is currently under investigation.

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