

Development and Characterization of Hydrocarbon Polyol Polyurethane and Silicone Magnetorheological Polymeric Gels

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ABSTRACT: Magnetorheological polymeric gels (MRPG) have been developed for use in semi-active magnetorheological fluid (MRF) dampers and other magnetorheological (MR) devices. The novel MRPGs are prepared by suspending iron particles in polymeric gels. Off-state (i.e. no applied magnetic field) viscosity and settling behavior can be controlled through the selection of polymeric gels. In this study, tunable rheological properties were investigated with a piston-driven flow type rheometer with a shear rate varying from 20 s^{-1} to $6,000 \text{ s}^{-1}$. Silicone MRPG (with 84.5 wt % iron particles) has controllable viscosity and a high shear yield stress over a wide range of shear rates. Silicone MRPG (79.5 wt % iron particles) has the lowest viscosity of those studied. Polyurethane MRPG has the lowest settling rate. The order of addition of magnetic particles and polymer during the polymerization process affects the MRPG final off-state ap-

parent viscosity (80% increase in apparent viscosity for silicone MRPG polymerized after adding iron particles). This indicates that polymer gels modify the surface properties of the magnetic particles, causing interaction among particles. The dynamic shear yield stress is higher for fluids with better dispersion stability. Polyurethane MRPG, which has the lowest settling rate, has a high dynamic yield stress (23 kPa at 350 mT). Both dynamic and static shear stress values of the MRPGs were found to be similar in magnitude (5–8 kPa at 120 mT for silicone MRPG with 84.5 wt % iron particles and polyurethane MRPG), indicating that MRPGs can provide consistent performance in devices. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 1176–1182, 2004

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INTRODUCTION

Magnetorheological fluids (MRFs) can respond to an externally applied magnetic field with a rapid and reversible change in their apparent viscosity. MRFs can be used in a variety of applications. These applications include engine mounts, shock absorbers, clutches, and seat dampers.^{1–4} Other applications are exercise equipment and aspherical optical lens polishing.³

Without an applied magnetic field, a typical MRF behaves similarly to a Newtonian fluid.^{1,2} When a magnetic field is applied, a dipole moment is induced within the suspended iron particles by which they align “head-to-tail” and form chains parallel to the magnetic field.² A MRF becomes a weak viscoelastic solid as the chains form into column-like structures when the applied magnetic field increases. As a result, the rheological properties of the material changes.⁵ MRF properties are controllable because of the dramatic change in their rheological properties under an

externally applied magnetic field. Therefore, they provide a new means of controlling electromechanical devices.³

A new generation of MRFs, known as magnetorheological polymer gels (MRPGs) are used in vibration control and damping devices.^{6,7} These fluids have the advantage of providing controllable viscosity of the carrier fluid as well as reducing the settling of magnetic particles in the fluid. This behavior is possible because the polymer gel distributes itself between the carrier fluid and the surface of the magnetic particles. In this respect, MRPGs are significantly different from traditional MRFs.

Previous work on MRPGs⁶ describes the development and characterization of poly(ethylene glycol) (PEG) and polyether polyol based polyurethanes and silicone polymer gels. These polyurethane gels are limited by their poor compatibility with hydrocarbon carrier fluids. These MRPGs were characterized using thermal analysis for determination of the kinetic behavior of the polymer gels.

MRPGs are disclosed in U.S. Patent 6,527,972.⁷ These fluids contain partially crosslinked polymer gels, which are synthesized using non-stoichiometric amounts of monomers. They may include plasticizer,

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diluent, or other additives. These fluids can be synthesized with a wide range of controllable viscosities.

Polymer gels have been utilized in the development of a variety of other magnetic fluids as well. Patent WO 97/02580⁸ describes a ferrogel that is a crosslinked polymer swollen by a ferrofluid. This gel is designed for control of its shape. JP 05-25316⁹ describes a gel or rubber material containing magnetic particles. This patent describes gels of poly(vinyl alcohol), polyacrylamide, polystyrene, poly(alkyl thiophene) and several other polymers. The polymer gels described in this patent have controllable elastic modulus and shape. Magnetic colloidal particles with sizes from 5–100 nm are described in JP 10-296074.¹⁰ A dispersion of the colloidal particles in a crosslinked polymer is disclosed in this patent. The polymers used were copolymers of isobutene and maleic anhydride, copolymers of methyl vinyl ether and maleic anhydride and natural products including starch, agar, gelatin and casein.

Control of the rheology of MRFs is the most important characteristic of these fluids. The behavior of MRFs under external magnetic fields can be described by the Bingham plastic model.¹¹ When the applied shear stress, τ , is less than the controllable shear yield stress, τ_y , of the MRF, the relation between the required shear stress and the shear strain γ is illustrated by eq. (1):

$$\tau = G' \gamma, \quad \tau < \tau_y. \quad (1)$$

Here the materials are viscoelastic solids having non-zero shear moduli, G' . When the shear stress, τ , is greater than τ_y , the material starts to flow. The Bingham equation can be used to relate the shear stress, τ , to the shear strain rate, $\dot{\gamma}$, in the post-yield state:

$$\tau = \tau_y + \eta \dot{\gamma}, \quad \tau \geq \tau_y. \quad (2)$$

where η is the plastic viscosity.

There are two different yield stress values, the dynamic yield stress ($\tau_{y,d}$) and the static yield stress ($\tau_{y,s}$), that are usually reported.¹² Weiss¹² describes "the dynamic yield stress of a controllable fluid as the zero-rate intercept determined through a linear regression curve fit to the measured flow data, while the static yield stress corresponds to the shear stress necessary to initiate flow. The plastic viscosity of a controllable fluid corresponds to the slope of the linear regression curve fit used in analyzing the flow data."

According to Weiss, the dynamic and static shear yield stress values measured for a controllable fluid need to be similar in magnitude in order to simplify the design of a device and guarantee consistent device performance. For many controllable fluids the static shear yield stress values are greater than the corresponding dynamic shear yield stress values. This phenomenon is known as stiction.¹² Knowledge of both

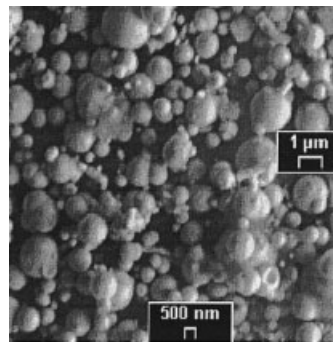


Figure 1 SEM image of carbonyl iron particles with 97.5% purity (X3500).

the static and the dynamic shear yield stress values is important for the design of MR devices in which the MRFs are working in either a 'static' (for example, MR polishing facility) or a 'dynamic' state (MR dampers, MR clutches, etc). In this study, two measurement methods are developed to investigate both the dynamic and static yield stresses of the MR materials.

In general, three components constitute a MRF: iron particles, carrier fluid and dispersant/additives. Iron particles are suspended in the carrier fluid along with surfactants and other additives. The carrier fluid is the medium used to suspend all components. Additives are necessary for mixture stability and surface protection (anti-wear and corrosion-control), which extend the operating life of MRFs and their devices.

In this study, novel MRPGs with controllable viscosity and magnetic particle settling behavior were developed. These materials contain polymer gels based on non-stoichiometric ratios of reactants. Novel polyurethane gels, based on hydrocarbon polyols were developed. These polymer gels have superior compatibility with hydrocarbon carrier fluids, including polyalphaolefins (PAO). The polymer gel distributes itself between the carrier fluid and the surface of the magnetic particles depending on the order of polymerization and the addition of magnetic particles. In addition to the viscosity and particle settling, which are dependent on the properties of the polymer gels, the magnetic properties (B-H curve) and yield stress were characterized as well.

EXPERIMENTAL

Materials

Magnetic Particles

Spherical reduced carbonyl iron particles with purities of 97.5% and 99.5% were used in this study (ISP Corp., S-1651 and ISP Corp., R-2430, respectively). These iron particles have particle sizes ranging from 1 to 10 μm in diameter. Figure 1 is a scanning electronic microscope (SEM) image of carbonyl iron particles (1–3 μm) with 97.5% purity.

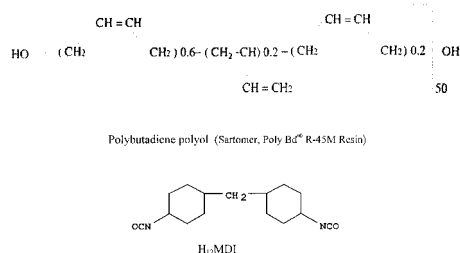


Figure 2 Structures of polyurethane materials.

Polyurethane Gels

Polyurethane gels were prepared using PAO as the carrier fluid for polyurethane MRPG. Polyurethane was prepared by the reaction of poly(butadiene polyol) and isocyanate. The poly(butadiene polyol) was hydroxy-terminated (Sartomer, Poly Bd® R-45M Resin) with an equivalent molecular weight of 2800 and a functionality of 2.2-2.4. The isocyanate was H₁₂MDI (Bayer, Desmodur W) with an equivalent molecular weight of 132 and a functionality of 2.0. PAO (Chevron, 2cst) was used for viscosity control of the polyurethane system. Reduced carbonyl iron particles with 99.5% purity were used for formulation of the polyurethane MRPGs. Figure 2 shows the chemical structures of the polyurethane materials. There was 10.0 wt % polyurethane in the system, and the equivalent ratio of H₁₂MDI/polybutadiene polyol was 0.80.

Silicone Gels

Silicone MRPG was prepared by mixing silicone oil with a silicone polymer and iron particles. Vinylpolydimethylsiloxane (VPDMS) resin (GE, Silicones RTV6136A) is difunctional, with a molecular weight of approximately 10,400. Dimethyl methylhydrogenpolysiloxane (DMMHPS) is a crosslinker with a molecular weight of approximately 10,400 (GE, Silicones RTV6136B). Silicone oil with low viscosity (5 cp) (GE, Silicones SF96-5) was used for viscosity control of the silicone polymer system. Iron particles with a purity of either 97.5 or 99.5% were used. Fumed silica (CABOT, CAB-O-SIL® TS-720) was added as a dispersion additive. The structures of silicone polymers are shown in Figure 3. There was 2.0 wt % polymer in the system, and the equivalent ratio of DMMHPS/VPDMS was 0.11.

Methods and Instrumentation

MRPG Synthesis

For the preparation of MRPGs, the polymer system was first added to the hydrocarbon oil or silicone oil. The dispersants/additives were then added to the hydrocarbon oil or silicone oil and mixed completely (at low shear rate). Iron particles, 79.5 to 84.5 wt %,

were dispersed in a carrier fluid. The mixture was mixed at 1000 rpm for 10 min at room temperature. Two different polymer MRPGs, polyurethane MRPG and silicone polymer MRPG, were developed. Commercial MRF (MRF-132LD) from Lord Company was examined as a benchmark in this study. Table I is the component list of two silicone MRPGs and one polyurethane MRPG.

Instrumentation

Piston-Driven Rheometer. Several experimental approaches using different measurement geometries have been investigated to measure the MR properties of MR fluids.¹³⁻¹⁶ For this study, a piston-driven flow type rheometer was designed and built to evaluate the tunable rheological properties of the various MRPGs.

Figure 4 shows the piston-driven flow type MR rheometer with a rectangular cross section. An electromagnet was built of low-carbon steel that provided a magnetic field normal to the rectangular channel flow. The magnetic flux density inside the channel induced by the coil was measured by a Gauss meter and varied from 0.12 T to 0.35 T.

The MRFs were confined in a well-sealed channel cell and pressurized to initiate flow through the channel between two parallel electromagnetic poles. The flow rate was controlled by the velocity of the piston driven hydraulic source, which was connected to an Instron 8821S material testing machine. The pressure drop across the channel was measured by two fluid pressure transducers. The flow channel was 1.0 mm in height (h), 10 mm in width (w) and 14 mm in length (l).

The shear stress of the MRF was calculated as follows:

$$\tau_w = \frac{h\Delta p}{2l} \text{ for } h \ll w \quad (3)$$

where Δp is the pressure drop across the channel. The shear rate of the MRF was obtained using eq. (4), which assumes Newtonian flow inside the channel:

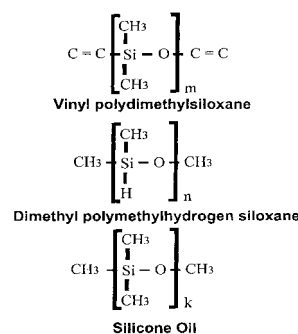


Figure 3 Structures of silicone gel components.

TABLE I
MRPGs Components List

	Iron Particle	Base Oil	Polymer Gel		Dispersion
Silicone MRPG	79.5 (97.5% purity)	18.1 (5cp silicone)	1.8 (VPDMS)	0.2 (DMMHPS)	0.4 (fumed silica)
Silicone MRPG	84.5 (99.5% purity)	13.6 (5cp silicone)	1.35 (VPDMS)	0.15 (DMMHPS)	0.4 (fumed silica)
PAO MRPG	80 (99.5% purity)	10 (2cst PAO)	9.55 (polybutadiene-diol)	0.45 (H ₁₂ MDI)	—

* Numbers are weight percentages of each component

$$\dot{\gamma}(\tau_w) = \frac{6Q}{h^2w} \tag{4}$$

$$\tau_y = F_y/2a^2 \tag{5}$$

where Q is the volumetric flow rate of MRF.

Investigation of the physical properties of the MRPG was also performed through the use of a dynamic mechanical analyzer (DMA, Perkin-Elmer DMA-7e). The DMA is a force-controlled instrument that can apply a tensile or a compressive force. The main bearing is a magnetic bearing, and it is driven by a linear motor consisting of a magnetic coil and a coaxial shaft. It records strain measurements using a linear variable differential transformer (LVDT). A custom-built shear test device was used to measure MRPG static shear yield stress. Applied magnetic field is generated by a c-frame electromagnet driven by a DC power supply (BK Precision). Samples are contained in a rectangular channel within the generated magnetic field. By controlling the electric current of a power supply, a variable magnetic field was created. This custom-designed shear accessory is used in measuring the shear yield stress of the MRPG under the influence of a magnetic field. Figure 5 is a diagram of the DMA shear yield stress test device. The aluminum probe was inserted into a channel filled with the sample.

After activating the electromagnet, a static force was applied on the probe. When the static force reached yield threshold, the probe was withdrawn from the channel instantly, and at this point the measured force directly provided the shear yield force of the sample. This force was divided by the probe area to determine the shear yield stress. The shear yield stress can be calculated from eq. (5):

where a^2 is the side area of the probe. Figure 6 is a typical result for the DMA static shear test. F_y can be determined from the intersection of the force-time curve and the probe position-time curve (see Fig. 6). The static force applied to the probe was increased linearly at a constant rate of 40 mN/min from 0 to 520 mN. The probe position changed slowly before reaching the yield point, when the static force was lower than 396 mN. When the static force reached the critical point of 396 mN, we observed a large movement in the probe position. At this point, the iron particle columns broke because the fluid had reached the yield stress. Brookfield Viscometer. A Brookfield viscometer (Model # DV-III) was used for apparent viscosity measurements. The apparent viscosity versus shear strain rate (0–200 s⁻¹) was measured at room temperature. Spindles 21, 27, 28 and 29 were used in these studies.

Particle Settling

Since the density of the dispersed iron particles was much higher than that of the carrier fluid, settling of iron particles was generally observed. The particle settling rate can be determined by measuring the rate of formation of a clear fluid layer on the surface of the fluid when a sample is permitted to settle for a period of more than 60 days at room temperature. This layer is measured as a clear fluid volume fraction as the iron particles settle into the carrier fluid in a graduated

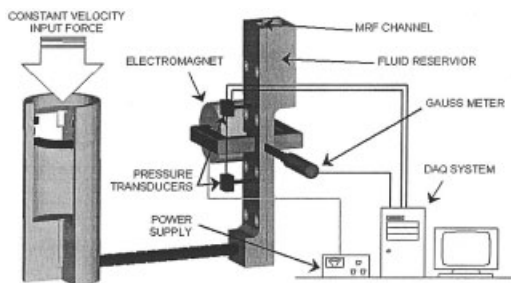


Figure 4 Piston-driven flow type MR rheometer.

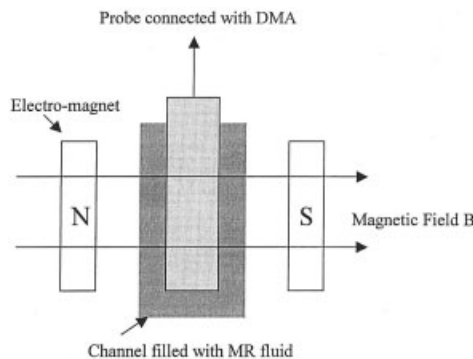


Figure 5 DMA shear stress-strain test device

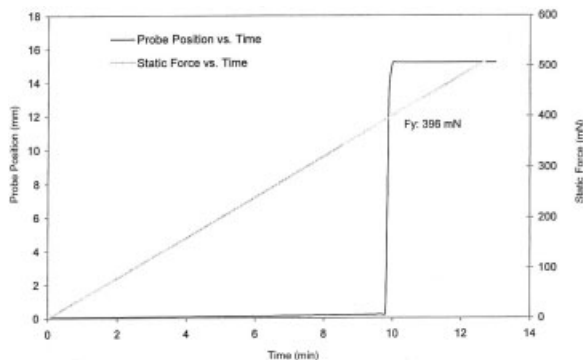


Figure 6 Typical DMA shear testing force-position results.

glass cylinder (10 mL). The clear fluid volume fraction rate of change is measured as a function of time.

Magnetic Properties of MRPGs (M-H Curve)

The static magnetic properties of the samples were measured using a simple magnetic-induction-based technique. The sample was held in one of a pair of identical but counterwound wire coils; the other coil was empty. The coils were placed in the gap of a C-frame electromagnet (GMW 5403), and the applied magnetic field H was measured by a Hall-effect Gaussmeter (Lakeshore 450). By integrating the voltage generated in the coils when a field is applied, a signal proportional to the sample magnetization M was obtained.

RESULTS AND DISCUSSION

Off-state apparent viscosity

Figure 7 compares the off-state (i.e., zero applied magnetic field) apparent viscosity of MRPGs and a commercial MRF at room temperature. All MRPGs and the

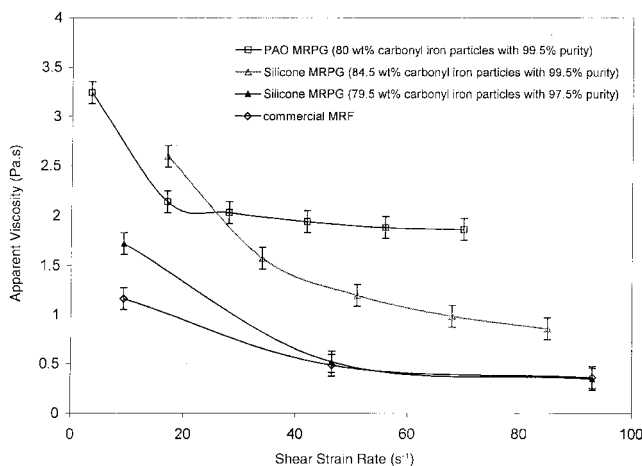


Figure 7 Apparent Viscosity of various MRPGs and a commercial MRF.

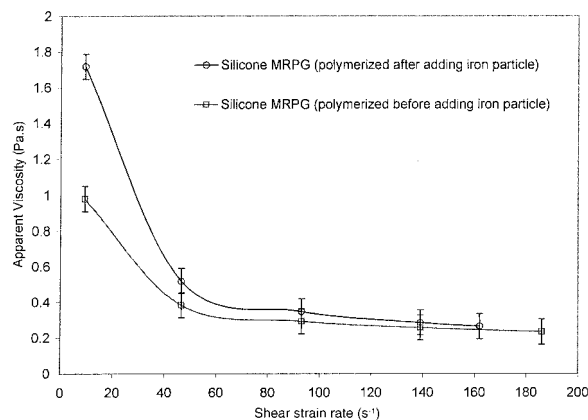


Figure 8 Polymerization process effect on MRPG apparent viscosity.

commercial fluid exhibited shear-thinning behavior. At higher shear strain rates, the viscosity decreased, ultimately reaching a constant value. The silicone MRPG (79.5 wt % carbonyl iron particle with 97.5% purity) was designed to have a similar viscosity to commercial MRF at higher shear strain rates. The polyurethane MRPG (80 wt % carbonyl iron particle with 99.5% purity) containing polyurethane gel set the upper viscosity values for these fluids. A polyurethane MRPG with 85% iron particles was synthesized but was not fully characterized due to the excessively high viscosity of this fluid. MRPGs can be easily synthesized with intermediate viscosities based on their required application. The polymerization process, specifically, the order of addition of the polymer and magnetic particles, affects the apparent viscosity of the MRPGs. This is shown in Figure 8 for silicone MRPG. The data in Figures 7 and 8 are connected with smoothed lines. Generally, it takes 24 h for silicone to cure at room temperature. One silicone MRPG formulation was prepared by adding the iron particles before adding the silicone curing agents. For another silicone MRPG formulation, the silicone curing agents were reacted for 24 hours before the iron particles were added. Figure 8 shows that at low shear rates the silicone MRPG (polymerized after adding iron particles) had 80% higher viscosity than the silicone MRPG (polymerized before adding iron particle). This was most likely caused by the polymerization of polymer gel on the particle surface in the case of the MRPG polymerized after adding the magnetic particles. It was proposed that the interaction between the polymer gel on the nearby particles caused the observed increase in viscosity at low shear rates. This indicates that the surface of the iron particles may be substantially modified.

Particle settling

Figure 9 shows the settling data of MRPGs and a commercial MRF. In this figure, larger volume frac-

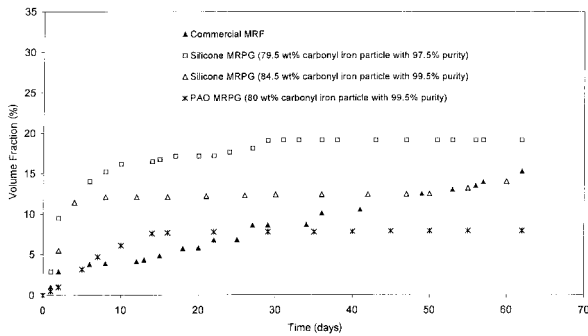


Figure 9 Settling rate results of various MRPGs and a commercial MRF.

tions (vol %) indicate greater settling. For long settling times, MRPG had a lower settling rate compared to commercial MRF and other MRPGs. For short settling times (<20days), all MRPGs had higher settling rates than commercial MRF. Unlike MRPG, commercial MRF likely incorporates surfactants to stabilize the dispersion. MRPGs reach a steady rate of settling faster than commercial MRF. A possible explanation for this behavior is that MRPGs have high sedimentation volume because of the formation of a polymer network. Furthermore, since these fluids exhibit shear thinning behavior (Fig. 7), can easily be redispersed under shear. The error in the particle settling measurements was estimated at 1%.

Static shear yield stress

Figure 10 shows the static shear yield stress of several MRPGs and a commercial MRF under different magnetic fields. The tests were performed under low applied magnetic fields ($0 \approx 0.125$ T). Static yield stress at low magnetic fields was compared using a linear fit because the force between magnetic particles increases linearly with magnetic field below the magnetic field

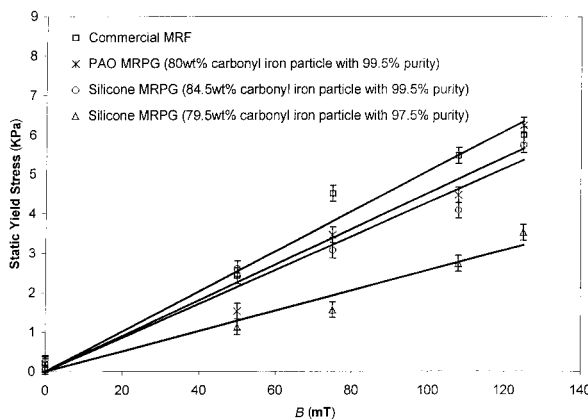


Figure 10 Comparison of static shear yield stress of various MRPGs along with a commercial MRF under different applied magnetic fields.

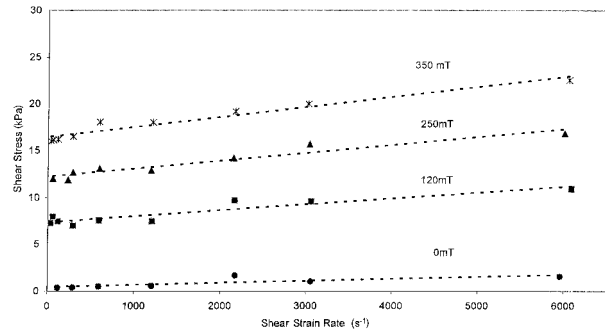


Figure 11 Dynamic shear stress versus shear strain rate under different applied magnetic fields for silicone MRPG (79.5 wt % carbonyl with iron particles of 97.5% purity).

saturation level. At these low applied magnetic fields ($0 \approx 0.12$ T), commercial MRF exhibits the highest static shear yield stress.

Dynamic shear yield stress

The effect of applied magnetic field on the dynamic shear yield stress of the MRPGs was investigated using the piston-driven flow type MR rheometer. The results of the shear yield stress versus strain rate for a typical silicone MRPG are shown in Figure 11. The straight lines are representative of Bingham Plastic behavior [eq. (2)]. The dynamic shear yield stress can be obtained by extrapolating shear stress data back to a zero strain rate. The plastic viscosity (η) is the slope of the shear stress data. For an increase in magnetic field strength, a similar increase in the respective shear yield stress is observed, while the plastic viscosities are not significantly affected. For example, for silicone MRPG (79.5 wt % iron particles), at a 250 mT magnetic flux density, the dynamic yield stress was 12.2 kPa, while at 350 mT the yield stress was 16.4 kPa.

Figure 12 shows the results of the dynamic shear yield stress of different MRPGs and a commercial MRF measured using the piston-driven flow type MR rheometer. The data in Figure 12 were correlated by fitting smoothed curves through the data. At lower

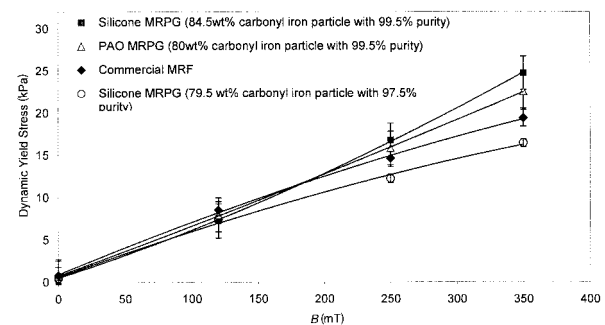


Figure 12 Comparison of dynamic yield stress with different MRPGs.

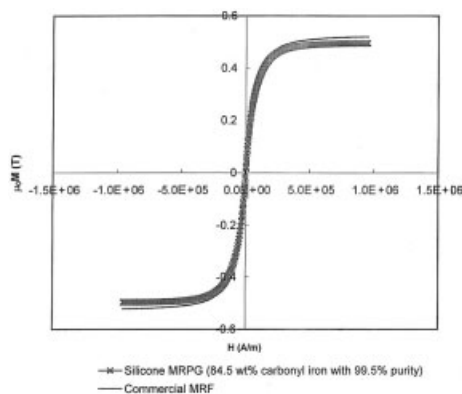


Figure 13 M-H curves for a silicone MRPG and a commercial MRF.

applied magnetic fields ($0 \approx 150$ mT), the dynamic shear yield stresses of all fluids were similar. By increasing the magnetic field, some MRFs were made to exhibit much higher shear yield stress than others. It was speculated that the differences in behavior among the fluids are related to their relative dispersion stabilities. Under a 350 mT field, the silicone MRPG (84.5 wt % iron particles) exhibited the highest shear yield stress value of 24.7 kPa. The shear yield stress of polyurethane MRPG (80 wt % iron particles) at 350 mT field was 22.5 kPa, while the commercial MRF had a shear yield stress around 19.4 kPa. It was noted that the shape of the dynamic yield stress curves differed depending on their dispersion stability. The most stable fluid, based on settling data (Fig. 9), was the polyurethane MRPG. This fluid exhibited high dynamic yield stress, which increased sharply with magnetic field. On the other hand, the least stable fluid, based on settling data, was the silicone MRPG (79.5% iron particles). This fluid had the lowest dynamic yield stress and increased only slightly with magnetic field. Furthermore, the static and dynamic shear yield stress values (Figs. 10 and 12, at magnetic fields between 0 and 120 mT) were found to be similar (5–8 kPa at 120 mT for silicone MRPG with 84.5 wt % iron particles and polyurethane MRPG), indicating that MRPGs can provide consistent performance in devices.

M-H curve

A typical field-dependent magnetization (M-H) loop for a silicone MRPG, and the commercial MRF are shown in Figure 13. As the applied field was increased, the magnetization first increased rapidly, then eventually saturated. For the silicone MRPG and the commercial MRF, $\mu_0 M_s$ was about 0.5 T. This value was dependent on the concentration of magnetic particles present, which was approximately 79.5–84.5 wt % for both fluids.

CONCLUSIONS

Silicone and polyurethane MRPGs were prepared by suspending iron particles in polymeric gels. The results of this investigation indicate that off-state viscosity and particle settling can be controlled using MRPGs. Silicone MRPG (79.5 wt % iron particle) had the lowest viscosity. The order of addition of magnetic particles and polymer during the polymerization process affects the MRPG final off-state apparent viscosity (80% increase in apparent viscosity for silicone MRPG polymerized after adding iron particles). This indicates that the polymer gel modified the surface of the magnetic particles, and the interaction between nearby particles affected the viscosity at low shear rates. Polyurethane MRPG was developed for low settling rates. The dynamic shear yield stress is higher for fluids with better dispersion stability. Furthermore, the static and dynamic shear yield stress values were found to be similar (5–8 kPa at 120 mT for silicone MRPG with 84.5 wt.% iron particles and polyurethane MRPG), indicating that MRPGs can provide consistent performance in devices.

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