Development and Characterization of Magnetorheological Polymer Gels

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ABSTRACT: Magnetorheological materials have been used in many applications in recent years. To develop new materials, polyurethane and silicone polymer gels were investigated. Rheology is qualitatively controlled for each system by controlling the concentration of reactants and diluents. The resulting polymers have solid, gel, or liquid states, depending on the crosslinking and dilution. The gels were characterized through kinetic analysis. Differential scanning calorimetry (DSC) was used with analysis methods to find the kinetic properties for diluted and undiluted polyurethane systems. Heat of reaction, order of reaction, preexponential constant, and activation energy were obtained from the experimental DSC data. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 84: 2733–2742, 2002

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INTRODUCTION

Magnetorheological polymer gels (MRPGs) are a new generation of materials used for vibration control, damping, and clutch applications as well as other energy-absorption applications. These composite polymeric fluids permit control of viscosity, provide high yield stress, and exhibit low particle settling behavior.[1]

Although many characteristics of MRPGs are similar to those of magnetorheological fluid (MRF), the MRPG differs from the traditional MRF in several ways. Through formulation, the MRPG may have a viscosity customized for a given application. A higher viscosity results in reduced particle settling and aids in fail-safe operation in the event that the magnet fails. Also, the polymer may react before or after inclusion of the iron particles. By doing so, it can alter the

properties of the MRPG. This is suspected to be a trade-off between yield stress and stability as reacting in the presence of the iron particles will coat them, which in turn may improve stability but decrease the magnetic interaction between particles. Thus, even though applications are similar, there is better control of the material used in a specific device, such as a damper; therefore, the designer of the device has more flexibility in design.

MRFs are commercially available magnetic fluids that are currently used for a variety of applications. These include use in automotive parts: engine mounts, shock absorbers, and seat dampers.[2–6] Other applications cover a range from exercise equipment to aspherical optical lens polishing. In the area of vibration control and damping, earthquake-resistant structures are built that utilize these fluids using semiactive control.[2,3,5–7]

MRFs excel in these applications because their rheological properties are controlled over several orders of magnitude. Without an applied magnetic field, the typical MRF acts like a Newtonian

fluid.[3,8] When a field is applied, a dipole moment is induced in the particles in the MRF. This causes the particles to align "head-to-tail" and form chains of particles parallel to the magnetic field.[3] The MRF becomes a weak viscoelastic solid when the chain or column structures form. As the magnetic field increases, the material exhibits a rapid and nearly reversible increase in yield stress. Because of the change in material properties under the influence of a magnetic field, the MRF properties are controlled and therefore provide a new means of controlling electromechanical devices.[3,6,9]

Although MRFs may be similar to ferrofluids, they also have important differences. They are composed of three components like ferrofluids; thus, they have a carrier fluid, magnetic particles, and additives.[10] However, the particles used in ferrofluids are superparamagnetic iron oxide nanoparticles ($\sim$ 5–10 nm).[2,10] As a result, they do not exhibit a shear yield stress like that of MRF while under an applied magnetic field.[2,5] This is attributed to a reduced tendency to form chains under a magnetic field. Rather, the field acts as a body force on the entire material. Thus, although viscosity changes can be observed, they are small.[5,11] In addition to being used in seals, the ferrofluids have applications in stepper motors and sensors.[10]

For an MRF, magnetic particles, such as iron, can be suspended in a fluid. Under a magnetic field, these particles form chains[2,12,13] that significantly increase the yield stress of the material. The carrier fluid acts as the medium for other components. Suspended in the medium are the magnetic particles that form chains when a magnetic field is applied. Finally, additives are used to provide stability to the mixture, corrosion control, lubrication, antioxidants, pH shifters, dyes and pigments, salts, and deacidifiers.[2,8,12,14]

Typically, the medium is a silicone oil or hydrocarbon fluid.[2,8] This is because it exhibits many of the properties that are desirable in MRFs. Ideally, the fluid should be thermally stable and have a high boiling point. The carrier fluid should be noncorrosive and nonreactive with the magnetic particles and other components, and it should be nontoxic. The fluid should contribute to the stability of the mixture, but at the same time enable the redispersibility of the magnetic particles. The temperature dependency of the medium's viscosity is also very important, and is in fact the dominating factor in the operating range of the MRF.

Finally, the fluid should not cause sealing problems in the device in which it will be used.[3,12]

The dispersed phase usually is a soft magnetic material such as iron particles of 1–10 μm size.[2] Several important factors must be considered in the choice of the dispersed phase. The volume fraction of the magnetic materials is usually 0.3 to 0.5 volume fraction of carbonyl iron. This leads to a reasonable yield stress but does not have the potentially undesirable higher off-state viscosity of a higher volume fraction.[12] The particle size has a great influence on the rheology of the on and off states of the fluid. For larger particles (5–7 μm) the yield stress is greater than that for smaller particles ($\sim$ 2 μm). Particles larger than 10 μm have increased settling and thus form less-stable MRFs. Several problems occur when the particles are too small, which are more influenced by the carrier fluid than are the larger particles. They are also more sensitive to temperature.[12] Also, the possibility of agglomeration increases. Nano-MR fluids are described in the literature.[12,15] BASF researchers created stable (by using polyelectrolyte adsorption) nano-MR fluids by using ferrites (<100 nm). However, the yield stress is only about 6 kPa and it is temperature sensitive.[12]

The manufacture of iron and iron-based alloys is achieved using several methods: decomposition of iron pentacarbonyl, sol–gel ultrasonic decomposition of organometallic precursors, plasma torch synthesis, electroexplosion of metal wires, chemical reduction and precipitation, and laser ablation. Preferably, soft magnetic materials like iron are used for their high saturation magnetization. Fe–Co alloys have the highest saturation magnetization ($\sim$ 2.4 T), but cost and unavailability make them undesirable unless the higher material strength is needed. Ferrimagnetic materials such as manganese–zinc ferrite and nickel–zinc ferrite ($\sim$ 2 μm in size) have a lower saturation magnetization and thus they have a lower maximum yield stress.[12]

MRF additives are necessary to prevent agglomeration and settling. As the particles settle and the distance between them decreases, the small level of remnant magnetization could play a role in agglomeration. Some of the materials used as additives are nanostructured silica, fibrous carbon, and various polymers. Nanoscale silica forms a coating on magnetic particles as a thixotropic network.[12]

Several approaches for development of MRFs are documented in the patent literature. Patent 5,985,168 describes the use of a bridging polymer to modify the surface of the iron particles. This approach leads to improved stability and redispersibility. In this patent only three polymers are described: polyvinylpyrollidone, polyethyleneamine, and poly(4-vinylpyridine).[16]

Organic polymers are also used to coat the surface of iron particles that are described in Patent 5,989,447. This patent describes many families of polymers that are used and exhibit reduced abrasiveness and produce high stability with regard to settling.[17]

Polymeric thixotropes are also described in Patent 5,645,752.[18] The mechanism for stability in this invention is ascribed to hydrogen bonding. A large number of polymeric materials are included in this patent for increased viscosity. They are used to exhibit minimum particle settling over a broad temperature range.

Polymerization of MRPGs takes place either before addition of the iron particles or in the presence of the iron particles. The latter case may result in precipitation of polymeric gels on the surface of the iron particles. This may have an additional effect on the stability of the materials.

Polymer gels used in this investigation are polyurethanes and silicones. The rheology of each system is shown to be controllable. MRPGs are prepared by suspending iron particles in the polymeric gel before (or during, or after) crosslinking. Rheological properties are investigated with and without magnetic field. Because MRPGs can be developed at different levels of "off-state" properties through formulation of resin and crosslinkers, the material viscosity is custom suited to a particular device and, in the case of dampers, a fail-safe characteristic is possible. Additionally, because polymer crosslinking may also take place on the ferromagnetic particle surface by the reaction taking place in the presence of the particles, settling of the ferromagnetic particles is reduced.

To investigate kinetic properties, several methods were examined. Ortega[19] describes a controlled rate thermal analysis (CRTA). The objective is to try to control the rate of heating such that the reaction rate remains constant. Sbirrazzuoli et al.[20] describe several isoconversional (where properties are assessed at a set conversion) and peak maximum evolution (where properties are assessed at the thermal peak) methods of analysis. These include Friedman, Ozawa, "Ozawa-corrected," and Kissinger–Akahira–Sunose for isoconversional methods and Kissinger and Malek for peak maximum evolution methods. Single heating rate and multiple heating rate methods (such as Kissinger) may also be found in Turi.[21] The former is not well suited for systems reacting over a large time–temperature range. The Ozawa–Flynn–Wall method can be used as an isoconversional or as a peak maximum evolution multiple heating rate method.[20,21]

EXPERIMENTAL

Materials

Three different matrix materials were investigated. A polyurethane system and a silicone system were all developed.

The polyurethanes were formulated from reactions between polyols and isocyanates. Two polyols were studied: a polyglycol and a polyether polyol. The polyglycols were linear polymers of alkylene oxides. The polyglycol used was polyethylene glycol (PEG), which has an average molecular weight of 600 and a functionality of 2.0 (Polyglycol E-600; Dow Chemical, Midland, MI). A second polyurethane system was based on a polyether polyol (Voranol 360; Dow Chemical) with equivalent weight of 162 and functionality of 4.5. The isocyanate used was polymethylene polyphenyl isocyanate (p-MDI, Dow PAPI 27), which has a functionality of 2.7 and an equivalent weight of 134. The nonreactive plasticizer used was propanol, oxybis-, dibenzoate (PODB, Benzoflex; Velsicol Chemical Corp., Wayne, NJ).[22]

A silicone polymer system was also investigated. Vinylpolydimethylsiloxane (VPDMS) resin is difunctional with a molecular weight of about 10,400 and contains a platinum catalyst (RTV6136A polymer gel; GE Silicones, Waterford, NY). Dimethyl methylhydrogenpolysiloxane (DMMHPS), which is the hydride crosslinker composing about 5–10% by weight of the second part of the RTV silicone with the remainder VPDMS (RTV6136B polymer gel; GE Silicones). DMMHPS has a molecular weight of about 10,400. Manufacturer recommendation is 1 : 1 w/w of part B to part A for forming the silicone gel. Low-viscosity (5 cP) silicone oil (SF96-5; GE Silicones) is used for viscosity control.[23]

Carbonyl iron was used for the magnetic particles in the MRPG (S-1651; ISP Technologies, Wayne, NJ), with an average particle size of about 6 μm and a spherical shape. They were
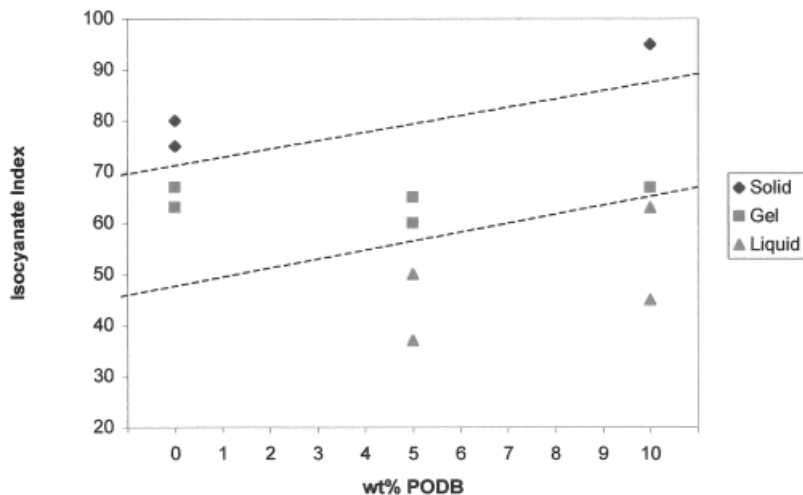
Figure 1   Polyurethane phase diagram for the PEG-600 system. Decreasing index and increasing diluent result in more liquidlike state. Increasing index and decreasing diluent result in more solidlike state.[22]

dispersed in the silicone polymer at a ratio of 1 : 1 by weight.

Instrumentation

For thermal analysis differential scanning calorimetry (DSC) was used. The Pyris 1 DSC (Perkin Elmer Cetus Instruments, Norwalk, CT) was used to measure the heat flow relative to that of a reference. Temperature scans ranging from 0 to 190°C were performed. From the heat flow data gathered, the heat of reaction, conversion, and kinetic constants can be evaluated. Analysis was
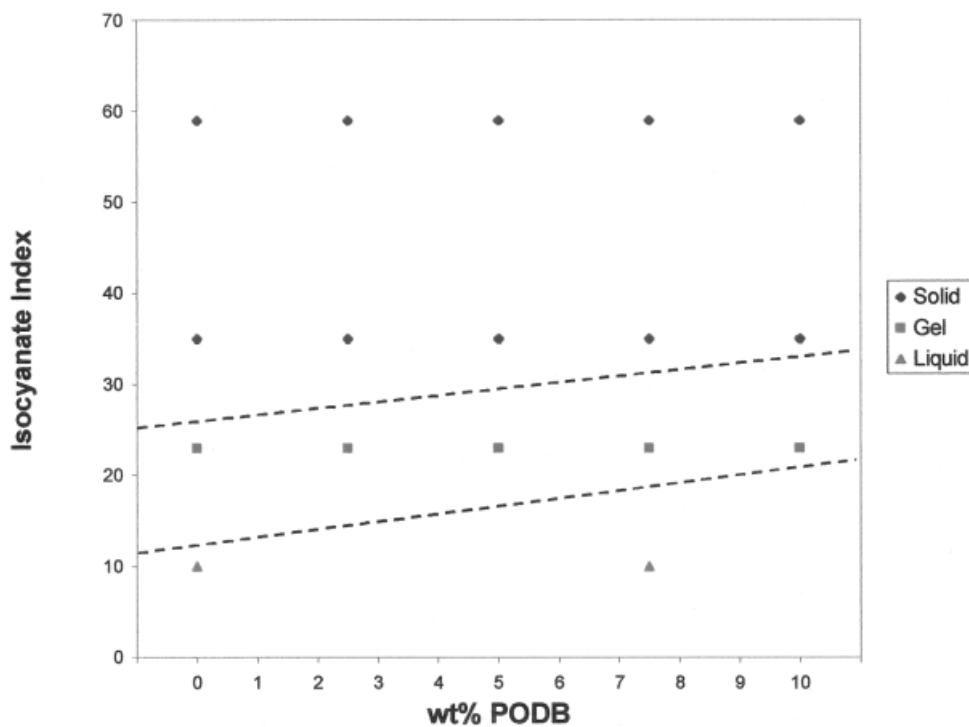

Figure 2   Polyurethane phase diagram for the polyether polyol system. Gel formation occurs close to an isocyanate index of 25.
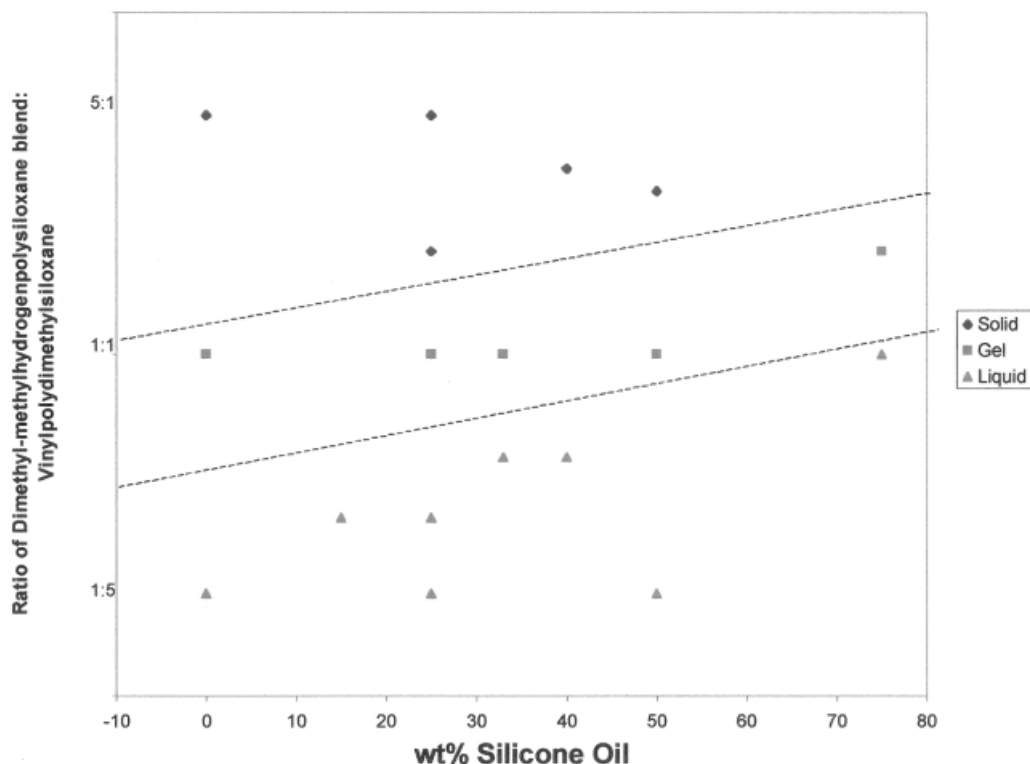
Figure 3 Silicone phase diagram. Decreasing index and increasing diluent result in more liquidlike state. Increasing index and decreasing diluent result in more solidlike state. Most gel formation is near 1 : 1 by weight ratio of the DMMHPS/VPDMS component to the VPMDS/catalyst component.

performed on the polyether polyol/p-MDI polyurethane system.

Procedure

For the polyurethane system, PODB was added to the polyether polyol. The p-MDI (cooled to about 10°C) was then added. The components were then mixed thoroughly. For DSC studies, the sample was placed into the pan and weighed immediately after mixing. Cure was complete after about 6 h at room temperature.

For the silicone system, silicone oil was added to the DMMHPS, after which VPDMS was added. If the system would contain iron, it should be added before thorough mixing. Complete cure took place in about 12 h at room temperature.

RESULTS AND DISCUSSION

Phase Diagrams

After reaction the polymers were categorized as behaving as solid, gel, or liquid. Samples exhibit-

ing properties of an elastic solid were identified as solid-state behavior in the phase diagram. The liquid was characterized by viscous and freely flowing behavior. The gel has intermediate properties between the solid and liquid states. The dashed lines in Figures 1 to 3 have a positive slope that represents how, as the stoichiometric ratio is increased; the material remains liquid at higher diluent concentrations.

Phase Diagram: Polyurethane Systems

By controlling the composition of the polyurethane using the three components described in the Experimental section, the polyurethanes vary from a viscous liquid to a solidlike gel to an elastic solid. For a larger isocyanate index (the isocyanate index is the molar equivalent ratio of isocyanate to polyol), a greater degree of crosslinking occurs. With this increase, the polyurethane becomes more viscous. In the case of the PEG-600 system, shown in Figure 1,[22] an index less than 45–55 typically results in a liquid. For an index greater than 70, the material is solid. Gels form
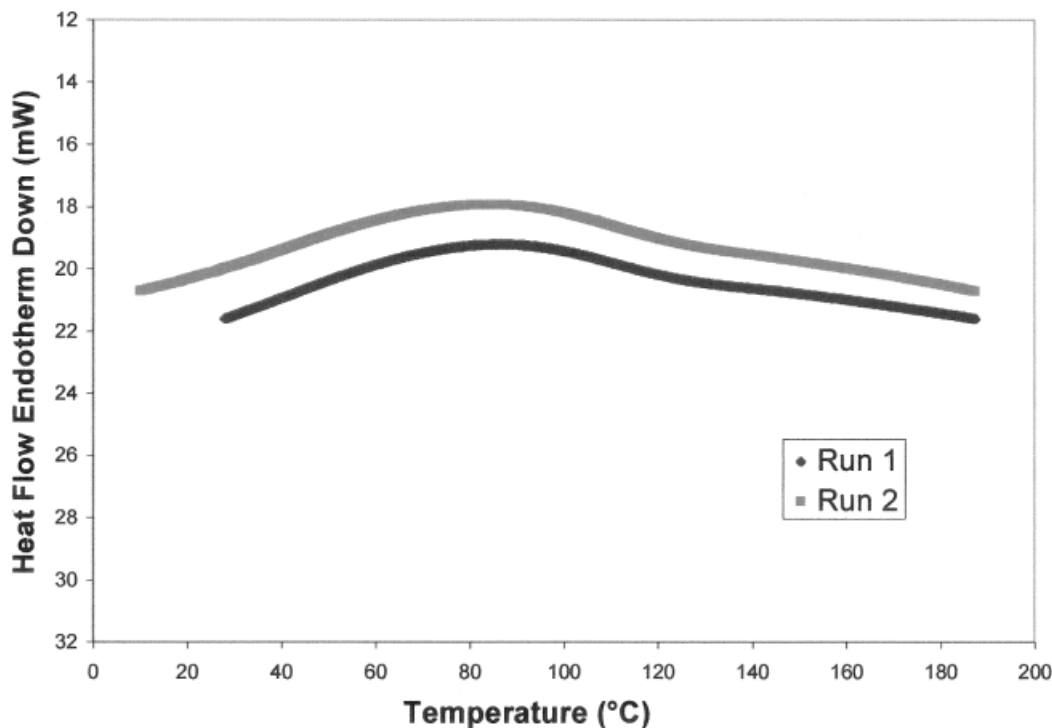
Figure 4 Thermogram of polyether polyol/pMDI system. Reaction without catalyst results in broad peak over the temperature range. Two runs shown are both for Ca° = 1.05 mol/L at a heating rate of 5°C/min with no PODB.

between these indices as shown in the figure. Figure 2 shows the phase diagram for the polyether polyol system. In this system, without plasticizer, the gel region is at an isocyanate index of about 15–25, with liquids below an isocyanate index of about 10–15. Two samples sets were run for the isocyanate indices at both 0 and 7.5% PODB with consistent results. Iron particles were not used in preparation of the polyurethane gels.

Phase Diagram: Silicone System

A silicone polymer is composed of a resin and a crosslinker and diluted by silicone oil. Altering the ratio of the resin to the crosslinker and the percentage of silicone oil forms polymer gels. As can be seen by Figure 3, at low silicone oil levels, a large crosslinker-to-resin ratio (greater than 1 : 1) will produce a rubbery solid, whereas a low crosslinker-to-resin ratio (less than 1 : 5) will produce a viscous liquid. The formation of a gel at a 1 : 1 ratio with no diluent is consistent with the manufacturer's recommendations (GE Silicones). At a high content of silicone oil (e.g., >70%), the material remains a viscous liquid up to a nearly 1 : 1 ratio and forms a gel at higher ratios. The

dashed lines again reflect where the phase should change with a change in crosslinker/resin ratio or diluent concentration.

For the same ratios, iron particles are suspended in the silicone polymers. The presence of iron particles in a similar set of samples did not qualitatively alter the rheology of the gels from what is shown in Figure 3.

Reaction Kinetics

From the DSC heat flow data, the heat of reaction can be found directly through integrating under the heat flow–temperature curve. By assuming that the heat flow is proportional to the conversion, the fraction of the area at any given point is the fraction of conversion. From this, the method of initial rates can be used to find the order of reaction. Kissinger's method is employed to find the activation energy and preexponential constant.

To assess the order of reaction, a least-squares linear regression is completed on data using the initial rates method. In this method, we assume the reaction is represented by

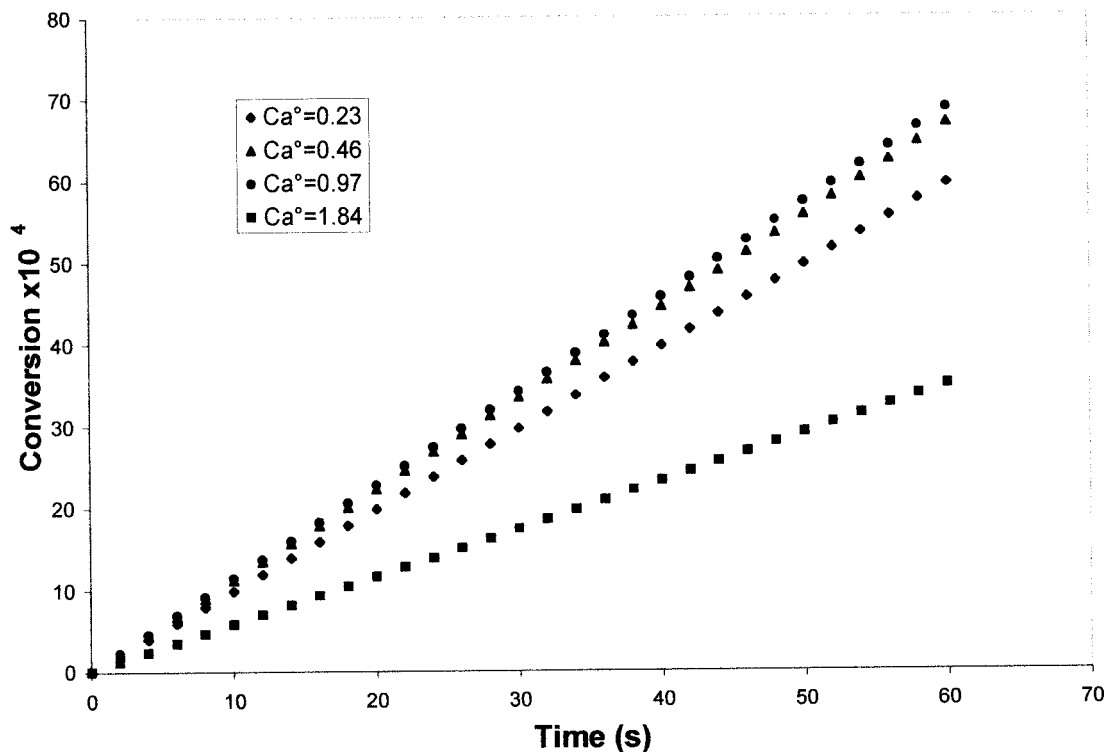$$- dC_A/dt = - r_A = kC_A^{\alpha}C_B^{\beta} \qquad (1)$$

Figure 5 Conversion of polyurethane with 7.5% PODB for 10°C/min. Using the assumption of proportionality between conversion and fraction of heat released in reaction, conversion is found as a function of time. The values shown are for the initial reaction for different starting concentrations.

where k is the rate constant, C_A is the concentration of isocyanate, C_B is the concentration of polyol, α is the order of reaction with respect to A, and β is the order of reaction with respect to B. Initial values are designated with a superior degree symbol ("°"). Thus initially,

$$(- dC_A/dt)° = - r_A° = k(C_A°)^\alpha(C_B°)^\beta \qquad (2)$$

Taking the natural log of this equation linearizes it, and by performing experiments at different initial concentrations, different initial rates are found. The data may then be regressed to find the most suitable values for the parameters α, β, and $\ln(k)$.

Once the parameters are found, the order of reaction is determined. This is then used to find E_a and A from the Kissinger method. The Kissinger method is used to find kinetic properties by varying the heating rate for each experiment. Activation energy is found by

$$E_a = mR \qquad (3)$$

where R is the gas constant and m is the slope of the line found by plotting $-\ln(\phi/T_{\max}^2)$ versus $1/T_{\max}$; ϕ is the heating rate; and $T_{\max}$ is the peak temperature of the reaction. The preexponential rate constant is found by

$$A = [\phi E_a/(RT_{\max}^2)]/[e^{-Ea/(RT_{\max})}n(1 - \alpha_{\max})^{n-1}] \qquad (4)$$

where N is the order of reaction and $\alpha_{\max}$ is the conversion at the peak temperature. This constant yields units of inverse time for an Arrhenius-type rate constant.

Two systems were studied, one without dilution by PODB and the other with 7.5% PODB. Neither formulation contained iron.

Figure 4 shows the thermogram for two runs at $C_A° = 1.05$ mol/L at a heating rate of 5°C/min with no PODB. It is assumed that all the heat evolved is attributed to the reaction and thus conversion is proportional to the area under this curve. The first run shows a peak exotherm at 86.0°C and the second run shows a peak exotherm at 84.6°C, for a difference of 1.7%.

Table I Initial Concentrations and Reaction Rates for pMDI/Polyether Polyol Polyurethane System without PODB and Diluted by 7.5% PODB

No PODB			7.5% PODB		
C_A° (mol/L)	C_B° (mol/L)	$-dC_A^\circ/dt$	C_A°(mol/L)	C_B° (mol/L)	$-dC_A^\circ/dt$
0.25	1.39	2.38E-05	0.23	1.28	2.28E-05
0.50	1.28	6.32E-05	0.46	1.18	5.12E-05
1.05	1.05	7.83E-05	0.97	0.97	1.11E-04
2.00	0.66	4.57E-04	1.84	0.58	1.07E-04
3.00	0.24	4.51E-05			

The first experimental set was performed at different initial concentrations to find the order of reaction. The concentration of isocyanate ranged from 0.25 to 3.00 mol/L. Figure 5 shows a conversion versus time graph. This slope increases as the initial concentration increases until the stoichiometric concentration is passed, after which it decreases again. This suggests that the rate is best when the two components are near stoichiometric values, given that the rate is lowest as the reactions take place furthest from stoichiometric.

The initial rate was found for each run by numerically differentiating the concentration with respect to time. These values and the initial concentrations, shown in Table I, are regressed to find the parameters of α and β as described above. α was found to be 1.90 and β was found to be 2.10, for an overall order of 4.00.

To find the activation energy and the preexponential constant, the next set of experiments was performed at 1.05 mol/L isocyanate, which is stoichiometric. These were conducted at three differ-
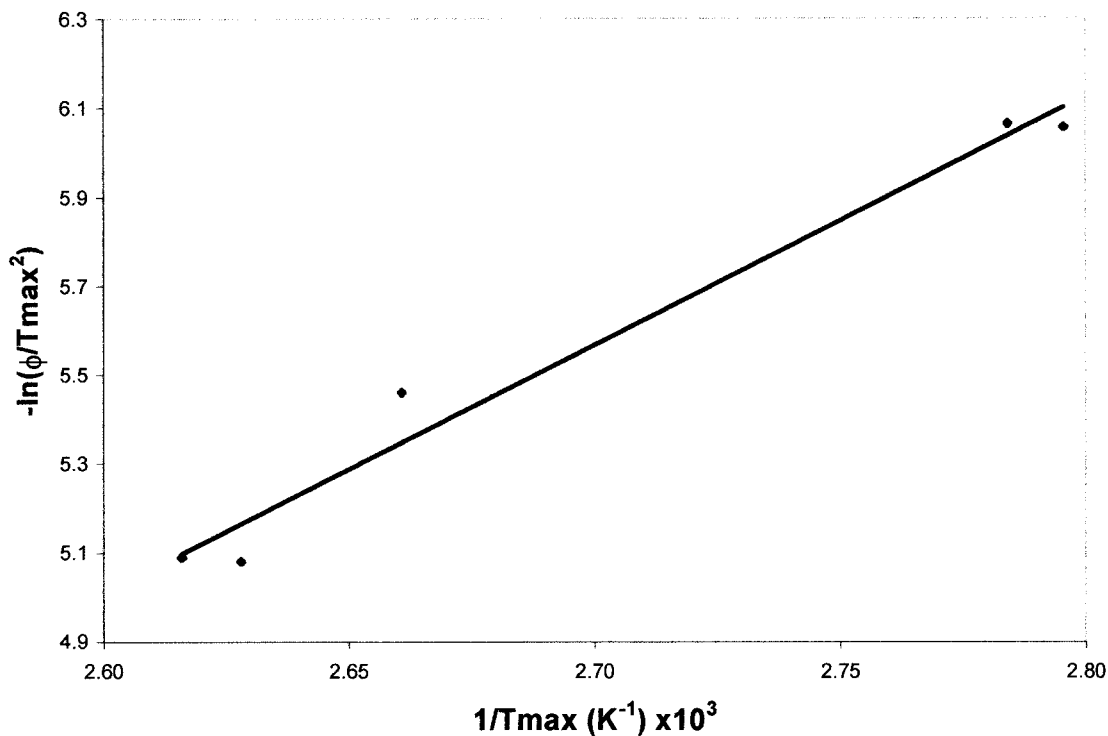

Figure 6 The Kissinger method is used to find E_a by plotting points for different heating rates as a linearized function of peak temperature for $E_a = mR$ and where the slope is m and the gas constant is R. The data shown are for Ca° = 1.05 mol/L and no PODB.

Table II   Kinetic Properties of pMDI/Polyether Polyol Polyurethane System

	Without PODB	With 7.5% PODB	Literature Values
Heat of reaction (kJ/NCO equiv)	-14.8 to -46.5	-17.0 to -42.7	-9.46 to -24.0[a] -60.3[b]
Preexponential constant	4.37×10^9 min^{-1} to 1.45×10^{10} min^{-1}	8.95×10^9 min^{-1} to 1.42×10^{10} min^{-1}	1.18×10^3 min^{-1} to 4.36×10^{10} min^{-1a} 6.28×10^9/min^{-1} to 1.31×10^{10}/min^{-1b}
Activation energy (kJ/mol)	46.5	49.0	25.5 to 64.9, average 44.8 for pMDI/polyether polyol system w/catalyst[a] 61.1 for TDI/PEA system w/o catalyst[a] 32 to 48 for TDI/polyglycol linear polyurethane system w/o catalyst[c]

[a]Marciano et al., 1982.
[b]Ferrillo et al., 1983.
[c]Krol, 1995.

ent heating rates: 5, 10, and 15°C/min. Using Kissinger's method, the activation energy was found to be 46.5 kJ/mol. The value of the preexponential constant was calculated to be, on average, 7.69×10^9 min^{-1}. Figure 6 shows the plot with slope E_a. Repeat experiments for the different rates were $\pm 2\%$. The difference was in the peak temperatures that, although close (~ 1.5 K), were not identical.

These experiments were repeated for the polyurethane above and below isocyanate index 23 and diluted with 7.5% PODB. α was found to be 1.44 and β was found to be 1.82, for an overall order of 3.26. The heat of reaction varies depending on the index. For isocyanate index 10, the heat of reaction was -127 J/g, whereas for isocyanate index 144 it was -319 J/g. Intermediate indices show intermediate heats of reaction. The activation energy calculated from Kissinger's method was 49.0 kJ/mol. The preexponential constant was calculated to be, on average, 1.16×10^{10} min^{-1}, which again agrees with literature values.

These values are compared to literature values in Table II. The values reported in the literature for the heat of reaction are close to the measured values. Little effect was noted between the diluted and undiluted systems because the heats of reaction covered almost the same range. The values for the preexponential constant and activation energy agree with literature values in both

cases. However, the effect of diluent appears to be an increase in the preexponential factor. The reaction order is greater, as calculated from data presented herein.

CONCLUSIONS

Magnetorheological polymeric gel (MRPG) systems were developed that allow qualitatively definable rheologies. This approach has been demonstrated in this study for two families of polymeric gels, polyurethanes and silicones. In all cases adjusting the ratio of reactants and the concentration of modifier (reacting or nonreacting) yielded widely alterable qualitative rheological behavior from a liquid to an intermediate gel to a solid as crosslinking increases and the diluent decreases. Kinetic characteristics of the polyurethane system were investigated. Kinetic constants were measured and the preexponential constants and activation energy values were similar to those reported in the literature.

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