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PETC FUEL RHEOLOGY LABORATORY

By
Amy C. Ekmann
James M. Ekmann

August 1984
Date Published

Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania

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PETC FUEL RHEOLOGY LABORATORY
Amy C. Ekmann and James Ekmann

I. INTRODUCTION

This document summarizes the results of investigations into the rheological properties of alternate fuel mixtures, such as coal-oil mixtures, coal-water mixtures, and coal-alcohol mixtures. Primary emphasis in this paper will be placed on the procedures and techniques used to evaluate alternate slurry fuels in the rheology laboratory at PETC through the spring of 1983. Sophisticated, bench-scale equipment was used to examine viscometric properties and to make settling velocity measurements.

The use of coal-based slurries as retrofit fuels has been suggested and researched several times over the past 100 years.^{1,2,3} Historical interest has focused on coal-oil mixtures; research was undertaken into the combustion characteristics and the flow or handling properties of these fuels. In general, no satisfactory understanding of either area was developed. Research was discontinued in every case when the driving force -- supply disruptions or large price increases for premium fuels -- disappeared.

Recently, oil shortages and the high cost of imported oil resulted in renewed interest in slurry fuel technology. Due to price considerations and advances in slurry technology, coal-water mixtures (CWMs) have superseded coal-oil mixtures (COMs) as the fuel of choice.⁴ The study of the flow characteristics of alternate slurry fuels has been greatly facilitated by the sophisticated measuring equipment now available and by a significant increase in our understanding of non-Newtonian and slurry flow properties.

Alternate slurry fuels represent a highly complex solid/liquid system in which the physical and chemical characteristics of the coal and the carrier liquid play a significant role in determining the overall fluid properties. A survey of the literature^{5,6,7} on COM before 1976 shows that a wide range of compositional parameters had been studied in an effort to develop an understanding of COM rheological properties and stability characteristics. The range of experimental methods was as broad as the range of COM characteristics. A similar situation exists for CWM, although experience with COM has allowed experimenters to narrow the selection of analytical techniques to those showing promise on that fuel. Furthermore, the nature of the liquid phase permits a more classical approach to solid/liquid interactions.

This report describes the results of studies at PETC into characterizing and understanding the flow and stability of various alternate slurry fuels. Of necessity, a significant part of any such activity must focus on testing and evaluating methods for making these measurements. Few standardized methods exist that can be applied to yield quantitative results for measurements of quantities such as viscosity, static and dynamic stability, and particle size. For coal-oil mixtures, efforts at PETC have been directed toward viscosity, static

stability, and particle size; work elsewhere has also included measurements for dynamic stability.⁸

For coal-water mixtures, a formalized effort is underway to compare laboratory methods routinely used for analysis of CWM properties.⁹ This activity includes consideration of the following measurements: solids loading, slurry viscosity, coal particle size and size distribution, composition, pH, stability, pumping characteristics, and freeze/thaw behavior. Work at PETC is focused on the following: solids loading, density, stability, viscosity, particle size and size distribution, and pH.

In all cases, procedures for testing alternate fuel characteristics and experimental conditions were chosen both to represent conditions of temperature and shear rate encountered in practice and to represent accelerated or severe tests of a particular property. Unfortunately, the definition of appropriate test conditions is not straightforward, as these laboratory studies often preceded large-scale studies designed to demonstrate the use of these fuels under practical conditions.

Examples of the data developed during this program will be given to support the choice of measurement procedures and test conditions. Furthermore, a brief discussion of the applicability of various mathematical models for settling characteristics and rheological properties will be included. Areas in which additional development of techniques is required will be discussed.

II. EQUIPMENT AND PROCEDURES

The instruments used in the PETC Fuel Rheology Laboratory have been selected to characterize physical properties of viscous coal-in-liquid slurries. The laboratory's analytical capabilities include measurement of specific gravity; of viscosity over a wide range of shear rates, shear histories, and temperatures; of coal particle size distribution; of stability; and of electrophoretic mobility of coal in aqueous slurries. These parameters are assessed routinely in evaluations of coal-oil mixtures, coal-water mixtures, and other slurry types.

A. Coal-Oil Mixtures

Many of the procedures for the use of analytical equipment have been adapted specifically for COMs. Specific gravities are measured using pycnometer bottles (either modified Hubbard-Cormick pycnometers, made of glass, or aluminum screw-type pycnometers for semifluids). The specific gravity of every coal-oil sample prepared in the laboratory is measured, not only as a measurement of the fuel density but also as a means of checking solids concentration. Mixtures are kept agitated to ensure homogeneity prior to measuring specific gravity; however, mixing must not be so violent as to entrap air. Most specific gravities are

measured at 60°C, although measurements are sometimes made over a range of temperatures.

Viscometric measurements are made using either a Haake Rotovisco* (a rotary viscometer) or a Burrell-Severs Extrusion rheometer (a capillary viscometer). Both types operate over a range of temperatures and shear rates. Both the sample and the viscometer must be heated before a test if viscosity is to be measured at an elevated temperature. Most the routine viscosity measurements are made with the Rotovisco. The laboratory has several measuring heads and several sensor systems (cups and rotors) that can be combined to measure viscosities at a range of appropriate shear rates. Measurements are possible from below 50 cp to above 20,000 cp over a range of shear rates that varies with each combination of sensors, measuring heads, and fluids. Two of the sensor systems consist of cups and rotors that are "profiled"; vertical ridges in both pieces inhibit slippage and cavitation (see Figure 1). Routinely, viscosities are measured at 60°C, although data may be taken at several temperatures between 40°C and 120°C. This type of rotary viscometer provides measurements over a continuously increasing range of shear rates, and the shear stress vs. shear rate relationship is plotted automatically on an X-Y recorder. Generally, stress is measured at shear rates from 0 to 250 sec⁻¹. On samples with very low viscosities, the maximum shear rate may be increased to as much as 1000 sec⁻¹ so that a more accurate measurement may be taken. Measurements are also made at constant shear rates against time (generally two minutes). Most COMs exhibit some degree of time-dependent, shear-thinning behavior, which is recorded clearly in the rheograms. (Examples of both types of plots are shown in Figure 2 for a base-line oil. The slight hysteresis in the stress vs. rate plot is the result of the instrument damping.) Slurry rheograms exhibit more dramatic non-Newtonian behavior, as can be seen in Figure 3. The factors used for calculating viscosities from Haake rheograms are checked every three or four months using liquid viscosity standards.

Apparent viscosities are measured at high shear rates (hundreds to thousands of reciprocal seconds) with the Burrell-Severs Extrusion Rheometer, a capillary viscometer. With this instrument, measurements are made of the mass flow rate through a capillary of known dimensions at constant temperatures and pressures. Viscosity is calculated using the mass flow rate, the slurry density, the pressure, and the capillary dimensions.¹⁰

Brookfield rotary viscometers are used less frequently in the Rheology Laboratory. Measurements are similar to those made with

*Use of brand names facilitates understanding and does not imply endorsement by the U.S. Department of Energy.

the Haake Rotovisco but are restricted to specific rates of shear and to a much smaller range of shear rates.

Coal particle size is monitored by several methods. Sieve analyses are carried out on samples of dry coal before slurry preparation; both dry and slurried coals are analyzed using Leeds and Northrup's Microtrac Particle Size Analyzer. For COMs, the Microtrac is fitted with a small-volume sample cell, which facilitates the dilution with solvents. Dilutions are made of the particles in isopropanol alone if the coal is dry, or in toluene and isopropanol if the coal is in oil. Laser light is passed through an aliquot of particles kept suspended by gentle agitation. The diffraction of the laser light is used to calculate a size distribution.¹¹ Before measurements are made, the oil is dispersed and loose agglomerates are broken up by ultrasonic mixing. The instrument reports a variety of distribution data (see Figure 4); the mean volume diameter (MV) is the value generally used if the distribution is to be represented by a single number.

Settling of the coal from a COM can be evaluated subjectively by observing the speed of separation when agitation is stopped. Quantitative measurements of COM stability are made using settling columns. These columns, made of stainless steel, are 30 inches tall and 3 inches in diameter. Two nonintrusive ultrasonic sensor systems measure density at 9 inches and at 15 inches from the column top. Thermocouples and valved sample ports are located at the column bottom, at each sensor location, and at an intermediate spot (see Figure 5). The sample ports are used to draw off aliquots of slurry from various locations; solids concentration can be calculated to check the ultrasonics, and measurements of viscosity or of particle size may be made at different locations. Columns are kept in controlled-temperature enclosures to ensure a constant (generally 60°C) temperature throughout the settling tests.

Some early tests were conducted in settling columns of a slightly different design (also shown in Figure 5). These were equipped with gamma radiation detectors for monitoring density changes. These columns were 36 inches high to accommodate the nuclear gauges. The physical differences between the two types of column made comparisons of settling data difficult; when an electronics problem necessitated a temporary shutdown of the radioactive sensors, a decision was made to eliminate these columns. Correlations between pycnometer measurements and density measurements made using both types of sensors have been satisfactory.

B. Coal-Water Mixtures and Coal-Methanol Mixtures

The water and alcohol slurries evaluated in the Rheology Laboratory are generally much higher in coal concentration than the oil slurries. Concentrations are chosen to produce mixtures appropriate for combustion in PETC's Combustion Test Facilities.

Loadings must be high enough to make up a combustible fuel, yet not so high as to produce an excessively high viscosity (a viscosity of ~500 cp at ambient temperatures has proven to be practical).

Some of the test procedures developed for the analysis of COMs are inappropriate for coal-water and coal-methanol slurries. The polarity and the low viscosity of the liquids, along with the higher coal concentrations (50% to 70% by weight), cause these slurry fuels to have properties that are very different from COM properties. Small variations in coal properties also appear to greatly affect the properties of water and alcohol slurries.

Although these slurries do settle and, like COMs, must be handled and mixed in ways that insure homogeneity prior to evaluation, short-term stability is not as serious a problem because of high solids loadings. Solids concentrations are measured simply by evaporation in a 105°C oven.

Most viscometric measurements are made using the Haake Rotovisco; the profiled sensor systems are always used, since slippage is a problem. Many slurries are highly time-dependent as well as shear-dependent, which makes interpretation of shear stress vs. shear rate rheograms less straightforward. Measurements of shear stress vs. time at several discrete rates of shear are preferred in these cases. Apparent viscosities may then be compared for points at which shear histories are identical; the Rheology Laboratory generally reports viscosity measurements that correspond to both a specific shear rate and a specific time in the measurement (e.g., "after 50 seconds at 100 sec⁻¹").

Measurements of coal-water mixture viscosities made with the capillary viscometer have only been successful for homogeneous slurries that were highly stable; phase separation or pockets of trapped air, which occur frequently, make accurate measurements impossible. It is planned to develop new procedures and to improve techniques for using this instrument with coal-water mixtures, since viscosity measurements at shear rates high enough to approximate nozzle conditions could be very useful.

Particle size measurements are made using the methods previously described for COMs; however, solvents are not necessary to disperse the coal, and the small volume sample cell is no longer required. Larger samples (10 g - 20 g slurry) are evaluated using the standard sample-mixing chamber.

Methods for the evaluation of stability in aqueous and alcoholic slurries are being developed. The settling columns and ultrasonic sensors used for COM analyses are not completely appropriate. New methods are planned that will combine measurements of stability to viscometric change with measurements of phase separation under dynamic and quiescent conditions. In the

interim, subjective evaluations of stability are made using a simple penetrometer or visual observations of settling in glass cylinders.

A Micromeritics Zeta Potential Analyzer has recently been purchased to evaluate coal-water mixtures. Research at Carnegie-Mellon University has shown that variations in electrophoretic mobility correspond to variations in slurry viscosity.¹² It is planned to use this instrument to evaluate the manner in which variations in coal characteristics may affect the properties of coal-water mixtures.

III. RESULTS

A. Selected COM Data

It has been suggested that the control of only three parameters -- particle size, temperature, and concentration -- would eliminate the need for chemical stabilization of COMs.⁶ Certainly these parameters deserve considerable study, although other physical and chemical characteristics that affect the handling and holding properties of a COM cannot be ignored. It would be helpful to be able to predict changes in slurry characteristics resulting from variations in any of the physical parameters or from the addition of various additives.

Temperature and Concentration

The effects of temperature change on COM properties are controlled to a great extent by changes in the properties of the oil. Increasing oil temperature decreases its density and its viscosity; an increase in COM temperature decreases its density and viscosity, and so decreases stability. The tendency for particles to settle more rapidly as a result of decreased viscosity and density is predicted by Stokes' law; however, the presence of many particles in a slurry makes the settling behavior much more complicated. Increases in coal concentration will increase COM density and viscosity, and settling of individual particles will be impeded by the presence of the other particles. Tests performed in the Rheology Laboratory have demonstrated the interrelated effects of temperature and concentration. For specific coal and oil combinations, mixes with concentrations ranging from 0 to 55% coal by weight have been tested at temperatures between 40°C and 80°C: specific gravity and viscosity have been measured. Viscosity relationships for some of these combinations are shown in Table 1. A graph of the viscosities of some combinations of PETC No. 6 oil* and Pittsburgh seam coal as they change with temperature is shown in

*PETC No. 6 oil is a low viscosity No. 6 oil used for many of the COM tests in the Fuel Rheology Laboratory.

Figure 6 as a further example of the pattern of these changes, which retain a similar relationship as coal and oil types are varied.

The relationships between temperature, concentration, and specific gravity for a few specific coal and oil types have been reduced to simple mathematical relationships. Examples of these equations, which are used to calculate percent solids from pycnometer data, are given in Table 2.

The effects of temperature and concentration on stability have been evaluated less extensively. The tests that were carried out supported predictions of decreased stability with increased temperature and decreased concentration. Graphed examples can be seen in Figures 7 or 8. A summary of other COM stability data can be found in the Appendix.

Particle Size

Stokes' law also predicts a settling velocity that increases as the particle diameter increases and as the number of individual particles for an equal weight percent of coal decreases. Figure 9 shows the variations in settling behavior with particle size for five sizes of Pittsburgh seam coal; the same data are plotted in Figure 10, where percent change in concentration is plotted against mean volume diameter. These data suggest, however, that there exists a minimum particle size below which further size reduction is of no practical value in enhancing stability. For the 30% mixtures of Pittsburgh coal and PETC No. 6 oil at 60°C, this value appears to be around 10 μm , although this "minimum" size would change with variations in other parameters. Testing of particle size effects has been carried out only with combinations of Pittsburgh seam coal and PETC No. 6 oil. Limited testing was carried out on COMs made of Pittsburgh coal and Gulf Low Sulfur oil,* but data were not extensive enough to demonstrate a "minimum" for effective size reduction. The stabilizing effects of reduced particle size were demonstrated.¹³

Coal and Oil Types

Variations in coal and oil types control to a great extent COM characteristics. (Comparison of data in Table 1 demonstrates the magnitude of the effects of coal and of oil.) Coal density and surface chemistry, as well as oil viscosity and chemistry, will affect the slurry behavior; however, limited attempts to correlate physical with chemical parameters in the Rheology Laboratory have not been highly successful.¹⁴

*Gulf Low Sulfur Oil is a low sulfur, high viscosity oil sent to the Rheology Laboratory as a sample for test purposes.

Additives

Additives are screened by the Fuel Rheology Laboratory for their effectiveness in COM stabilization. To facilitate comparison, tests on all additives are run under the same conditions: slurries of 30% Pittsburgh seam coal in PETC No. 6 oil are held in settling columns for a minimum of 3 days and more generally for 5-10 days. Additives are used according to manufacturers' instructions; if no instructions are provided, they are mixed with the oil prior to the addition of coal. Additives that minimized or eliminated changes in density for at least 100 hours were considered to be successful. A compilation of the additive tests (and unstabilized settling tests) is provided in the Appendix, and graphical presentations of data from some representative additive tests are given in Figures 11, 12, 13, and 14. Figures 13 and 14 include tests done using other coal types; successful additives are tested on several combinations of coal or oil to evaluate the range of their effectiveness.

B. Selected Coal-Water and Coal-Alcohol Slurry Data

The fuels evaluated in PETC's Rheology Laboratory have been prepared primarily for specific combustion tests. Although few data were collected to evaluate the overall slurry properties, there are enough data to draw some general conclusions.

Concentration and Coal Types

The properties of coal-water and coal-methanol slurries are in many ways quite similar. Both liquids have low viscosities in comparison to No. 6 oils; unless concentration is high, these slurries settle very rapidly. Coal is wetted by methanol much more readily than by water, and methanol slurries are much easier to prepare and to resuspend if settling occurs. Slurry concentration is kept as high as possible to promote stability, as well as to ensure an economical, combustible fuel. Increased concentration also raises viscosity significantly; consequently, practical concentrations are limited to a narrow range. Precise upper and lower limits of coal loading vary over a wide range with coal type. There are some coals that can be mixed into a fluid slurry at concentrations well over 70 wt%; others, particularly porous coals with high intrinsic moisture contents, have upper limits of concentration under 50 wt%. The coal's surface chemistry controls the effectiveness of the viscosity-reducing additives that are usually necessary with these mixtures. Tables 3, 4, and 5 demonstrate the differences in viscosity between various types of coal for coal-water and coal-alcohol slurries, and for coal-water slurries that contained viscosity-reducing additive.

Particle Size

For coal-water and for coal-methanol mixtures, as for COMs, viscosity and stability increase with decreased particle size. Figure 15 shows one example of the relationship between particle size and viscosity in coal-water slurries, and Table 6 demonstrates the increase in viscosity that occurs with decreased particle size for some Pittsburgh seam coal-methanol slurries.

Additives

The selection of commercial viscosity reducers that the laboratory has evaluated is not extensive; only those used for combustion testing at PETC have received detailed study. Of these, Lomar D is used most frequently (although this additive, which is a sodium salt, is not considered to be ideal for combustion). Stabilizing additives are not evaluated in the Rheology Laboratory, since the storage time for slurries prepared for combustion tests is generally short. The effects on viscosity of chemicals added to coal-water mixtures to control pH have been examined for Pittsburgh seam slurries. A graph of viscosity as a function of pH indicates that this relationship is not straightforward (see Figure 16). The research done at Carnegie Mellon University has related this type of variation in viscosity to changes in electrophoretic mobility.¹² The Zeta Potential Analyzer in the Rheology Laboratory will be used to further examine this relationship.

Aging of Pulverized Coal

Variations in the properties of laboratory slurries prepared from stored coal have indicated that slurry properties change as a function of the storage period of the pulverized coal samples. Observations suggest that the direction and the degree of change vary from sample to sample. A sample of pulverized Pittsburgh seam coal was held for about seven months and used monthly to prepare slurries. The viscosities of these slurries decreased as the coal was aged; the effectiveness of Lomar D in viscosity reduction was also reduced (see Table 7). This is probably due, at least in part, to a reduction in surface activity as oxidation occurs. Great increases in viscosity after coal storage have been observed for some other samples, and it seems clear that laboratory samples must be stored in an inert atmosphere if slurry properties are to remain consistent.

Coal-Water-Methanol Mixtures

Coal-water-methanol fuels are also used in combustion testing at PETC. When a series of 60% Pittsburgh seam coal slurries was evaluated viscometrically, it was noted that there is a large viscosity "peak," i.e., the viscosities of both coal-water and coal-methanol are increased by the addition of the second liquid.¹⁵ The shape of the slurry viscosity curve is similar, on

a greatly increased scale, to the plot of viscosity vs. percent methanol for mixtures of the pure liquids (see Figure 17). This type of peak was observed for all of the coal types that were evaluated in water-methanol mixtures (see examples in Figures 18, 19, and 20), although the peak location and the precise curve shape appear to vary from coal to coal. Mixtures of ground glass in water-methanol, prepared for comparison, also displayed a viscosity peak. Curiously, mixtures of pulverized petroleum coke, which were also burned in combustion tests in water-methanol mixtures, did not exhibit a viscosity peak (see Figure 21). The explanation for this is not clear.

IV. DISCUSSION

The development of methods for measuring important properties of alternate fuel mixtures must be based on the planned end use for any data. Measurements of flow properties and of stability (dynamic or static) for alternate fuel mixtures must relate to their use in complex engineering systems in which many parameters (such as shear rate, shear history, and temperature) cannot be defined and controlled as rigorously as they can in the laboratory. Furthermore, measurements should be of such quality that they can be used in analytical expressions applicable to design. This discussion section is separated into two parts: a summary of the strengths and weaknesses of the methods employed at PETC, and a brief discussion of the analytical and conceptual models that have been applied to flow and stability data.

A. Laboratory Methods

The rheology laboratory at PETC relies on a Haake concentric cylinder viscometer for the bulk of measurements of flow properties. With this device, one can control both the rate of shear and the duration of shear at each rate. The ridged-walled sensors allow one to compensate for fluids that exhibit slippage at the wall. This instrument cannot directly measure yield stresses.

An extrusion-type, high-shear, capillary viscometer is also available. Although this instrument cannot directly measure non-Newtonian flow properties and may experience operational difficulties when used for rapidly settling suspensions, information of some value may be obtained when comparing slurries.

To be useful, viscometric data on slurries must also be reported in terms of the instrument so that the true shear rate and shear history can be deduced from the operation principles of the device. Neither a concentric cylinder viscometer nor a capillary viscometer alone is sufficient to completely characterize the flow properties of alternate fuel mixtures. The use of both types of instruments, or comparison of data from a laboratory instrument with flow data from a flow loop is suggested.

Particle size measurements of the suspended material are made with the Leeds and Northrup Microtrac, which can use a sample taken directly from the process. The required dilution of the slurry prior to use represents a minimal amount of processing; other methods that can be employed, including wet or dry sieving, require extensive handling of the slurry. A weakness of this type of device is that it is based on fixed optics and, for the model used at PETC, on light-scattering methods appropriate only to particles above a few microns in size. For particle size distributions that are skewed to either the upper or lower end of the size range, this instrument can give erroneous readings. Furthermore, this instrument cannot account for irregular particle shapes. Correlations of particle behavior based on particle shape would require information from some form of image analysis device. Two such instruments are available to the rheology laboratory.

Measurements of static stability as performed at PETC yielded useful information for COMs; however, settling columns are of limited use when evaluating CWMs. The CWM stability may be monitored better by measuring changes in consistency or flow properties. Changes in CWM flow properties would be monitored as a function of storage time and shear history. Such a method will require development of a more sophisticated test system.

Finally, surface properties appear to control many slurry characteristics. The need for measurements of properties such as zeta potential (or electrophoretic mobility) in CWMs is indicated. Work along this line is in progress.

B. Analytical Models

Analysis of the settling and flow characteristics of alternate fuel mixtures has relied on fairly simple conceptual models drawn from other areas in which suspensions are commonplace. Early attempts to analyze stability included use of Stokes' law, hindered settling correlations, and computations based on percent change in weight at a given location or on the change in the center of gravity of a compound pendulum.^{16,5} Later attempts included analyses based on compressive settling, flow through porous media, and semiquantitative models based on principles drawn from experience with colloidal suspensions.^{17,18,19}

In general, those stability models that can readily be expressed in mathematical form cannot account for all factors influencing particle-particle and solid-liquid interactions. As the role of surface active agents and stabilizers becomes more important -- as in a coal-water mixture -- the capacity of these models to predict behavior diminishes substantially. However, those models that are based on a qualitative understanding of these interactions are difficult to express in a concise mathematical form. It appears that no model has been proposed that can be expressed in equation form (which makes it useful in an engineering sense)

while accounting for the major nonmechanical interactions occurring in alternate fuel mixtures.

Models for analyzing the viscometric properties of alternate mixture fuels are more widely available. Methods based on both suspension rheology and flow properties of homogeneous non-Newtonian fluids have been applied in a useful manner.^{20,21} Rheological models for suspensions are generally in the following form: $\eta_{red} = \eta/\eta_0 = 1 + k_1 \phi + k_2 \phi^2 + \dots$, where ϕ = solids loading by volume. These equations do predict the rapid increase in viscosity as the solid loading is increased, but they do not predict non-Newtonian behavior, such as shear thinning, unless the carrier fluid shows non-Newtonian behavior (as embodied in η_0). Models for homogeneous, non-Newtonian fluids have been used successfully for various alternate fuel mixtures.^{22,23,24} Generally, time-independent relationships are preferred, such as a pseudoplastic fluid modeled by the power law $\tau = K\sigma^n$, where τ = shear stress, σ = shear rate, and K and n are constants. Other relationships, such as a Bingham fluid or a yield pseudoplastic, have been applied. These models can predict certain aspects of the flow properties of alternate fuel mixtures, but they are not broadly applicable. The assumption of homogeneity is restrictive, and the constants in each model may change substantially for reasonably small changes in fuel composition, such as shifts in the solids loading, alteration of the particle size distribution, or addition of selected chemicals such as dispersants. These models do not have an explicit functional relationship between important characteristics of the fuel composition and the values of the rheological parameters.

Although adequate rheological models exist to express the behavior of a given alternate fuel mixture for some range of compositional and operational parameters (shear rate for example), insufficient data exist to select a form that is generally useful. The relationship between particle size distribution, the performance of additives, the solids loading, and perhaps other factors related to the nature of the coal surface must be elucidated before the current reliance on extensive laboratory analyses can be reduced.

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TABLE 1. Selected Viscosity Data for
Four Coal and No. 6 Oil Combinations

Coal Concentration Weight Percent	Temperature °C	Shear Rate* sec ⁻¹	Viscosity cPs
Pittsburgh Seam Coal and PETC No. 6 Oil			
0	50	102	58
20	50	89	106
30	50	89	158
40	50	99	346
50	50	99	1286
55	50	303	1840
0	60	102	38
20	60	89	61
30	60	98	86
40	60	84	247
50	60	117	840
55	60	251	2214
W. Kentucky Coal and PETC No. 6 Oil			
0	50	102	58
20	50	1251	90
30	50	677	165
40	50	822	389
50	50	439	1290
55	50	439	2842
0	60	102	38
20	60	52	129
30	60	99	139
40	60	99	352
50	60	103	1576
55	60	253	2201

TABLE 1. Selected Viscosity Data for
Four Coal and No. 6 Oil Combinations (Continued)

Coal Concentration Weight Percent	Temperature °C	Shear Rate* sec ⁻¹	Viscosity cPs
Pittsburgh Seam Coal and Gulf High Sulfur Oil			
0	50	80	300
20	50	94	575
30	50	94	925
40	50	94	1800
50	50	94	1985
55	50	94	4293
0	60	98	172
20	60	94	310
30	60	94	450
40	60	94	969
50	60	94	1268
55	60	94	2768
Pittsburgh Seam Coal and Gulf Low Sulfur Oil			
0	50	290	385
20	50	216	516
30	50	125	893
40	50	162	1718
50	50	167	6683
55	50	95	15520
0	60	587	191
20	60	355	314
30	60	225	496
40	60	296	943
50	60	176	3455
55	60	148	7507

* When available, viscosities are listed at similar rates of shear for comparison; even when shear rate varies, data still demonstrate the overall effects of coal and oil type, concentration, and temperature.

TABLE 2. Equations Relating Weight Percent Coal
to Specific Gravity and Temperature

PETC No. 6 Oil and Pittsburgh Seam Coal

Weight

$$\text{Percent} = (-7.522T + 885.5) \text{ SG}^2 + (15.41T - 1545.0) \text{ SG} + (-7.730T + 667.0)$$

Coal

$$\text{Specific Gravity of a 30\% COM at } 60^{\circ}\text{C} = 1.004$$

PETC No. 6 Oil and Western Kentucky Coal

Weight

$$\text{Percent} = (-3.420T + 311.6) \text{ SG}^2 + (6.151T + 312.4) \text{ SG} + (-2.830T + 17.04)$$

Coal

$$\text{Specific Gravity of a 30\% COM at } 60^{\circ}\text{C} = 1.009$$

PETC No. 6 Oil and Montana Coal

Weight

$$\text{Percent} = (0.865T - 410.1) \text{ SG}^2 + (-2.010T + 1074.0) \text{ SG} + (1.203T - 647.6)$$

Coal

$$\text{Specific Gravity of a 30\% COM at } 60^{\circ}\text{C} = 1.029$$

Gulf High Sulfur Oil and Pittsburgh Seam Coal

Weight

$$\text{Percent} = (-3.800T + 109.9) \text{ SG}^2 + (7.421T + 116.1) \text{ SG} + (-3.483T - 224.2)$$

Coal

$$\text{Specific Gravity of a 30\% COM at } 60^{\circ}\text{C} = 1.030$$

Gulf High Sulfur Oil and Montana Coal

Weight

$$\text{Percent} = (-.0317T - 378.5) \text{ SG}^2 + (-0.227T + 1067.0) \text{ SG} + (0.347T + 684.6)$$

Coal

$$\text{Specific Gravity of a 30\% COM at } 60^{\circ}\text{C} = 1.056$$

TABLE 2. Equations Relating Weight Percent Coal
to Specific Gravity and Temperature (Continued)

Gulf Low Sulfur Oil and Pittsburgh Seam Coal

Weight

Percent = $(-9.630T + 1140.0) SG^2 + (19.90T - 2060.0) SG + (-10.15T + 919.5)$
Coal

Specific Gravity of a 30% COM at 60°C = 1.046

These are examples of coal and oil combinations tested extensively in the
PETC Fuel Rheology Laboratory.

Specific gravities "SG" are at temperature T (relative to water at 25°C).

Temperature T in °F.

TABLE 3. Viscosities of Coal-Water Mixtures

Coal Type	Concentration* (wt%)	Viscosity** (cPs)	Shear Rate (sec ⁻¹)
Pittsburgh No. 2	50.0	61	200
	55.0	206	200
	60.0	767	200
"2% Ash" Kentucky Unicol	55.0	531	200
	57.5	870	200
	60.0	2640	200
"7% Ash" West Virginia Wellmore	55.0	144	600
	60.0	654	600
Texas Lignite	41.5	103	200
	46.5	439	200
	49.5	1490	200
Montana Rosebud	50.0	203	100
	52.5	288	200
	55.0	708	200

*Weight percent coal includes less than 5% inherent moisture for Pittsburgh, Kentucky, and West Virginia coals. Lignite was dried from approximately 24% moisture to less than 1%; the Montana coal was dried from approximately 28% inherent moisture. All coals are 85-95% through 200 mesh.

**Viscosity was measured after 50 seconds of shear.

TABLE 4. Viscosities of Coal-Water Mixtures
With Viscosity-Reducing Additive^a

Coal Type	Concentration ^b (wt%)	Viscosity ^c (cP)	Shear Rate (sec ⁻¹)
Pittsburgh No. 2	55.0	177	200
	60.0	295	200
	65.0	538	200
"2% Ash" Kentucky Unicol	55.0	127	200
	60.0	330	200
	62.5	475	200
"7% Ash" West Virginia Wellmore	60.0	144	400
	65.0	258	400
	68.0	957	400
Texas Lignite	46.0	221	200
	50.0	863	200
Montana Rosebud	48.0	63	200
	52.5	251	400
	55.0	298	200

^aLomar D added at 5% of total weight.

^bWeight percent coal includes less than 5% inherent moisture for Pittsburgh, Kentucky, and West Virginia coals. Lignite was dried from approximately 24% moisture to less than 1%; the Montana coal was dried from approximately 28% inherent moisture. All coals are 85-90% through 200 mesh.

^cViscosity was measured after 50 seconds of shear.

TABLE 5. Viscosities of Coal-Methanol Mixtures

Coal Type	Concentration* (wt%)	Viscosity** (cP)	Shear Rate (sec ⁻¹)
Pittsburgh No. 2	55.0	< 50	100
	60.0	354	100
	63.0	583	100
	65.0	1977	100
"7% Ash" West Virginia Wellmore	60.0	139	200
	65.0	494	200
	68.0	1888	200
Texas Lignite	44.0	162	200
	48.5	457	200
	51.0	2434	200
Montana Rosebud	45.0	46	200
	47.5	251	200
	50.0	652	200
	52.2	2360	200

* Weight percent coal includes less than 5% inherent moisture for Pittsburgh, and West Virginia coals. Lignite was dried from approximately 24% moisture to less than 1%; the Montana coal was dried from approximately 28% moisture. All coals are 85-90% through 200 mesh.

** Viscosity was measured after 50 seconds of shear.

TABLE 6. Viscosities of Coal-Methanol Mixtures Made
with Two Coal Sizes of Pittsburgh Seam Coal

	Coal Size (percent minus 200 mesh)	
	85%	93%
% Coal by Weight	Viscosity After 50 Seconds of Constant Shear	
55%	68 cp ^a	153 cp ^a
60%	354 cp ^b	380 cp ^a
63%	583 cp ^b	1932 cp ^b
65%	1977 cp ^b	c

^aShear rate of 500 sec⁻¹.

^bShear rate of 100 sec⁻¹.

^cToo viscous for accurate Rotovisco analysis.

TABLE 7. The Effects of Pulverized Coal Aging on CWM Viscosity and on Additive Effectiveness

Coal Storage	Without Additive		With 0.5% Lomar D		Additive Effectiveness (Percent Viscosity Reduction)
	Viscosity (cP)	pH	Viscosity (cP)	pH	
Freshly Pulverized	1977	4.9	502	4.9	74.6
After 28 Days	1637	4.1	631	4.4	61.3
After 56 Days	1062	4.0	811	4.6	23.6
After 84 Days	1092	3.9	435	4.4	60.2
After 112 Days	878	4.3	376	4.3	57.2
After 168 Days	914	4.0	376	4.2	58.9
After 196 Days	826	4.2	369	4.2	55.3

Slurries are 60% Pittsburgh seam coal. Viscosity measurements correspond to a shear rate of 200 sec^{-1} after 50 seconds of shear.

Figure 1. Profiled Sensor System for
Haake Rotovisco Viscometer

Figure 2. Shear stress vs. shear rate, and shear stress vs. time as plotted by a Haake Rotovisco Viscometer

BSI/4298

Figure 3. Shear stress vs. shear rate for 55% COMs as plotted by a Haake Rotovisco

BSI/4299

Figure 4. Typical Microtrac Printout

Figure 5. Schematic of COM settling columns

BSI/4302

Figure 6. Apparent viscosity vs. temperature: effect of coal concentration

Figure 7. Coal concentration as a function of time: Settling behavior in COMs affected by coal concentration

Figure 8. Percent change in concentration as a function of time: Pittsburgh Seam Coal and PETC no. 6 oil

Figure 9. Percent change in concentration as a function of time for COMs with various coal sizes

BSI/131

Figure 10. Percent drop in concentration as a function of initial mean volume diameter.

BSI/137

Figure 11. Percent change in concentration as a function of time for 30% COMs with ICI additives

Figure 12. Percent change in concentration as a function of time for 30% COMs with Diamond Shamrock Nopcosperse additives

Figure 13. Percent change in concentration as a function of time for 30% COMs with Lubrizol additive

Figure 14. Percent change in concentration as a function of time for 30% COMs with Nopco-sperse 092.

Figure 15. Viscosity as a function of particle size in coal-water mixtures

Figure 16-Viscosity of sixty percent coal - water mixtures as a function of pH

BSI/1510

Figure 17. Viscosity vs. percent methanol in water-methanol and 60% Pittsburgh coal-water-methanol mixtures.

Figure 18. Viscosity vs. percent methanol in water-methanol and 47.5% Montana Rosebud coal-water-methanol mixtures.

Figure 19. Viscosity vs. percent methanol in water-methanol and 50% lignite coal-water mixtures.

Figure 20. Viscosity vs. percent methanol in water-methanol and 56% Canadian Coal-water-methanol mixtures.

Figure 21. Viscosity vs. percent methanol in water-methanol and 50% petroleum coke-water-methanol mixtures.

APPENDIX

The following table lists, in summary form, the results of settling tests at PETC on coal-oil mixtures.

KEY

INDEX CODE identifies the run using 12-place code:

<u>Space 1</u>	U = no additive used A = additive used E = mechanically emulsified
<u>Space 2, 3</u>	Oil Type S6 = PETC #6 GH = Gulf high sulfur L1 = Gulf low sulfur - standard L2 = Gulf low sulfur NP = East coast #6
<u>Space 4, 5</u>	Coal Type PS = Pittsburgh Seam bituminous WK = Western Kentucky bituminous IL = Illinois bituminous Mn = Montana Rosebud subbituminous
<u>Space 6, 7</u>	% Coal
<u>Space 8, 9</u>	Temperature, °C
<u>Space 10, 11, 12</u>	Run Number

ADDITIVE I.D. - Name of additive as supplied to PETC; dosage; and water content.

$$\text{Percent Change} = \frac{C_o - C_{\text{upper}}}{C_o} \times 100$$

C_o = Initial Solids Concentration in Weight Percent

C_{upper} = Solids Concentration at Upper Part of Settling Column

Percent change figures are at 100 hours unless otherwise noted and labeled parenthetically.

*Starred tests showed less than 10% change in solids concentration after 100 hours.

Index Code	Additive I.D.	Percent Change	Hours
US6PS3060.028	----	24.6	100
US6PS3060.A31	----	29.2	100
US6PS4060.030	----	12.0	100
US6PS5060.017	----	(6.0)	(70)
US6PS5060.040	----	(97.0)	(1008)
US6WK3070.043	----	67.7	100
US6WK3060.080	----	79.5	100
US6MN3060.081	----	31.8	100
US6IL3060.096	----	50.9	100
UGHPS3060.020	----	34.0	100
*UGHPS4060.072	----	9.0	100
UGHWK3070.044	----	35.6	100
UGHMN4060.093	----	45.2	100
UGHIL3060.100	----	27.3	100
UL1PS3060.042	----	(83.5)	(1008)
UL1PS3060.075	----	26.0	100
UL1PS3060.092	----	29.5	100
UL1WK3070.055	----	14.9	100
UL1IL3060.097	----	23.1	100
UL1MN4060.098	----	53.5	100
UL2PS3060.018	----	28.0	100
AS6PS3070.034	0.20% BASF ES7071, 3% water	82.1	100
AS6WK3060.090	0.25% BASF ES7071, 0% water	39.5	100
*AS6PS4060.A86	0.25% BASF ES7071, 0% water	9.3	100
AS6PS3060.032	0.20% BASF ESM-14734, 3% water	33.6	100
AS6WK3060.056	0.20% BASF ESM-14734, 3% water	37.1	100
AS6PS3060.026	0.20% BASF ES7312, 3% water	35.9	100
AS6PS3060.062	0.50% BASF ES7263, 5% water	22.7	100
AS6PS3060.068	0.50% BASF ESV152756, 5% water	46.7	100
AS6PS3060.070	0.5% BASF PLURADOT NASSO, 5% water	48.0	100
AS6PS3060.071	0.5% BASF P-1000MS, 5% water	68.3	100
*AS6PS3060.019	0.15% ICI IL-540, 5% water	3.3	100

Index Code	Additive I.D.	Percent Change	Hours
AS6PS3060.033	0.15% ICI IL-540, 5% water	18.2	100
AS6PS3060.041	0.15% ICI IL-540	(100.0)	(1008)
AS6WK3070.046	0.15% ICI IL-540, 5% water	26.3	100
AGHPS3070.038	0.15% ICI IL-540, 5% water	28.3	100
ACSPC3060.105	0.15% ICI IL-1135, 5% water	(43.0)	(72)
AS6PS3060.106	0.15% ICI IL-1136, 5% water	(24.3)	(72)
AS6PS3060.109	0.5% ICI IL-1136, 3% water	(13.0)	(72)
AS6PS3060.110	0.5% ICI IL-1138, 5% water	35.6	100
AS6PS3060.107	0.5% ICI IL-1137, 5% water	(10.7)	(72)
AS6PS3060.108	0.15% ICI IL-1137, 5% water	(29.7)	(72)
AS6PS3060.159	0.25% ICI Atlas G-271	73.3	100
AS6PS3060.048	0.15% Dow Gellant	33.1	100
AS6PS3060.025	0.12% Dow XF-43130, 4.9% water	16.9	100
AS6PS4060.027	1% Imbiber Beads, 0% water	69.6	100
AS6PS4060.021	0.6% Cobey (Eagle Crusher) #1	21.7	100
AS6PS3060.022	0.6% Cobey (Eagle Crusher) #2	22.6	100
AS6PS4060.053	0.5% Monsanto MS-2046, 5% water	13.6	100
*AS6PS4060.054	0.5% Monsanto MS-2047, 5% water	6.7	100
AS6PS3060.063	0.5% Monsanto MS-2048, 5% water	10.0	100
*AS6PS3060.049	0.5% Monsanto MS-2049, 5% water	8.7	100
*AS6PS3060.064	0.5% Monsanto MS-2050, 5% water	2.0	100
*AS6PS3060.050	0.5% Monsanto MS-2051, 5% water	3.3	100
AS6PS3000.067	0.5% Monsanto MS-2051, 5% water	(100.0)	(816)
*AS6PS4060.051	0.5% Monsanto MS-2052, 5% water	7.7	100
AS6PS3060.084	0.5% Monsanto MS-2052, 5% kerosene	90.3	100
*AS6PS3070.059	0.5% Monsanto MS-2053, 5% water	0.0	100
*AS6PS4060.052	0.5% Monsanto MS-2054, 5% water	3.8	100
AS6PS3070.066	0.5% Monsanto MS-2054, 5% water	(100.0)	(816)
AS6PS3060.060	0.5% Monsanto MS-2055, 5% water	72.5	100
*AS6WK3060.099	0.5% Monsanto MS-2051, 5% water	0.0	100
AS6PS3060.160	0.25% Monsanto, 15501, 0% water	79.1	100
AS6PS3060.165	0.25% Monsanto, 15501, 3% water	45.6	100

Index Code	Additive I.D.	Percent Change	Hours
*AS6PS3060.061	0.5% Lubrizol OS-4703541A, 5% water	6.5	100
AS6PS3060.085	0.5% Lubrizol OS-4703541A, 5% kerosene	92.9	100
*AS6PS4060.101	0.25% Lubrizol OS-4703541A, 5% water	1.0	100
AS6IL3060.103	0.25% Lubrizol OS-4703541A, 5% water	38.0	100
*AS6PS3060.039	1% D. Sham. Nopcosperse 092, 0% water	1.9	100
*AS6PS4060.102	0.5% D. Sham. Nopcosperse 092, 3% water	0.0	100
*AGHPS3060.154	0.25% D. Sham. Nopcosperse 092, 3% water	0.0	100
AS6IL3060.104	0.25% D. Sham. Nopcosperse 092, 3% water	32.7	100
AS6PS3060.139	1% D. Sham. Nopcosperse KV, 0% water	36.0	100
AS6PS3060.130	1% D. Sham. Nopcosperse KV, 0% water	19.3	100
AS6PS3060.127	1% D. Sham. Nopcosperse UYV-1, 0% water	34.3	100
*AS6PS3060.146	1% D. Sham. Nopcosperse UYV-1, 0% water	4.3	100
AS6PS3060.128	1% D. Sham. Nopcosperse 100-D, 0% water	55.1	100
AS6PS3060.129	5% D. Sham Nopcosperse 54B, 0% water	34.1	100
AS6PS3060.137	5% D. Sham. Nopcosperse 54B, 0% water	19.7	100
AS6PS3060.136	5% D. Sham. Nopcosperse 54B, 0% water	36.7	100
AS6PS3060.035	1% D. Sham. Fuelsperse 055, 0% water	30.0	100.0
AS6PS3060.023	1% D. Sham. Tenlo 70, 0% water	26.7	100
AS6PS3060.117	1% D. Sham. Aluminum Octoate, 0% water	(46.7)	(72)
AS6PS3060.124	1% D. Sham. Aluminum Octoate, 0% water	73.0	100

Index Code	Additive I.D. ;	Percent Change	Hours
AS6WK3060.118	1% D. Sham. Aluminum Octoate, 0% water	(43.0)	(72)
AS6PS3060.082	0.25% D. Sham. Aluminum Octoate 0% water	73.3	100
AS6PS3060.069	0.5% Mooney 12% Cu Chemall, 0% water	55.2	100
AS6PS3060.073	0.5% Mooney 12% Fe Neonap, 0% water	48.9	100
AS6PS3060.141	0.25% Makon 4, 0% water	43.3	100
AS6PS3060.143	0.25% Makon 6, 0% water	45.3	100
AS6PS3060.161	0.25% Ethyl D, 0% water	76.0	100
AS6PS3060.167	0.25% Ethyl E, 3% water	52.4	100
ES6PS4070.036	0%, 6.5% water	11.7	100
*ES6PS4060.047	0.5% BASF ES-7071, 12% water	2.5	100
ES6PS3070.065	0.5% BASF ES-7071, 12% water	(68.5)	(816)
ES6WK3060.089	0.28% BASF ES-7071, 5.4% water	43.0	100
ES6WK3060.078	0.5% BASF ES-7071, 12% water	57.5	100
*ES6PS4060.A87	0.25% BASF ES-7071, 5% water	1.27	100
ES6PS4070.045	0.13% DOE XF4310, 3% water	27.6	100
ES6PS3060.091	0.28% Monsanto MS-2055, 5.4% water	60.8	100
ES6IL3060.095	0.28% Monsanto MS-2055, 5.4% water	52.2	100
ES6PS4060.037	0.20% Amergy 5000, 6.5% water	24.8	100
ES6PS3060.074	0.50% Amergy 5200, 12% water	69.4	100

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