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Rheology of Coal-Water Slurries Prepared by the HP Roll Mill Grinding of Coal

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September 1 - November 30, 1992

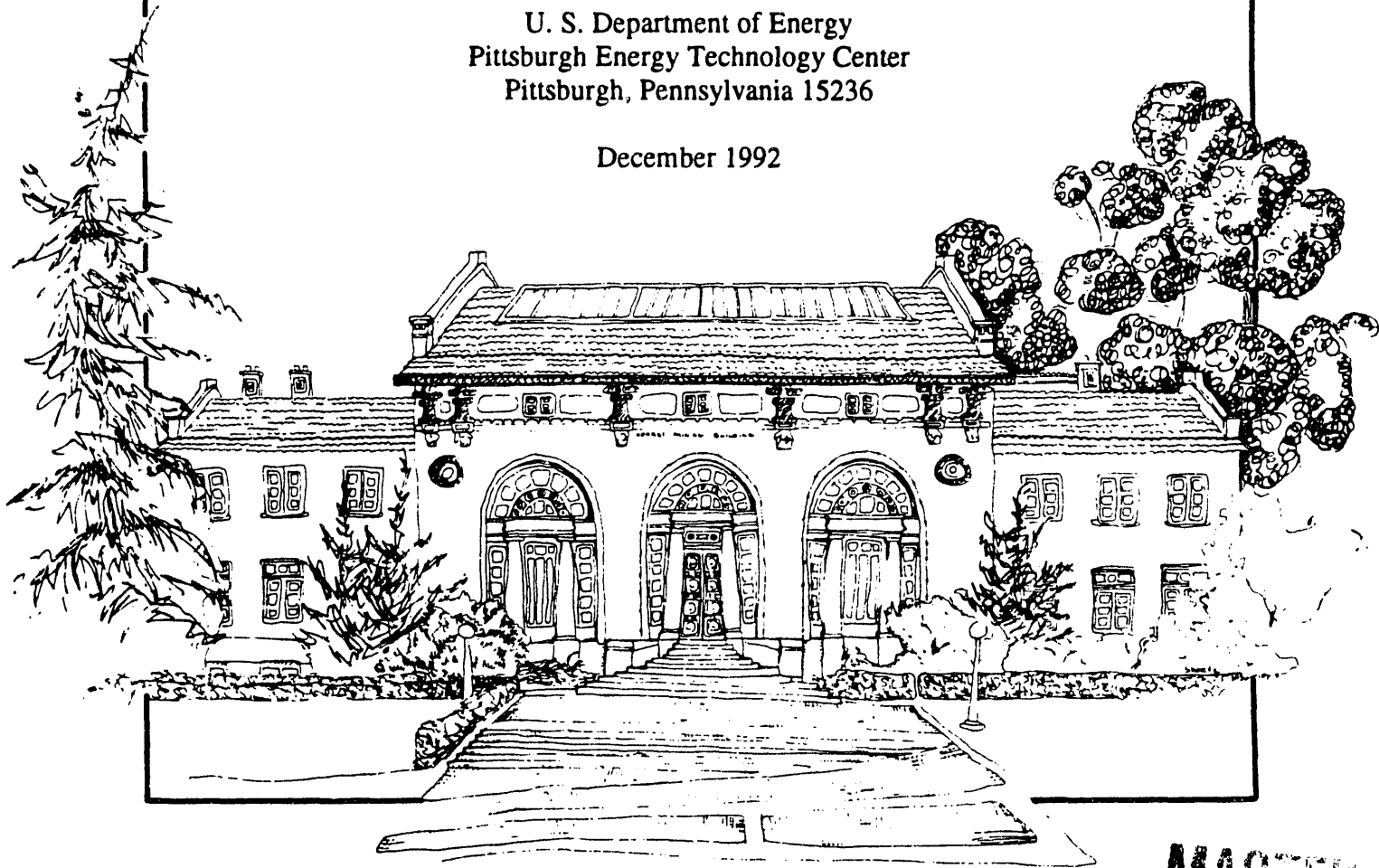
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INTRODUCTION

The objective of the research is the development of improved technology for the preparation of coal-water slurries, which have potential for replacing fuel oil in direct combustion. This is of major importance if the United States is to reduce its dependence on imported oil and rely more fully on its enormous low-cost coal resources.

The fine grinding of coal is a crucial step in the manufacture of coal-water slurries. In this context, currently available grinding mills exhibit poor energy efficiency for size reduction and non-optimum packing characteristics of the ground coal. The first increases the cost of manufacture of coal-water slurries and the second adversely affects their rheological properties. The newly invented choke-fed, high-pressure roll mill is up to 50% more energy efficient and, moreover, there are reasons to believe that it produces a size distribution of ground particles which is closer to the dense packing composition. The high-pressure roll mill (which is perhaps the only really significant innovation in industrial comminution in this century) has lower capital cost, occupies less floor space, shows negligible wear rate, accepts feed with a wide range of moisture contents and, of particular importance, it can be scaled up to grind hundreds of tons of solids per hour. The high-pressure roll mill provides a unique opportunity to develop an improved technology for preparing coal-water slurries. Our research group in the University of California at Berkeley not only has a fully instrumented, laboratory-scale, choke-fed, high-pressure roll mill (the only one of its kind in the United States) but also fully instrumented laboratory ball mills for comparative fine coal preparation purposes.

In this research program, our plans are to systematically investigate comminution energy consumption, deagglomeration procedures, and the stability and rheology of coal-water slurry fuel prepared with high-pressure roll mill, and to compare the results with slurry prepared with

ball-milled coal. The particle size distribution and packing density of the ground products will be determined. To delineate the difference in behavior of coal-water slurries prepared with ground coal produced with the high-pressure roll mill and a ball mill, the effect of coal concentration, pH, chemical additives, temperature and storage time on the viscosity, the sedimentation rate and sediment volume will be studied. Fundamental study of such surface phenomena as the zeta potential and wettability of the ground coal particles and adsorption of surfactants on coal surfaces will be carried out in order to fully understand the rheological and sedimentation behavior of slurries prepared with high-pressure roll milled coal.

The authorization date for initiating work on this project was September 1, 1992. The research work conducted during the first quarter was directed towards i) calibrating viscometry apparatus, ii) selecting a coal for initial study, iii) standardizing experimental procedures for sample preparation, grinding and rheological measurements, and iv) obtaining basic information regarding the rheological behavior and size distribution of coal ground in a ball mill under different grinding conditions. In addition, considerable effort was taken to update our literature related to this project.

PRELIMINARY COAL SAMPLE PREPARATION

In this investigation, a sample of Pittsburgh No. 8 bituminous coal is being used for the initial work. The sample, which was pre-washed, had a top size of 5 cm and a moisture content of 3.6 percent. Through a series of crushing steps in which the crushers were purged with argon gas to minimize surface oxidation of the coal, a minus 8-mesh (2360 μm) sample was prepared as feed for the subsequent fine grinding experiments. In order to obtain a coal sample with low and constant moisture content for dry grinding, the minus 8-mesh product was dried under vacuum at 40°C for 48 hours. Proximate and sulfur analyses of the minus 8-mesh Pittsburgh No. 8 coal sample showed that the material contained 49.7% fixed carbon, 40.0% volatile matter, 10.3% ash and 4.3% total sulfur. The drying step reduces the moisture content of the sample from 3.6% to 0.4%.

FINE GRINDING OF THE COAL SAMPLE

In order to compare rheological behavior of coal-water slurries prepared by the high-pressure roll mill grinding of coal with that of conventional ball milling, it is important to establish a basis for the comparison. Therefore, to begin, we first studied the grinding behavior of Pittsburgh No. 8 coal in a ball mill both under wet and dry conditions to delineate the grinding conditions required to produce suitable material for preparing coal-water slurries. Wet grinding tests were carried out with a 5 x 5.5-inch mill at 60% of critical speed, using 1/2-inch stainless steel balls as the grinding media. The pulp density in the mill was 55% solids by weight. Preliminary dry grinding tests were conducted with an 8 x 10-inch mill at 60% of critical speed. Size analyses of the ground products were carried out with a standard wet-dry sieving technique. The size distribution of the minus 200-mesh fraction was determined using a L & N Microtrac Particle Size Analyzer.

As an example, Figure 1 shows the size distribution of the products from the dry grinding of the minus 8-mesh feed in the ball mill for different time periods. After 30 minutes of grinding, the particle size is already 90 % finer than 75 μm with a mean particle size (X_{50}) of 27 μm , which is adequate for standard boiler feed. In the case of the wet grinding experiments, 32 minutes of grinding was sufficient to produce a product 90% finer than 75 μm with an X_{50} of 27 μm .

The size distributions of both the wet and dry ground products roughly follow the Gaudin-Schuhmann distribution,

$$Y = \left(\frac{X}{X_m} \right)^n \quad (1)$$

where Y is cumulative weight percent finer than size X, X_m is the size modulus, and n is the distribution modulus. The distribution parameters for these ground products can be evaluated by fitting the linear portion of the plot (cumulative percent finer from 10 to 80%) to the above equation. For the material ground wet for 32 minutes, the distribution parameters are $X_m = 78$

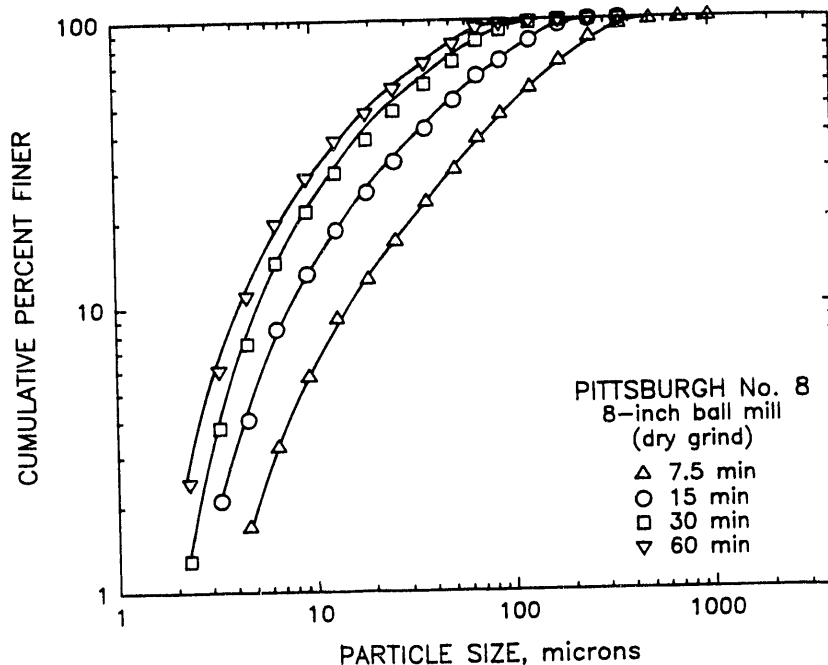


Figure 1. Size distribution of Pittsburgh No. 8 coal ground in an 8-inch ball mill under dry conditions for different times.

μm and $n = 0.75$. For the products ground dry for 30 minutes, the parameters are $X_m = 85 \mu\text{m}$ and $n = 0.60$, a value of n that is slightly larger than the 0.4 reported for maximum dense packing. Because it simplifies procedures if coal-water slurries of different solid contents are prepared with dry ground material rather than with wet ground slurries, dry grinding procedures were used to prepare the coal samples for preliminary rheological measurements in the first phase of this investigation.

RHEOLOGICAL MEASUREMENTS

The rheological behavior of coal-water slurries are being measured with a Haake viscometer with an MV II sensor system, which is suitable for characterizing non-Newtonian suspensions with viscosities in the range of 50 to 5×10^6 mPa.s. The Haake Rotovisco RV 12 consists five main parts: i) a control unit RV 12, ii) a measuring-drive-unit M 500, iii) a sensor system MV II, iv) a temperature control vessel, and v) a refrigerator bath and circulator unit A-

80. The temperature of the slurry can be controlled within $\pm 0.1^{\circ}\text{C}$. This device should permit full evaluation of the rheological behavior of the coal slurries and not just the usual single apparent viscosity.

Before measuring the rheological behavior of coal-water slurries, the Haake Rotovisco RV 12 viscometer was calibrated using a Newtonian oil (Standard E2000 supplied by Haake Instruments Inc.) with a viscosity of 1929 mPa.s at 20°C . The measured viscosity is very close to that of the standard, giving an average error of +1.7%, which is in the range of accuracy for this type of viscometer ($\pm 2\%$). In addition, glycerin (99.5+% pure) obtained from Aldrich Chemical Company, Inc. was also used to check the calibration of the equipment. At 20°C , the actual viscosity of glycerin is 1474 mPa.s, whereas the measured viscosity is 1498 mPa.s (an error of +1.6%). As expected, these results indicate that the equipment is properly calibrated and that glycerin can be used as a standard for future calibration of the Haake Rotovisco RV 12.

Preparation of coal-water slurries for preliminary rheological measurements involved the accurate weighing and mixing of the ground sample with triple-distilled water at the required solids content. The slurry was then conditioned for a given period of time at $20 \pm 1^{\circ}\text{C}$ and 210 rpm, using an Environ-Shaker 3597 from Lab-Line Instruments, Inc. In each test, 46 ml of slurry was charged to the viscometer, and the shear stress and shear rate of the coal slurries were determined in a manner similar to that used in the standard calibration procedure. Since coal-water slurries are pseudoplastic liquids with yield points (non-Newtonian-plastic liquids), the viscosity of a given slurry was calculated by graphical estimation of the slope of the flow curve at different shear rates. For coal-water slurries, some hysteresis was observed in the experiments, which resulted in different yield points. Since the sensor system is not adequate to detect such low shear stress values, only the values obtained from increasing shear rate were used for the calculations. All rheological measurements were conducted at 20°C .

To evaluate the effect of conditioning time on the rheological behavior of coal-water slurries, slurries having 60% solids content were conditioned for periods ranging from 0.5 to 48 hours, after which the rheological measurements were then conducted. Typical results are given

in Figures 2 and 3, which show that coal-water slurries exhibit a pseudoplastic rheological behavior with yield. This behavior was expected since coal particles can develop strong interactions with water due to the heterogeneous character of the coal surface. Possible interactions include double layer effects, hydrophilic interactions with polar functional groups, hydrophobic interactions with carbonaceous and hydrocarbon materials, etc.

Figure 2 presents representative flow curves for a sample of dry-ground Pittsburgh No. 8 coal for two different conditioning times. As can be seen from the plots given in this figure, for a given shear rate, the shear stress decreases as the conditioning time increases. Plots of the viscosity as a function of shear rate for two different conditioning times, given in Figure 3, show that the viscosity of the suspension decreases slightly as the shear rate increases at low shear rate and levels off at shear rates higher than 58 s^{-1} . These results are in agreement with other observations reported in the literature. Furthermore, the viscosity of the slurry decreases with conditioning time, especially at lower shear. At shear rates higher than 100 s^{-1} , the difference in the viscosities of the slurries is not significant.

The effect of particle size on the rheological behavior of Pittsburgh No. 8 coal was also studied with a slurry containing 60% solids after 16 hours of conditioning at 20°C . The results presented in Figure 4 show that at the same shear rate, the shear stress of the slurries increases as the grinding time increases. Figure 5, which presents the viscosity as a function of mean diameter of the size distribution (X_{50}) at two different shear rates, clearly indicates that the viscosity of the slurries increases as the mean diameter of the coal particles decreases, similar to other observations on the behavior of slurries.

RESEARCH WORK PLAN FOR THE NEXT QUARTER

During the second quarter, efforts to correctly characterize the rheology of coal slurries will be continued. To understand the nature of slurries prepared from dry-ground coal, studies of the immobilization of water by penetration into the pores or by interaction with oxidized functional groups on the coal surface will be carried out and correlated with coal rheology.

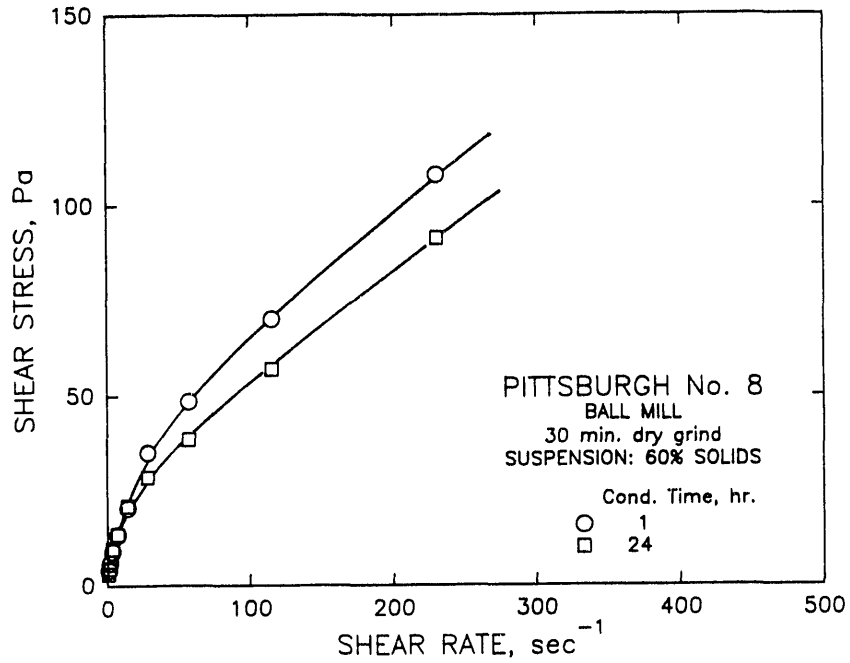


Figure 2. Effect of conditioning time on flow curve for Pittsburgh No. 8 coal dry-ground in an 8-inch ball mill for 30 minutes.

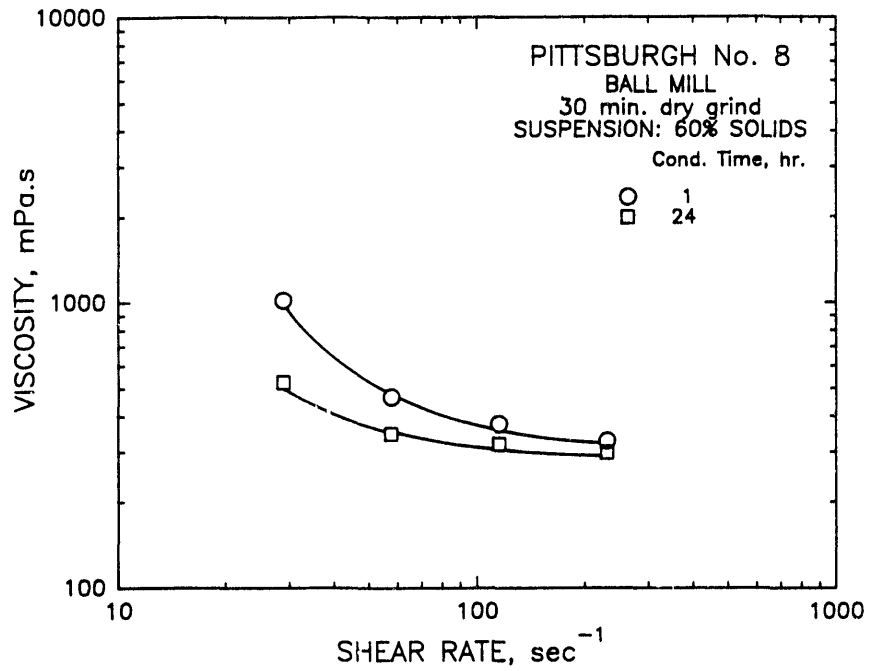


Figure 3. Effect of conditioning time on viscosity curve for Pittsburgh No. 8 coal dry-ground in an 8-inch ball mill for 30 minutes.

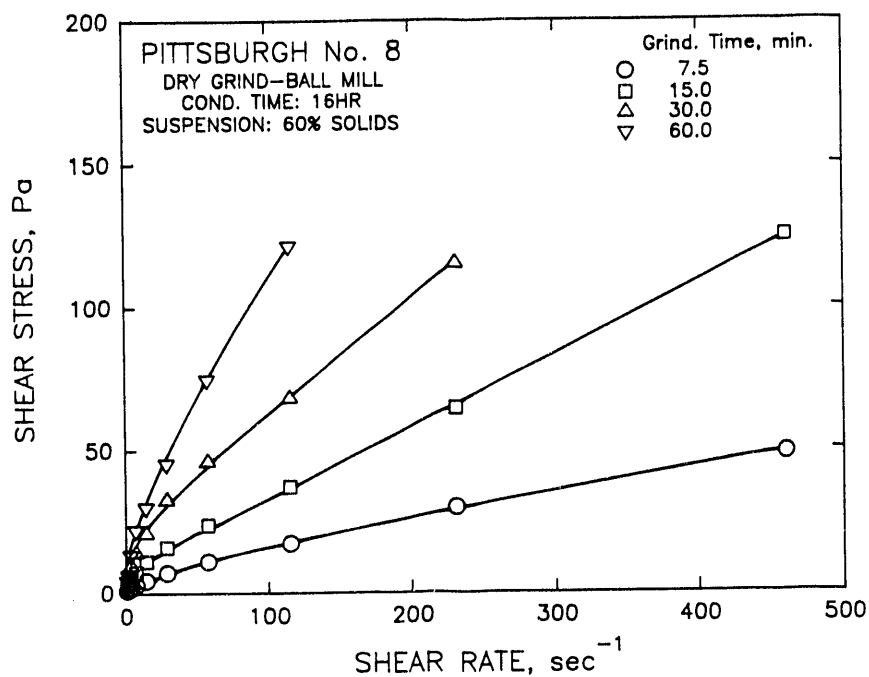


Figure 4. Flow curve for Pittsburgh No. 8 coal dry-ground in an 8-inch ball mill for different times.

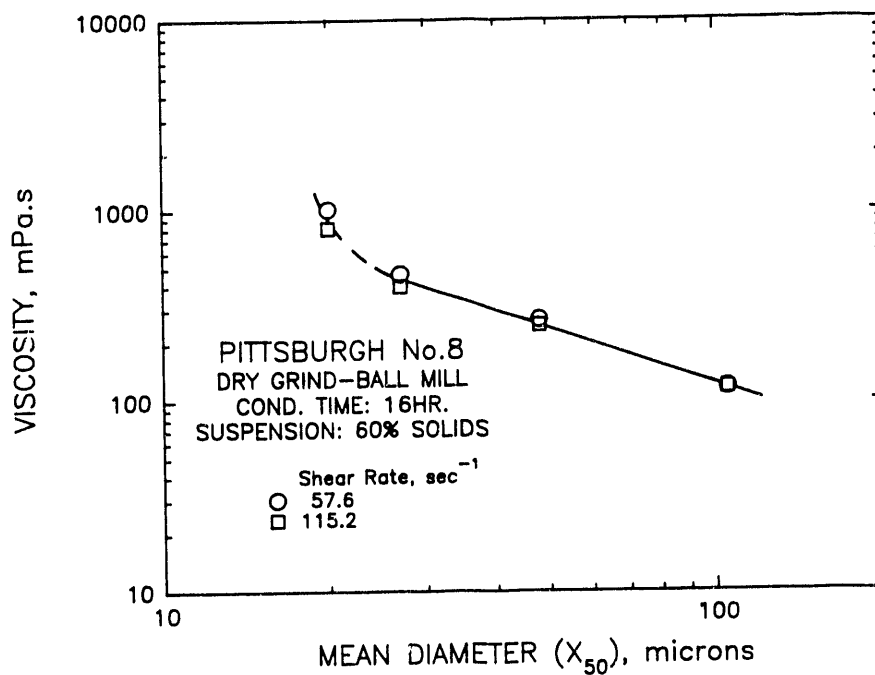


Figure 5. Viscosity of the slurries as a function of mean diameter (X_{50}) of Pittsburgh No. 8 coal at two different shear rates.

In addition, the investigation will be extended to study of the effect of such chemical additives as polyacrylate and polyethoxylated nonyl phenols (with different numbers of ethoxy groups) on the viscosity, sedimentation rate and sedimentation volume of the coal-water slurries.

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