DOE/PC/92526--T10

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

DOE Grant No. DE-FG22-92PC92526

Quarterly Technical Progress Report No. 10
December 1, 1994 - February 28, 1995

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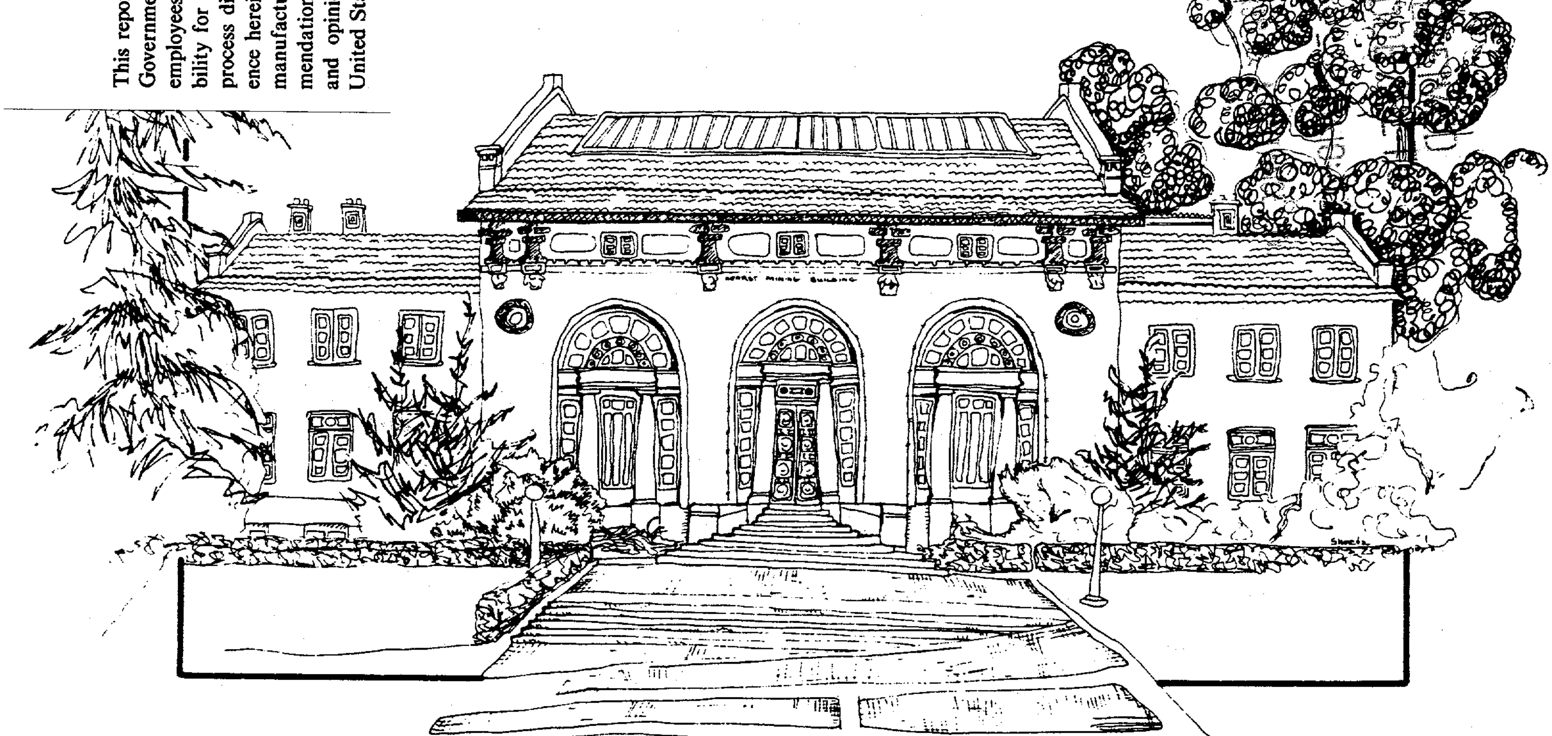
March 1995



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INTRODUCTION

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

In accordance with this objective, in the first stage of this project, considerable work was conducted to standardize experimental procedures for sample preparation, coal grinding, and rheological measurements to assure reproducibility of the experimental data. Since a Haake RV-12 viscometer with an MV-DIN sensor system was found to give the most reproducible results for measurement of slurry viscosities, it has subsequently been used for all of our rheological measurements. Methods were developed for applying the acoustophoresis technique for studying the electrokinetic behavior of concentrated coal-water suspensions. These measurements were carried out using this technique to identify the potential of chemical additives for functioning as reagents for effective dispersion. Detailed investigations of the effect of solids content and chemical additives on the rheology of coal-water slurries, prepared with fines produced by the ball milling of Pittsburgh No. 8 coal, were conducted during the first phase of our research program. These experiments were to provide a baseline against which the rheological behavior of slurries, prepared with fines produced by high-pressure roll milling or hybrid high-pressure roll mill/ball mill grinding, could be compared.

In the second stage of this project, study of the grinding behavior of coal in the high-pressure roll mill was initiated. Preliminary investigations showed that although the high-pressure roll mill grinding of Pittsburgh No. 8 coal resulted in a briquetted product, due to the plastic nature of bituminous coals, deagglomeration of the briquettes and further reduction in particle size could be achieved by grinding the roll mill product in a ball mill with modest additional energy expenditure. Our experimental results indicated that a given degree of size reduction could be achieved by hybrid high-pressure roll mill / ball mill grinding with significantly lower energy expenditure as compared to that required for grinding in a ball mill alone. Viscosity measurements showed that the rheological properties of the slurries prepared with fines produced by the hybrid grinding of coal are similar to or better than slurries prepared with fines produced by grinding coal in a ball mill only. A commercially available popular reagent used to prepare the slurries, Coal Master A-23-M from Henkel Corporation, proved to be a very efficient dispersant. Our research had shown that high-pressure roll mill grinding could result not only in potential energy savings for the production of fines but also in the preparation of slurries with improved rheological properties. However, in order to derive the maximum advantage from the use of high-pressure roll mill for fine grinding of coal, optimum operating conditions needs to be identified.

Study of the aging behavior of slurries showed a non-linear increase in their apparent viscosity over time. This increase was found to be partly due to iron released through the oxidation of pyrite contained in the coal. This was confirmed through spectroscopic studies and chemical analysis. Removal of iron by washing coal with iron-complexing reagents significantly lowered the apparent viscosity of freshly prepared coal-water slurries and slowed down the degradation of the rheological properties of the slurries, but only temporarily.

The research during this quarter was, therefore, directed towards: 1) systematic study of preparation of coal fines by high-pressure roll mill grinding and by high-pressure roll mill/ball mill hybrid grinding, 2) investigation of the rheological behavior of slurries prepared with fines produced by these techniques, and 3) study of the effect of coal cleaning on both short term and long term slurry rheology.

EFFECT OF HIGH-PRESSURE ROLL MILL GRINDING OF COAL ON THE SLURRY RHEOLOGY

High-pressure roll mill comminution of coal results in the generation of highly fractured, stressed or otherwise weakened daughter particles. However, because of the plastic nature of coal and the intense localized stresses encountered by the particles during the comminution process, high-pressure roll mill products usually come out in briquetted form. The deagglomeration of the briquettes and further reduction in particle size can be achieved by grinding the roll mill product in a ball mill with moderate additional energy expenditure. Grinding coal first in the high-pressure roll mill and then in a ball mill, a configuration we refer to as the hybrid circuit configuration, could lead to significant savings in the overall energy expenditure for comminution, as compared to that required for grinding coal to any given degree of fineness in a ball mill alone. Particle-bed comminution, as exemplified in high-pressure roll milling, also produces a characteristic packing effect on the ground products that would differ from those produced in conventional grinding mills. In particle-bed comminution, when the pressure in the bed increases, edges and corners of larger particles break off preferentially. Smaller particles produced this way fill the voids between the larger ones. If the pressure applied is high enough, this process stops only after most of the voids are filled up by smaller particles. As a consequence, interparticle crushing automatically produces a particle size distribution corresponding to near maximum packing density. On the other hand, the extent of briquetting, and the hence the energy required for subsequent deaggregation of the briquetted product, increases with the corresponding increase in the applied pressure during the high-pressure roll mill step.

Comminution experiments carried out during this past quarter were designed to delineate the effect of high-pressure roll mill grinding of coal on the rheology of coal-water slurries prepared using fines produced by subsequent ball milling of the high-pressure roll mill product. Minus 8-mesh Pittsburgh No. 8 feed was first ground in the laboratory high-pressure roll mill at applied loads of 2.0, 3.7 and 9.0 tons, which correspond to energy expenditures of

0.69, 1.17 and 2.86 kWh/t, respectively. The high-pressure roll mill product was then ground in an 8-inch stainless steel ball mill in both open-circuit and closed-circuit modes. The grinding media consisted of a distribution of 8.62 kg of 1-inch diameter steel balls, 5.18 kg of 0.75-inch steel balls and 2.85 kg of 0.5-inch steel balls (the total weight being 16.65 kg and occupying about 45% of the mill volume at rest). The mill was run at 56 rpm, which is 60% of the critical speed, with a coal charge of 500 grams. In the case of open-circuit grinding, the roll mill product was ground in the ball mill for various lengths of time, after which the ball mill discharge was dry-sieved on a 200-mesh screen using a Ro-Tap machine. The minus 200-mesh product was used to prepare our coal-water slurries. In closed-circuit tests, the high-pressure roll mill product was ground in the ball mill for a fixed length of time, after which the mill discharge was dry-screened at 200-mesh, with the minus 200-mesh being the desired product. The screen oversize is mixed with fresh high-pressure roll mill product to make up the balance of charge to the ball mill for the next grinding cycle.

In addition, minus 8-mesh feed was ground in the ball-mill alone and in both the open- and closed-circuit modes for the production of minus 200-mesh fines. The rheological characteristics of the slurries prepared from this material provide the baseline against which behavior of the slurries prepared from fines produced by the hybrid high-pressure roll mill / ball mill grinding of coal could be compared. Table 1 shows the percentage minus 200-mesh produced by ball milling

Table 1. Rate of production of minus 200-mesh fines in open-circuit ball milling of roll mill products, produced with various energy inputs in the high-pressure roll mill.

Grinding time in the ball mill, min.	Energy expended in the high-pressure roll mill, kWh/t			
	0 (ball mill only)	0.69	1.17	2.86
	percent minus 200 mesh produced			
1		18.0	20.0	23.4
2	16.5	24.8	26.0	32.0
4	29.5	36.0	36.5	44.0
8	46			

of three high-pressure roll mill products and the minus 8-mesh feed in the open-circuit mode for different ball milling times. The results indicate that the high-pressure roll mill product grinds considerably faster than the primary feed in the ball mill. In particular, an energy expenditure of 2.9 kWh/t in the high-pressure roll mill results in a product that grinds twice as fast as the primary feed in the ball mill.

The minus 200-mesh product from the open-circuit hybrid grinding tests were used to prepare coal-water slurries containing 67 wt% solids. CoalMaster A23-M was added as dispersant in each of these slurries, the dosage being 1 wt% based on the solids. Figure 1 shows the flow curves for slurries prepared with fines produced by the ball mill grinding of high-pressure roll mill products, produced at an energy expenditure of 1.2 kWh/t, for different lengths of time. The flow curves of slurries prepared with fines produced by ball milling of the three high-pressure roll mill products, for two minutes in the open-circuit mode, are compared in Figure 2. As can be seen from the plots given in Figure 1, these slurries behave like Newtonian fluids with a small yield. The small yield probably indicates a low soluble iron content of the fines. The fact that these slurries have practically identical flow curves suggests that the time of grind in the ball mill does not significantly alter the characteristics of the fines. It is apparent from Figure 2, however, that the increase in energy expended in the high-pressure roll mill results in a slight decrease in the viscosity of the slurries.

Locked-cycle grinding tests were performed with each of the high-pressure roll mill products, with 2 minutes grind time in the ball mill per grinding cycle. An additional locked-cycle test was carried out with the minus 8-mesh feed and a 4-minute grind time per cycle. The percentages of minus 200-mesh produced at steady state were 29, 20, 22 and 25 for the feed and the 0.69-kWh/t, 1.17-kWh/t and 2.86-kWh/t high-pressure roll mill products, respectively. Figure 3 shows the flow curves of slurries prepared with fines produced at steady state in these locked-cycle grinding tests. The beneficial effect of high-pressure roll milling on the rheology of coal-water slurries is clearly discernible from the figure. In general, the inverse relationship

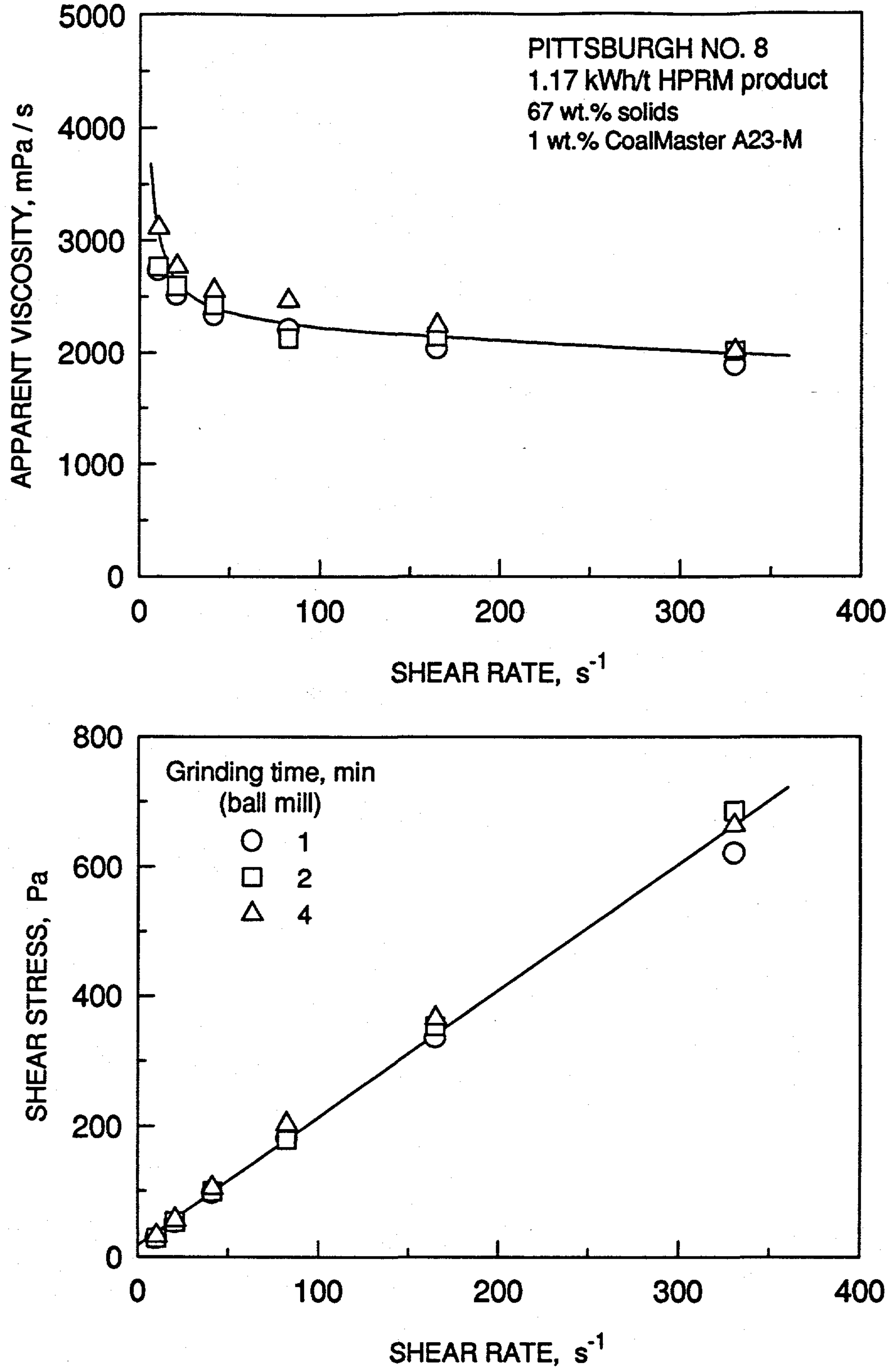

Figure 1. The effect of ball mill grind time on the viscosity and shear stress, measured as a function of shear rate, of slurries prepared with fines produced by open-loop ball milling of 1.17 kWh/t high-pressure roll mill product.

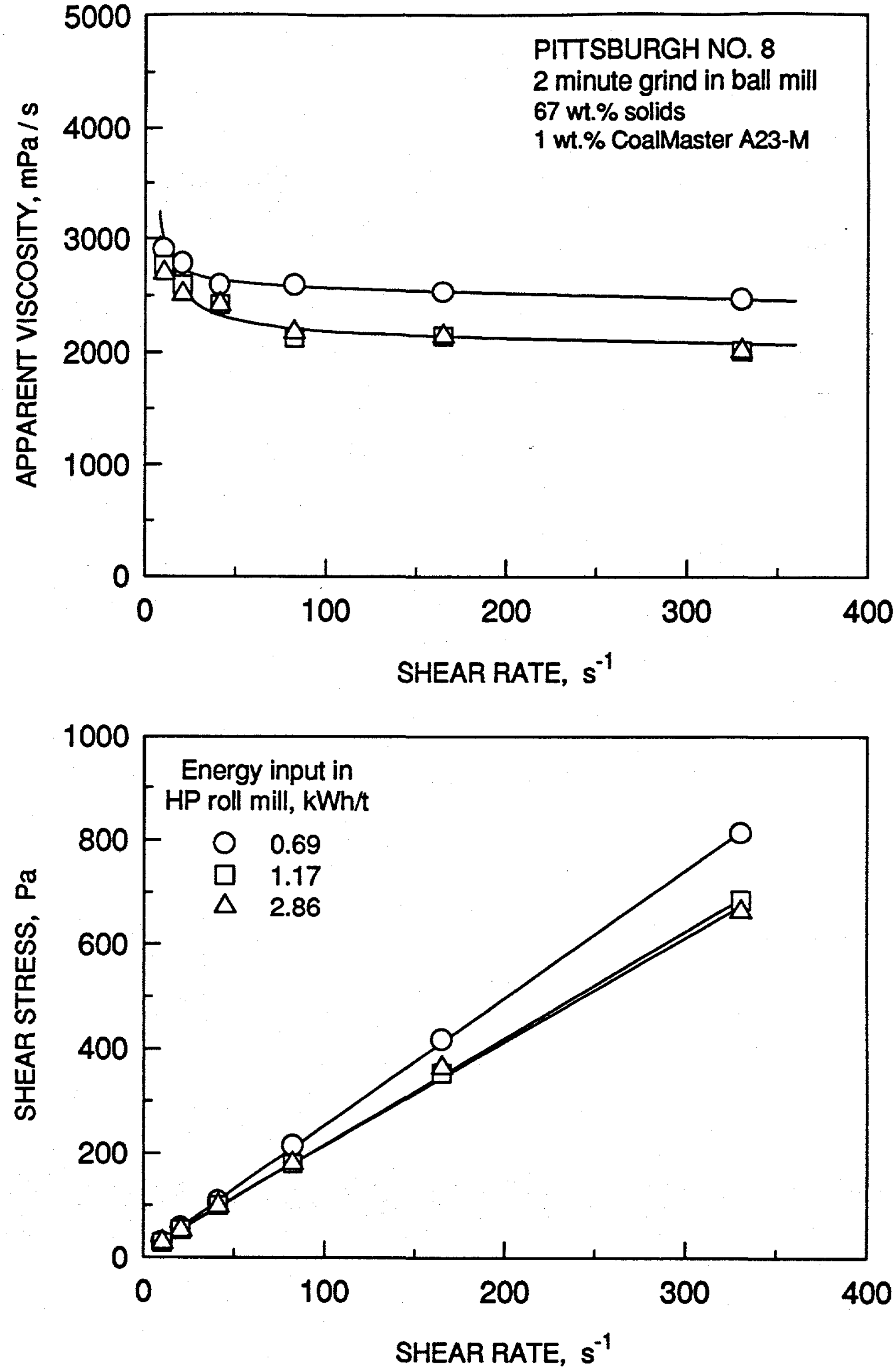

Figure 2. The effect of energy input in the high-pressure roll mill on the viscosity and shear stress, measured as a function of shear rate, of slurries prepared with fines produced by open-loop ball milling of high-pressure roll mill products for 2 minutes.

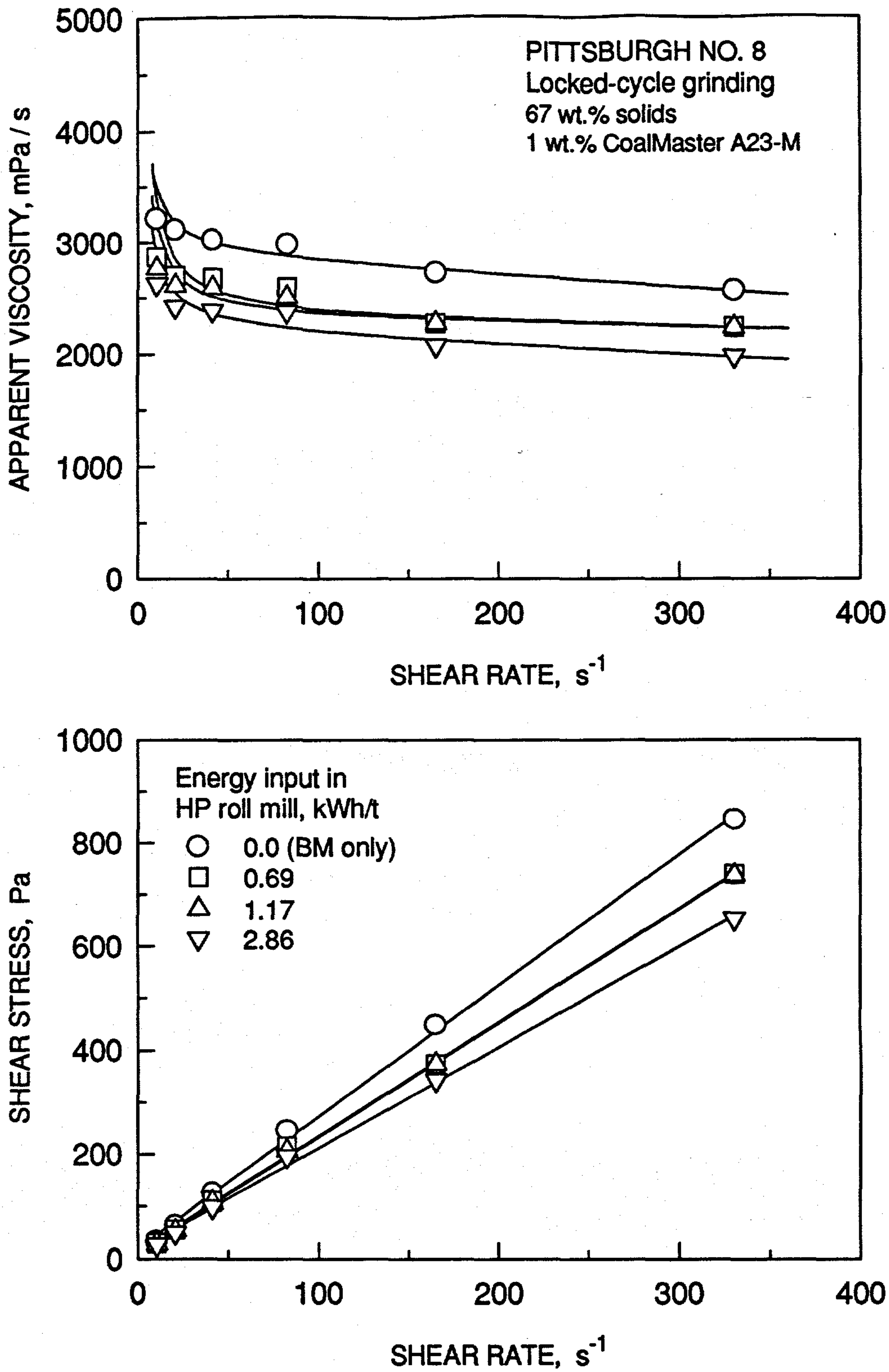

Figure 3. The effect of energy input in the high-pressure roll mill on the viscosity and shear stress, measured as a function of shear rate, of slurries prepared with fines produced by closed-loop ball milling of various high-pressure roll mill products.

between the energy expenditure in the high-pressure roll mill and the viscosity, observed earlier for slurries produced with fines generated by open-circuit hybrid grinding, holds for slurries prepared with fines produced by closed-circuit hybrid grinding.

THE EFFECT OF COAL CLEANING ON THE RHEOLOGY OF COAL - WATER SLURRIES

Our previous research had shown that the presence of soluble iron adversely affects both the short-term as well as long-term characteristics of coal-water slurries. We observed that removal of iron by washing coal with water, both in presence and absence of iron-complexing reagents, prior to preparation of slurries at least temporarily improves the aging stability of the slurries. Since pyrite contained in the coal is the source of the iron, it was thought that a partial removal of pyrite by either gravity separation or by froth flotation could slow down the degradation of the slurry rheology. Accordingly, clean coal samples were prepared by both gravity separation, using a standard laboratory Wilfley table, and by flotation.

Preparation of clean coal by gravity separation: A 2.0-kWh/t high-pressure roll mill product was ground in our 10-inch torque ball mill for one minute, essentially for deagglomerating the roll mill product. A 25-wt% coal-water suspension was prepared from the mill discharge which was then cleaned using the Wilfley table. The concentrate from the tabling operation was dried and then ground in the 8-inch ball mill to 95% minus 200-mesh.

Preparation of clean coal by flotation: The same roll mill product was ground in the 8-inch ball mill to 95% minus 200-mesh. A part of the mill discharge was then floated in a 2-liter Denver cell for 3 minutes, with GH4 as collector and MIBC as frother. The flotation concentrate, after filtering and drying, constituted the clean coal sample.

The proximate analysis (dry basis) and densities of the three fine coal samples are given in Table 2. These results show that both cleaning methods result in a significant reduction in the ash content, with gravity separation resulting in removal of more than half the ash.

Table 2. Composition of as-ground and cleaned coal samples

Sample	Combustible matter	Ash	Density
Ground only	88.15	11.85	1.43
Ground & floated	91.75	8.25	1.35
Tabled & ground	94.63	5.37	1.30

Each of the fine samples was used to prepare a 67 wt% slurry, with 3 wt% CoalMaster A-23M as the dispersant. Rheology measurements on these slurries showed that cleaning of the coal results in a slight lowering of the viscosity. The benefits of cleaning are, however, more apparent if we examine the change in the combustible matter content and the volumetric solids content of the slurries as a result of cleaning. Gravity separation results in nearly 5% increase in the combustible matter content and a 3% increase in the solids content on a volume basis. The results are summarized in Table 3.

The study of the aging behavior of these slurries showed that cleaning of coal by flotation appreciably slows down degradation of the slurry. Slurries prepared with coal cleaned by gravity separation, on the other hand, exhibited slightly worse aging characteristics compared to that exhibited by the slurry prepared from coal fines that had not been subjected to any cleaning process. The apparent viscosity of these slurries as a function of slurry age is shown in Figure 4. The effect of coal cleaning on the phenomena involved in slurry aging is being further investigated.

Table 3. Effect of coal cleaning on the composition and rheology of coal-water slurries

Sample	App. viscosity at $100 s^{-1}$, mPa/s	Wt% combustible matter (CM)	Vol. % solids	Percent CM per unit volume
Ground only	2520	57.2	58.6	71.7
Ground & floated	2340	59.9	60.1	72.5
Tabled & ground	2390	62.1	61.0	73.5

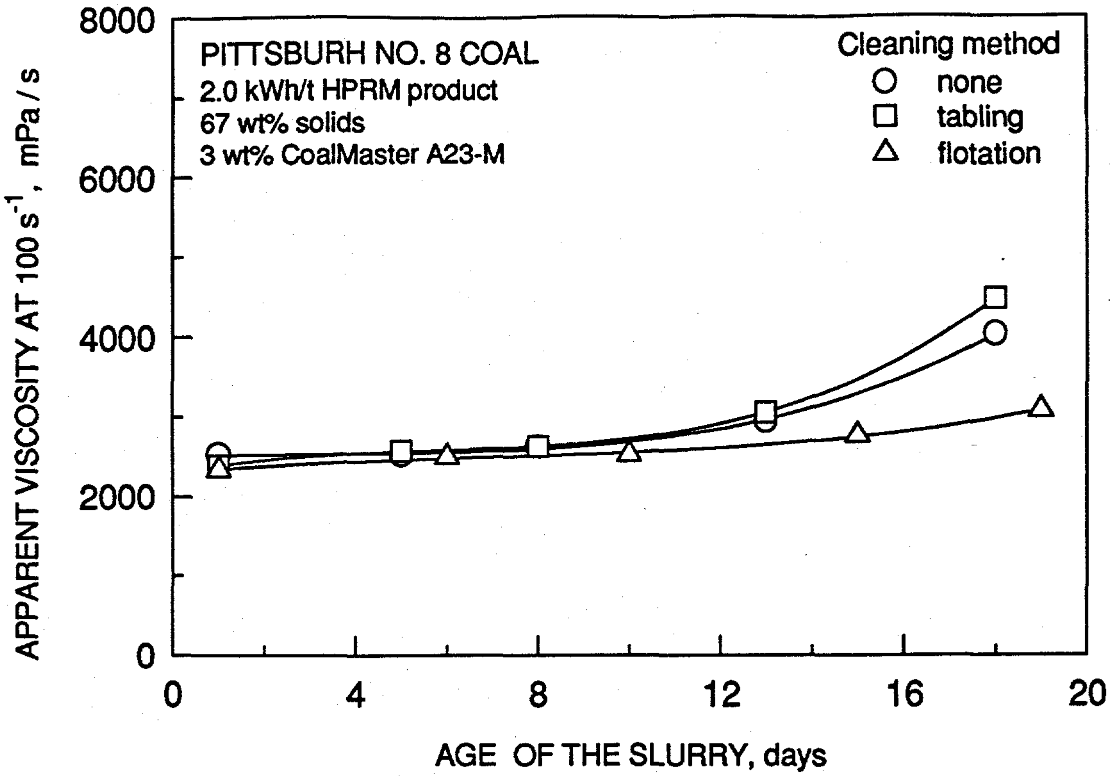

Figure 4. The effect of coal cleaning on the aging behavior of coal-water slurries.

RESEARCH WORK PLAN FOR THE NEXT QUARTER

More work on coal cleaning for the removal of iron will be performed during the next quarter. This will require gravity separation and coal flotation. The influence of pH and presence of stabilizer (xanthan gum) will be investigated. The effect of supplementary modifying reagents on the slurry rheology will be investigated in greater detail. The influence of added iron cations on the viscosity of very clean coal will be studied in order to finally prove that iron removal is the solution of our problem. More work will be done on studying long term viscosity and stability of beneficiated / cleaned coal-water slurries. Grinding studies will be carried out in high-pressure roll mill/ball mill hybrid grinding circuits to identify the effect of operating conditions and the circuit configuration on the product size distributions. Investigations will be carried out to determine the relationship between the product size distribution and the slurry rheology.