C = cathode
g = gas
l = liquid
lm = log mean

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The Rheological Characterization of Coal Liquefaction Preheater Slurries

Rheological characterizations were made for a coal liquefaction preheater slurry; measurements were made in line at high temperature and high pressure. Above 400°K, the coal-solvent slurry (35 wt. % coal) was pseudoplastic and was adequately modeled by a power-law equation. Experimental data over a temperature range of 400 to 700°K were correlated, and critical slurry velocities for transition from laminar flow were calculated for flow in several pipe diameters.

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SCOPE

The rheological characterization of coal-solvent slurries in liquefaction preheaters has received little attention because of the difficulties encountered in making measurements under severe process conditions. The usual approach in collecting rheological data on preheater slurries has been to take measurements on autoclave samples that have been reduced in pressure and temperature. The results are then extrapolated to the process temperature and pressure by means of correlations developed mainly for the petroleum industry. Few measurements have been made at actual process conditions to verify the use of such procedures. Basic rheological data are essential to ensure optimum design of the large demonstration plant preheaters presently under consideration.

The objective of the present work is to present a method for collecting rheological data for coal-solvent slurries at high pressure and high temperature. Measurements are made in line with a capillary-tube or pipeline viscometer. Experimental results and an empirical data correlation are presented for slurries at high pressure and at several temperatures. These results are expected to be particularly helpful in establishing the effects of rheology on heat transfer correlations and in providing basic data for proper multiphase flow regime mapping of the preheating process.

CONCLUSIONS AND SIGNIFICANCE

Rheological characterizations of a coal-solvent slurry containing 35 wt. % coal were made at a pressure of 13.9 MPa and at temperatures ranging from 400 to 700°K. Rheograms (shear-stress versus shear-rate plots) of the slurry at four different temperatures are shown in Figure 7. An empirical correlation between temperature-corrected shear stress and $8V/D$ was developed that allowed correlation of the data with a standard deviation of 3.6%. Critical velocities for the change of flow type from laminar to transitional were calculated for the slurry in

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pipes of various diameters.

A method and approach for obtaining rheological data on preheater coal slurries at elevated temperature and pressure have been demonstrated. Data from the pipeline viscometer used under severe process conditions of high temperature, high pressure, and particle-laden slurry were found to correlate well in a manner useful for scale-up of preheater design. The slurry behaved as a Newtonian fluid up to 400°K. As the temperature was increased to 700°K, the slurry became strongly pseudoplastic. The power-law model sufficiently characterized the slurry over the range of shear rates tested (100 to 600 s^{-1}). Both the consistency factor (k) and the flow behavior index (n) were found to be highly temperature dependent, apparently as a result of chemical reactions occurring during heating in this range. From the critical velocity analysis and the projected design velocities, flow in the projected large-diameter demonstration-plant preheaters will be either transitional or turbulent, which should enhance heat transfer significantly.

INTRODUCTION

In most coal liquefaction processes, oil- or gas-fired preheaters are used to heat the coal-solvent slurries to about 725°K. During the heating process, coal dissolution and chemical reactions occur that cause significant changes in the rheological properties of the slurries. These changes and their causes are neither well defined nor understood, and few investigations have been made to characterize them. Most previous work has concentrated on batch autoclave experiments, in which the slurries are heated and pressurized for specified time periods and then are reduced in pressure and temperature to permit rheological measurements. The results are then extrapolated to the high temperatures and pressures typical of preheating process conditions (temperatures up to 750°K and pressures of 11 to 21 MPa). Such extrapolations are useful in predicting trends, but are inadequate for large-scale preheater design. The present investigation was initiated to measure the rheological properties of the coal-solvent slurries in line at process conditions and to develop both rheological and heat transfer correlations for use in future design.

PREVIOUS RESEARCH

Extensive tests on coal-solvent slurries of the type under consideration today were conducted by the Pittsburg and Midway Coal Mining Company (Wright et al., 1974). These tests consisted of a series of batch autoclave experiments in which the slurries were brought up to preheating process temperatures and pressures. Viscosity measurements were taken with several different types of viscometers, depending on the conditions of the particular test. A Dynatrol vibrational viscometer was used to make continuous measurements for tests run over a limited range of temperatures and pressures. Cannon-Fenske and Brookfield viscometers were used to measure viscosities of samples that had been reduced in pressure and temperature. Similar procedures with Brookfield viscometers were followed by Guin and Tarrer (1977) and by Traeger et al. (1979). The autoclave tests generally showed a pronounced slurry thickening, or gelation, with corresponding high viscosities in the temperature range of 525 to 675°K.

Battelle Columbus Laboratories developed a high-temperature (730°K), high-pressure (28 MPa) moving cylinder viscometer (Droege et al., 1977). The Battelle viscometer, a batch charge unit, was first used to measure viscosities of Synthoil products. Droege and Chauhan (1978) also used the Battelle unit to study the rheological properties of coal-Solvent Refined Coal (SRC) mixtures. Battelle is presently under contract to Amoco Oil Company to measure rheological properties of coal slurry samples to be delivered from the H-coal pilot plant (Vasalos et al., 1978).

On-line rheological data at process conditions have been very limited. Pittsburg and Midway Coal Mining Company (1979) reports rheological characterizations for the feed slurries of the SRC II pilot plant at Fort Lewis, Washington. These measurements were taken with an on-line capillary tube or pipeline viscometer. The slurries, which contain recycle product, were found to exhibit pseudoplastic behavior. Although these tests were run in line, they were not conducted at the higher temperature and pressure conditions of the preheater. Exxon (1977) contracted the development of a high-temperature, high-pressure rotating cylinder viscometer capable of either batch or on-line use. Although many operational problems have been encountered with the unit, it has been extensively modified, and some data have been reported for both modes of operation (Exxon 1978 and 1979) for Exxon Donor Solvent (EDS) feed slurry and vacuum tower bottoms.

EXPERIMENTAL APPARATUS

A coal liquids flow system (CLFS), shown schematically in Figure 1, was designed to permit study of both rheological and other physical properties of coal liquefaction process streams and was used for the experimentation. Slurry feed, which was continuously mixed by an air-driven stirrer, was recirculated with a Moyno pump. A slip stream from the feed recirculation line was directed to a high-pressure, positive-displacement Milroyal pump, which fed slurry to the preheater at a maximum rate of 1.1×10^{-6} m^3/s. The feed tank weight was continuously monitored with a strain gauge weigh cell and digital output indicator. The preheater was 12 m long and consisted of 2.8-mm ID stainless steel tubing bent in a serpentine coil of seven horizontal runs. High-temperature electrical resistance heating tape was wrapped around the tubing and was electrically controlled via thermocouples attached to the outside wall of the preheater. The maximum

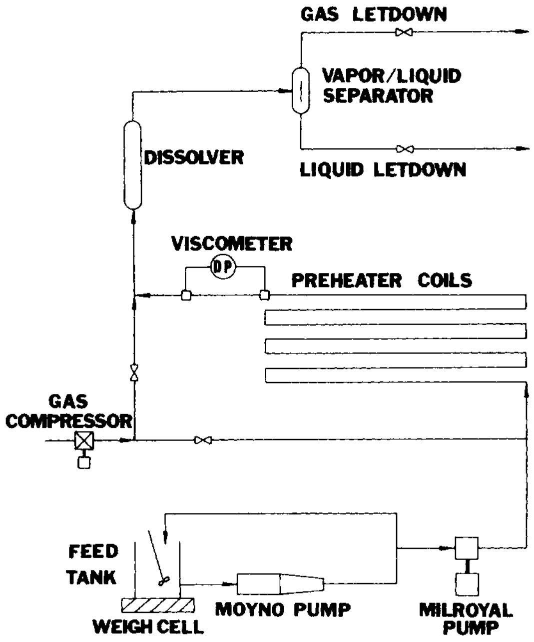

Figure 1. Schematic flow diagram of coal liquids flow system.

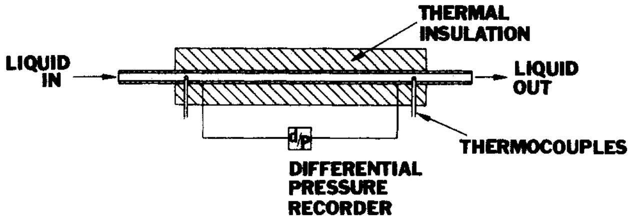

Figure 2. Schematic flow diagram of pipeline viscometer.

heat flux to the unit was 2.2 × 10^4 J/s·m^2. A capillary-tube or pipeline viscometer of the same internal diameter as the preheater (0.61 m long) was located immediately downstream from the preheater. Figure 2 is a schematic diagram of the viscometer, which was equipped with a differential pressure cell and two Chromel-Alumel thermocouples. The pressure cell was used to measure the pressure drop across the viscometer, and the thermocouples were used to measure the inlet and outlet slurry (or liquid) temperatures.

Located downstream from the viscometer were a dissolver, a vapor-liquid separator, and gas and liquid letdown valves. The dissolver had a capacity of 0.6 × 10^{-3} m^3, which provided for a slurry residence time of about 10 to 20 min, depending upon the slurry flow rate. The system pressurizing gas, usually helium, was injected into the dissolver downstream from the preheater and viscometer; thus, the slurry phase alone passed through the preheater and viscometer. However, the CLFS was valved so that gas could be admixed with the slurry before entering the preheater. Some tests using hydrogen in the combined-phase mode of flow have been conducted and are reported by Oswald et al. (1979).

EXPERIMENTAL PROCEDURE

A coal-solvent slurry consisting of 35 wt. % Illinois No. 6 coal and process recycle solvent was used for all experimentation. The coal was dried, pulverized, and screened to −170 mesh before being mixed with the solvent. An ultimate analysis of the coal is given in Table 1. The solvent was obtained from Southern Company Services' solvent-refined coal pilot plant, SRC I, in Wilsonville, Alabama. The solvent, a distillate with a boiling range of 475 to 725°K, was taken from Wilsonville's recycle stream, which was used to slurry the coal before the preheating step.

The experimental procedure consisted of the following steps. The slurry was prepared, continuously mixed in the feed tank, and recirculated, and a slip stream was fed to the preheater. The CLFS was pressurized with controlled gas back pressure, and the discharge temperature of the slurry was brought up to the desired level and allowed to stabilize. The pipeline viscometer temperature was held constant at the preheater discharge temperature. The slurry mass flow rate, the pressure drop across the viscometer, and the inlet and outlet temperatures were then recorded. This procedure was repeated at different mass flow rates but at the same preheater discharge temperature. Other series of mass flow rates were run at different discharge temperatures. Residence time was about 60 to 100 s in the preheater and about 3 to 5 s in the viscometer, depending on the flow rate.

RESULTS AND DISCUSSION

Rheological tests were conducted at four different temperatures (400, 478, 550 and 700°K). At each temperature, the pressure drop across the pipeline viscometer was measured at several different slurry flow rates. From these data, the flow curves shown in Figures 3–6 were developed. The wall shear stress ($\tau_w = D\Delta P/4L$) was determined from the pressure drop data, and the flow parameter ($8V/D$) was calculated from the slurry mass flow rate. At each temperature, the data were correlated with a least-squares linear regression.

The slope of the curve in Figure 3 has a value of unity, which indicates the slurry was Newtonian at 400°K. The flow character began to change above 400°K, however, as evidenced by the slope of 0.85 shown in Figure 4 for the 478°K slurry. The decreased slope indicates that the slurry was non-Newtonian, having taken on pseudoplastic characteristics. As the temperature was increased, the slurry became increasingly non-Newtonian, resulting in correlation slopes of 0.51 at 550°K and 0.30 at 700°K (see Figures 5 and 6). Only three data points were collected at 700°K because of

TABLE 1. ANALYSES OF ILLINOIS NO. 6 COAL

Component	Wt. %
Proximate Analysis[a]	
Volatile matter	37.6
Fixed carbon	49.3
Ash	13.1
Ultimate Analysis[b]	
Carbon	78.2
Hydrogen	5.8
Nitrogen	1.5
Sulfur	4.1
Oxygen	10.4
Higher heating value − 2.89 × 10^7 J/kg	

[a] On a moisture-free basis.
[b] On a moisture- and ash-free basis.

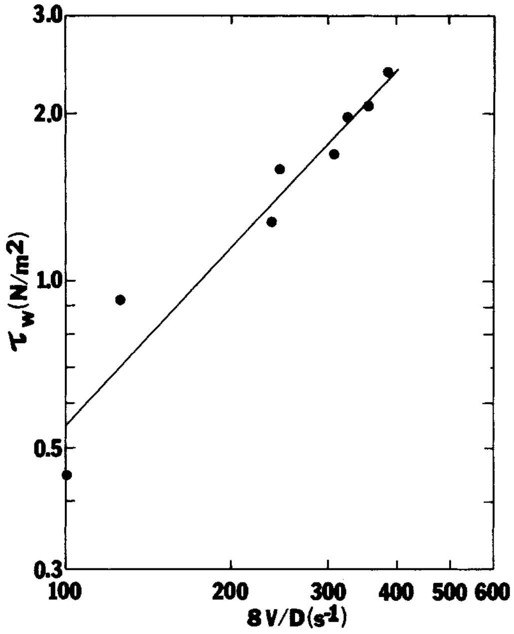

Figure 3. Flow curve for 35 wt. % coal-oil slurry at 400°K, n' = 1.00. Slurry is Newtonian.

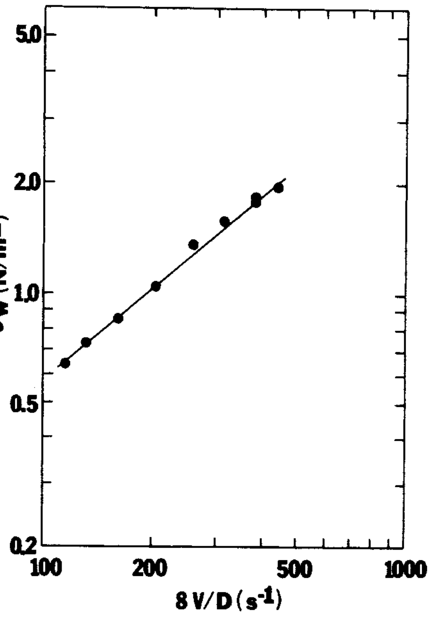

Figure 4. Flow curve for 35 wt. % coal-oil slurry at 478°K, n' = 0.85. Slurry is mildly pseudoplastic.

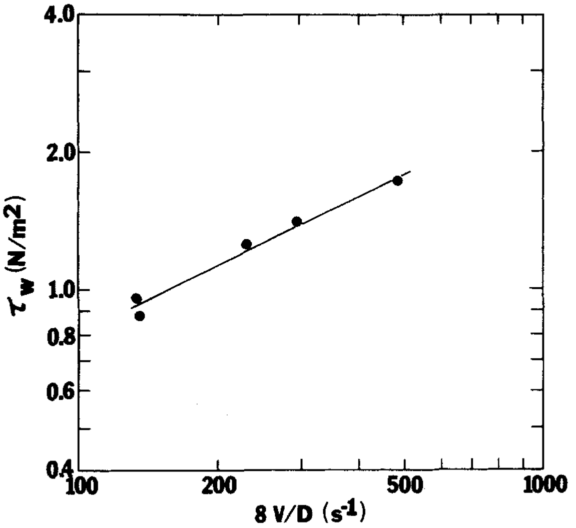

Figure 5. Flow curve for 35 wt. % coal-oil slurry at 550°K, $n' = 0.51$. Slurry is strongly pseudoplastic.

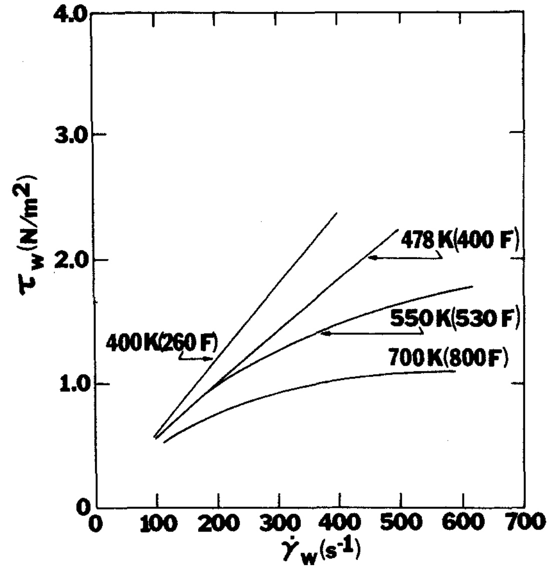

Figure 7. Rheograms for coal slurries generated from the flow curves by use of the Rabinowitsch-Mooney relation.

the difficulty of maintaining these experimental conditions. Although these points are certainly insufficient to warrant statistical correlation, they do indicate the trend in the changing behavior of the slurry.

The Rabinowitsch-Mooney relationship between $8V/D$ and the shear rate ($\dot{\gamma}$) was used to generate the rheogram plots shown in Figure 7 (Govier and Aziz 1972). From this figure, the apparent viscosity ($\tau_w/\dot{\gamma}_w$), consistent with expected behavior, is observed to decrease with increasing temperature between 400 and 550°K. The area between 570 and 670°K is known as the slurry thickening, or gelation region, in which apparent viscosity of the slurry is thought to increase substantially. Rheological determinations were not made between 550 and 700°K because of difficulties encountered in maintaining stable operation. However, an experiment was run in which the pressure drop across the viscometer was measured while the slurry flow rate was held constant and the preheater discharge temperature was varied between 500 and 750°K. Figure 8 shows the resulting plot of pressure drop versus temperature. The pressure drop, which is directly related to the apparent viscosity, was observed to increase about six-fold between 550 and 625°K. It then fell off rapidly to about its previous level. Such behavior would explain the low apparent viscosity observed in Figure 7 for the 700°K data.

As noted, above 400°K the slurry behaves as a pseudoplastic fluid. The power-law model

$$\tau = k\dot{\gamma}^n \tag{1}$$

was selected to describe the slurry behavior because the data do not warrant a more complex description. For many pseudoplastic fluids (Hanks and Christiansen, 1961), the consistency factor (k) is a function of the fluid temperature, whereas the flow index (n) is not. This is not the case with the 35-wt. % slurries. Figure 9 shows n to be a strong function of temperature.

The cause of this phenomenon is not known. It is speculated that it may arise from the swelling of the coal with increasing temperature or possibly from hydrogenation of the coal via the hydrogen donor capacity of the solvent.

Since the preheater residence time changes as the slurry feed rate is varied (to produce viscometer pressure drop-flow rate data for the rheological characterization of the slurry), the effect of preheater residence time on slurry rheology must be considered. Although no specific tests were run to quantify this effect, it appears that preheater residence time has a minimal effect on slurry rheology. By inspecting the family of flow curves (Figures 3, 4, 5 and 6), it is evident that profound changes in rheology, as evidenced by changes in slope, occur with changes in preheater discharge temperature; however, for any given flow curve, the slope does not

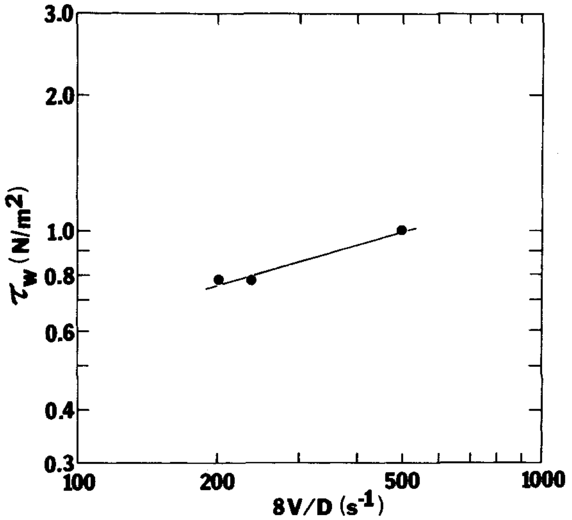

Figure 6. Flow curve for 35 wt. % coal-oil slurry at 700°K, $n' = 0.30$. slurry is strongly pseudoplastic.

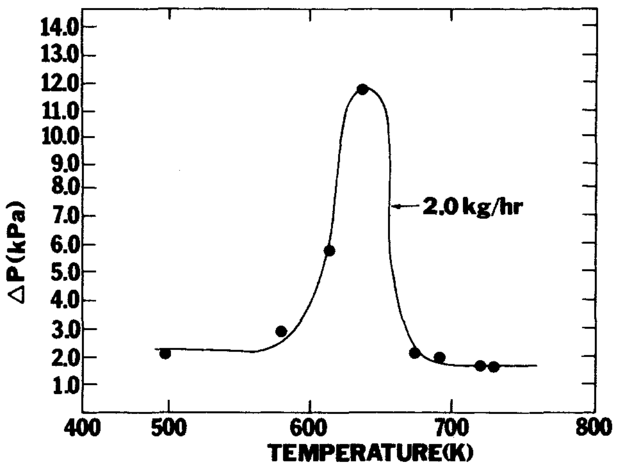

Figure 8. Pressure drop across viscometer at constant flow rate as a function of preheater temperature.

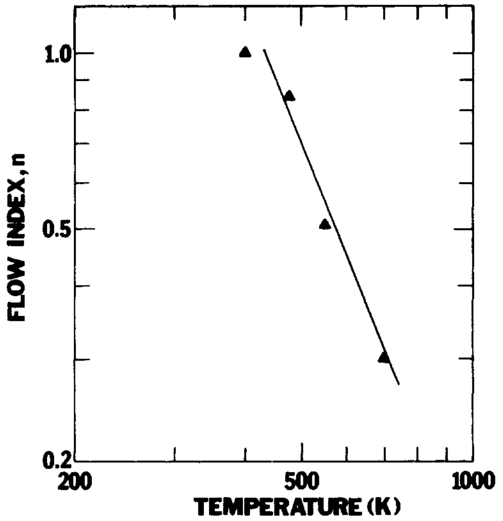

Figure 9. Flow behavior index as a function of temperature for 35 wt. % coal-oil slurries.

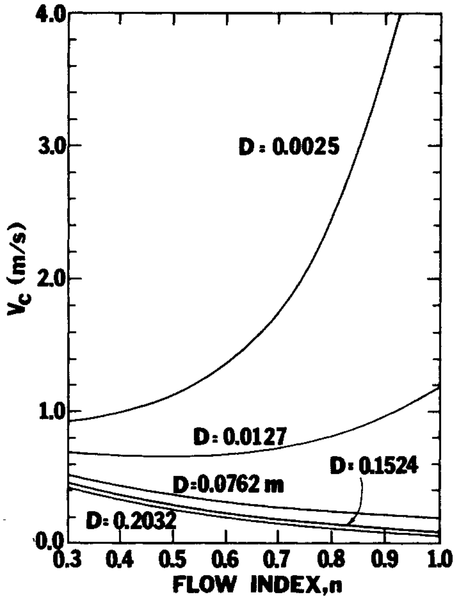

Figure 11. Predicted critical velocity of laminar-turbulent transition for 35 wt. % coal-oil slurry in pipes of various sizes as a function of flow index.

change from the shortest to the longest preheater residence time (i.e., largest value of $8V/D$ to smallest value of $8V/D$). Thus, at a constant temperature, slurry rheology is described by the same power law models constants k and n, regardless of preheater residence time. If this were not the case and slurry rheology was an appreciable function of residence time, the constant-temperature flow curves would not be straight lines. The slope would most likely decrease at a longer preheater residence time (small $8V/D$) if there was a significant increase in coal conversion.

Another possible source of variability in the data would be heterogeniety effects caused by particle settling. Any such effects were probably insignificant in these systems. The small particle size (maximum particle less than 3% of tube diameter) and viscous nature of the oil used make this slurry homogeneous.

The temperature dependencies of k and n were determined from the data taken at four different temperatures (400, 478, 550, and 700°K). The following empirical correlation was then developed:

$$f(\tau_w,\phi) = 7.95(8V/D)^{0.139}, \quad (2)$$

where $f(\tau_w,\phi)$ is a temperature-corrected function of wall shear stress given by the expression

$$f(\tau_w,\phi) = \tau^{1/\phi}\,\frac{\phi^{0.8}e^{3370/\phi T}}{[(1+0.417\phi)/(0.556\phi)]^{0.139}}, \quad (3)$$

where $\phi = (1000/T)^{2.37}$. Figure 10 shows a plot of $f(\tau_w,\phi)$ versus

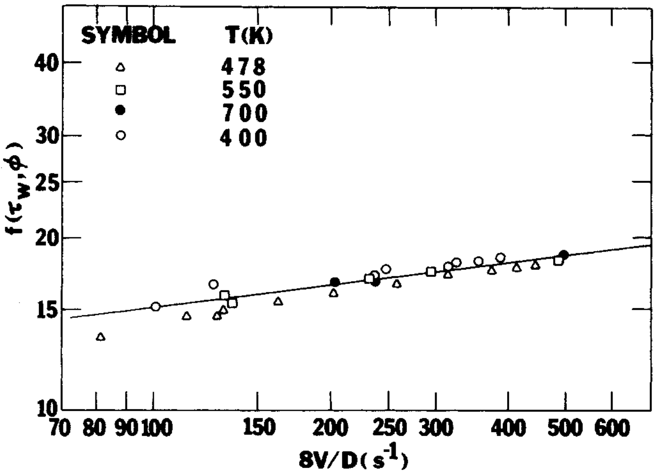

Figure 10. Correlation of temperature-corrected wall shear stress with 8V/D for 35 wt. % coal-oil slurry at four different temperatures.

8V/D. The data are correlated within a standard deviation of 3.6%.

It is emphasized that the correlation is empirical and based upon data from four experimental runs. No assurance is given that extrapolations beyond the range covered by the present data will be valid. It should be noted that although the correlation apparently covers the temperature range of 400 to 700°K, it is not necessarily expected to hold over the special range 570 to 670°K, in which gelation is thought to occur. This is because no data were obtained in this special region. Nevertheless, it is of particular interest to note that the 700°K data for slurry that has passed through the gelation phase does correlate well with the lower-temperature data. The correlation is not intended at this point to serve as a design basis, but to indicate behavior trends in the preheating process.

The power-law model and the correlation expressed by Eqs. 2 and 3 were used as a basis for analyzing the transition of the slurry from laminar to transitional and turbulent flow. A power-law Reynolds number defined by Govier and Aziz (1972),

$$Re_{PL2} = \frac{D^n V^{2-n}\rho}{k}\,8\left(\frac{n}{2+6n}\right)^n, \quad (4)$$

and a critical power-law Reynolds number (Ryan and Johnson, 1959; Hanks, 1969),

$$(Re_{PL2})_c = \frac{6464n}{[1+3n]^2[1/(2+n)]^{(2+n)/(1+n)}}, \quad (5)$$

were equated to derive critical velocities. Equations 4 and 5 were set equal to each other and solved for the velocity at which the slurry changes from laminar to transitional flow. Figure 11 shows a plot of calculated critical velocity versus the flow index. Several preheater diameters are plotted, from the small-diameter tube used in the CLFS to the 0.2-m diam. tubes under consideration for the SRC demonstration plant preheaters. The low critical velocities for the larger-diameter preheaters at all values of n are particularly interesting. Inasmuch as superficial design velocities for the large preheaters are 3 to 6 m/s, flow will be transitional or turbulent according to the curves shown in Figure 11. Figure 12 is a plot of the calculated critical velocity versus slurry temperature for various preheater diameters. For the largest diameters, the critical velocity will be ≤0.5 m/s for all temperatures encountered in the preheater. It is emphasized that Figures 11 and 12 do not reflect any slurry gelation data and that they should not be used for extrapolation outside the data range.

This experimental study and accompanying data analysis have demonstrated a workable approach to obtaining rheological data

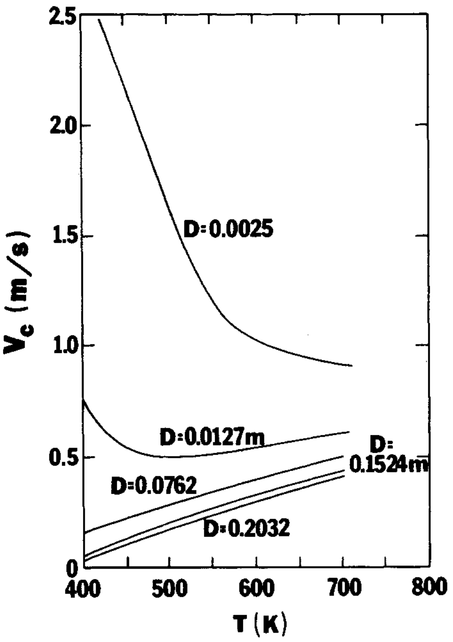

Figure 12. Predicted critical velocity of laminar-turbulent transition for 35 wt. % coal-oil slurry in pipes of various sizes as a function of slurry temperature.

for coal-solvent preheater slurries in line and at high pressures and temperatures. From the experimental and analytical results it is concluded that:

1. Preheater coal-solvent slurry (35 wt. % coal) behaves as a Newtonian fluid up to about 400°K. From 400 to 700°K, the slurry becomes increasingly pseudoplastic as the temperature is increased.
2. Over the range of shear rates tested, the power-law model is adequate for describing slurry behavior.
3. Both the consistency factor (k) and the flow behavior index (n) of the power-law equation are strongly temperature dependent.
4. Data from a pipeline viscometer used under severe conditions of high temperature, high pressure, and high slurry loading can be correlated in a manner useful for preheater scale-up.
5. The critical transition velocity for slurry in large-diameter pipes (0.15 to 0.2 m) will be less than 0.5 m/s for temperatures up to 700°K.

ACKNOWLEDGMENT

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NOTATION

D	= pipe inside diameter
k	= consistency factor
L	= pipe length
n	= flow behavior index for power-law fluids
n'	= slope of flow curve (= n for power-law fluids)
Re_{PL}	= power-law Reynolds number
$(Re_{PL})_c$	= critical power-law Reynolds number
T	= temperature
V	= slurry velocity
V_c	= critical slurry velocity
ΔP	= pressure drop across viscometer

Greek Letters

$\dot{\gamma}$	= shear rate
$\dot{\gamma}_w$	= local wall shear rate
ρ	= slurry density
τ	= shear stress
τ_w	= local wall shear stress
ϕ	= correlation parameter, $\phi = (1000/T)^{2.37}$

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