

The Effect of Pressure and Gas Composition  
on the Fluidity of Pittsburgh No. 8 Coal

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INTRODUCTION

The Gieseler method<sup>(1)</sup> of measuring the fluidity of coal has long been a parameter used by the steel industry for the evaluation of coke oven feedstocks. This analytical technique in which the rotational speed of a rabble arm stirrer held in a packed sample of ground coal is measured as it is twisted with a known torque while the sample is heated through the plastic range, is a measure of the pseudo viscosity of the molten or semi-molten coal.

The interest in recent years in the gasification of Eastern U.S. bituminous coals has led to concern over potential coking and handling problems of these coals as they are fed to the gasifier. Conoco's experience with the gasification of two highly fluid Eastern coals during the DOE sponsored Technical Support Program for the British Gas/Lurgi slagging gasifier project suggested that these coals demonstrate significantly different coking and fluidity properties under gasifier conditions than they do at standard atmospheric conditions. Hence Conoco began a program for the evaluation of gasifier feedstocks which includes examining the coking properties at simulated gasifier conditions.

As a part of this overall program Conoco has designed and built a pressurized Gieseler Plastometer in which it is possible to measure the fluidity of coals in atmospheres of any desired composition and at total pressures up to 450 psig. The initial series of tests in this apparatus, reported here, is a study of the effect of nitrogen and hydrogen pressure on the fluidity of a Pittsburgh No. 8 seam coal. Nitrogen was chosen as an "inert" gas to examine only the effect of total pressure while hydrogen - probably the most reactive gas in a gasifier - was chosen as a first approximation to a gasifier gas.

EXPERIMENTAL

Apparatus

The basic apparatus consists of a Standard Instrumentation Model P-11R research version Gieseler Plastometer with the measuring head and solder pot mounted in a pressure vessel and the associated electronics located externally. Figure 1 is a design drawing of the apparatus. The pressure vessel is fabricated from a 30" long, 10" dia schedule 40 pipe, capped at the bottom and flanged at the top. A solder pot rests on a perforated steel plate at the bottom and the measuring head and crucible assembly are suspended from the top flange. A sight gauge is provided to insure proper alignment during the lowering of the crucible into the solder pot.

Electrical leads are passed into the pressure shell through conax pressure seal fittings. The vessel is pressurized through a half inch inlet port and the pressure is monitored during a run via a 0-600 psig pressure indicator. The vessel has a pressure relief valve set at approximately 550 psig to insure against accidental over pressurization. Figure 2 is a flow diagram for the pressurized Gieseler plastometer at Conoco Coal Development Company.

## Procedure

The coal is sampled, ground and packed into the crucible in the standard manner.<sup>(1)</sup> The packed crucible assembly is then connected to the measuring head and the whole assembly is lowered, along with the attached top flange, into the pressure vessel. When the flange is flush with the top the crucible is at the proper position in the solder bath which is preheated to 320°C. After lowering, the flange is sealed and the vessel evacuated. The vessel is pressurized with the desired gas and, as soon as the solder pot temperature recovers to 320°C, the run is begun.

The sample is heated at a constant rate, normally 3°C/min, and the rotation of the rabble arm stirrer is constantly monitored and is printed out each minute. The unit of dial divisions per minute (DDPM) which is actually 100 times the rotational speed in RPM are the standard Gieseler fluidity units.

As the coal sample is heated it begins to soften and the stirrer begins to rotate. The point at which this occurs is called the softening temperature,  $T_S$ . Further heating the sample leads to an increase of the stirrer rotation until the maximum rate is reached at the temperature of maximum fluidity,  $T_M$ . Finally as the temperature of the coal is raised past  $T_M$  the fluidity begins to decrease until the temperature of resolidification,  $T_R$ , is reached. At this point all stirrer rotation stops and the sample is normally fully coked.

At the end of a run the vessel is depressurized and the sample, crucible and head assembly are removed for cleaning. A complete run requires about 1.5-2 hours.

## Results and Discussion

The coal used in this work was from Montour No. 4 mine and is a high volatile, highly caking Eastern U.S. bituminous coal from the Pittsburgh No. 8 seam. The run of mine (ROM) coal was screened at 3/4" and only the washed +3/4" lumps were used in this work. The coal was stored in a sealed plastic bag and was ground for the Gieseler work just prior to use. Table 1 shows the proximate and ultimate analyses of this coal together with the energy content, the standard Gieseler fluidity and Free Swelling Index (FSI).

A total of 44 runs were made in this study, 23 with prepurified nitrogen and 21 with hydrogen. The results of these experiments are given in Table 2. The fluidity given at each point is the average of the maximum fluidity value from at least two runs and for the unpressurized cases, four runs. The agreement between duplicate runs was always better than  $\pm 10\%$ .

Since the maximum fluidity obtainable at standard Gieseler conditions of 1.40 oz.in (141 gm.cm) of torque is about 28,000 most of the data obtained here were at lower torque settings of either 0.45 oz.in (45 g.cm) or 0.30 oz.in (30 g.cm). All the data reported in terms of the standard torque value. This value was obtained by multiplying the measured fluidity by the ratio of standard torque to the torque value actually used (i.e., 3.11 or 4.67 for the two cases in this work). When duplicate runs were made using the different torque settings the agreement between the corrected fluidities was as good as that obtained for duplicate runs at the same torque setting. This suggests that, at least in the case of highly fluid coals, a coal in its plastic state may resemble a Newtonian liquid.

If it is possible to treat this coal as a Newtonian liquid we can, by calibration of our plastometer crucible and stirrer with viscosity standards, estimate the viscosity of the coal in its plastic state. Such calibration gives

the following relationship between "pseudo" viscosity,  $\eta$  (poise) and fluidity, F (DDFM).

$$\log_{10} \eta = -\log_{10} F + 7.257 \quad 1)$$

This suggests that the apparent viscosity of the Montour No. 4 coal used in this work, at the temperature of its maximum fluidity, lies between about 170 and 2000 poise.

Figure 3, a plot of the data in Table 2 clearly shows the effect that pressure has on the maximum fluidity of this particular coal. Initially, the fluidity is increased quite rapidly by raising the pressure of both hydrogen and nitrogen, however, while the effect is seen to continue almost linearly for the hydrogen pressure, the nitrogen pressure has only a limited effect above about 100 psig. These efforts are consistent with those observed by Kaiho and Toda<sup>(2)</sup> who made a similar study for the medium fluidity, weakly caking Akabira coal. The initial large increase in maximum fluidity with pressure is probably due to the retarded rate of volatile evolution. With the more volatile and hence presumably less viscous components held in the "plastic" coal longer, an increase in maximum fluidity is logical.

Another, although seemingly less likely, possibility for the increased fluidity is the increased dissolution of the gas used and/or coal gases in the coal liquids which may lead to a decrease in the viscosity of the coal system and hence to an increase in the fluidity. The higher fluidities observed with nitrogen vs. hydrogen in the range 0-150 psig may be an experimental artifact caused by slightly lower heating rates for the hydrogen cases due to the higher thermal conductivity of hydrogen. It is known that heating rate is directly related to the fluidity of certain coals.<sup>(3)</sup>

As the pressure of the inert gas, nitrogen, is further increased, the leveling off of the fluidity may be due to approaching the limits of the effect of pressure alone on fluidity. Possibly pressures of 100-150 psig are sufficient to retard the escape of volatiles until the plastic coal has reached its temperature of maximum fluidity. Above these pressures it appears that any additional volatiles which are trapped have little effect on the fluidity.

The much more dramatic effect of hydrogen pressure on the maximum fluidity, Figure 3, is probably due to reactions between the hydrogen and components in the coal as well as the retardation of volatile escape. This observed hydrogen effect is, of course, consistent with the liquefaction of coal by very high pressures ( $\approx$  2000 psig) of hydrogen at approximately the temperature of maximum fluidity.

While the pressure has a major effect on the maximum fluidity of the coal it shows almost no relation to the temperature of maximum fluidity. Also no effect of pressure on the softening point or the resolidification temperature was observed. Throughout this work the softening temperature was  $360 \pm 5^\circ\text{C}$ , the solidification temperature was  $475 \pm 5^\circ\text{C}$  and the temperature of maximum fluidity was  $435 \pm 5^\circ\text{C}$ .

#### REFERENCES

1. Standard Method of Test for Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer. ASTM Designation: D 2639-71.
2. Kaiho, M. and Toda, Y., Change in the Thermoplastic Properties of Coal Under Pressure of Various Gases. FUEL 58, 397 (May, 1979).
3. Van Krevelen, D. W., Huntjens, F. J., and Dormans, H. N. M., "Chemical Structures and Properties of Coal I VI--Plastic Behavior on Heating," Fuel 35, 462 (1955).

TABLE 1.

Analysis of Montour No. 4 Coal

<u>Proximate Analysis</u>		
Moisture	(%)	1.69
Ash	(%)	6.97
Volatile Matter	(%)	38.79
<u>Ultimate Analysis (Dry Basis)</u>		
C	(%)	76.81
H	(%)	5.16
N	(%)	1.73
S	(%)	1.35
O (By Diff.)	(%)	7.98
<u>Ultimate Analysis (MAF)</u>		
C	(%)	82.67
H	(%)	5.55
N	(%)	1.86
S	(%)	1.46
O (By Diff.)	(%)	8.46
<u>Energy Content (Dry Basis)</u>		
HHV (Btu/lb)		14,110
Free Swelling Index		7-1/2
Fluidity (DDPM)		9300

TABLE 2

Fluidity of Montour No. 4 Coal in Nitrogen and Hydrogen Atmospheres

<u>Gas</u>	<u>Pressure (psig)</u>	<u>Fluidity<sup>(1)</sup></u>
Nitrogen	0	9,300
	50	22,300
	100	38,800
	150	44,600
	200	46,800
	250	48,100
	300	49,100
	350	51,200
	400	51,900
Hydrogen	0	9,400
	50	19,400
	100	34,700
	150	40,000
	200	51,100
	250	65,900
	300	80,800
	350	95,100
	400	104,900

(1) Fluidity rounded to nearest 100 DDPM based on average of at least 2 measurements.

All runs at  $3 \pm .1^\circ\text{C}/\text{min}$  heating rate.

