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FILTRATION OF SLURRIES FROM COAL LIQUEFACTION PROCESSES

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ABSTRACT

The heteroatom content, primarily phenolic oxygen and basic nitrogen, of coal derived asphaltenes and preasphaltenes contribute to intermolecular association and to the colloidal behavior in coal-derived liquids. The interaction between coal-derived liquids and between coal-derived liquids and mineral solids dominate the rheological characteristics of the slurry and filtrate, the specific cake resistance of the filtered solids and the cake compressibility.

The filtration of slurries form coal liquefaction processes are reviewed and the problems discussed.

INTRODUCTION

Solids separation in coal liquefaction has long been recognized as an extremely critical processing step.(1) Many approaches have been taken.(2) Each approach has advantages and disadvantages.(3) Each constitutes a significant fraction of the capital and operating expenses of a coal liquefaction plant.

Pressure precoat filtration has been tried in several pilot plants. It was used in the Cresap pilot plant beginning in 1963 but mechanical problems hampered operations.(4) More recently pressure precoat filtration was evaluated in the solvent refined coal (SRC) pilot plants at Wilsonville and Fort Lewis.(5,6) Mechanical problems resulted in 70-75% availability of the filters.(7) Filtration rates were low, depending closely upon the amount of very fine mineral particles and on the asphaltene and preasphaltene content. Screen blinding further reduced the filtration rate. The utilization of a precoat added time to the filter cycle as well as cost. Although the solids content in the extract could be reduced to as low as 0.02 wt.%, it was quite variable and could be in excess of 1 wt%.

In spite of its apparent disadvantages, pressure precoat filtration is still considered a viable alternative and Air Products is considering filtration for the SRC I demonstration plant.(8) Increases in filtration rates, reductions in precoat costs and attention to mechanical problems could well make filtration an attractive method for solids separation in coal liquefaction. In 1973 studies were initiated at the University of Michigan into the causes of the low filtration rates. Some of the results are reported here. Fundamental filtration studies have also been conducted at Oak Ridge (9,10) and at Johns-Manville.(11)

FILTRATION THEORY

The filtrate flow rate, $\frac{dV}{dt}$, at any instant in a pressure precoat filter can be expressed as

$$\frac{1}{A}\frac{dV}{dt} = \frac{-\Delta P_{total}}{\mu[\alpha wV/A + r]} \tag{1}$$

where A = filter area

V = filtrate volume collected at that instant

t = time

$-\Delta P_{total}$ = pressure drop accross filter

μ = filtrate viscosity

α = specific cake resistance, length/mass

w = mass dry cake solids per unit volume of filtrate

r = combined resistance of precoat and filter screen, length^{-1}.

The combined resistance of the precoat and filter screen, r is usually small in comparison to the instantaneous resistance of the filter cake, $\alpha wV/A$. The resistance r can be determined at the completion of precoating or it can be extracted from the filtration data by plotting the data in a way consistent with an integrated form of Equation (1). The specific cake resistance, α, can be obtained from the same data plot or from a differential analysis using Equation (1). In any event, the viscosity, μ, must be measured separately in a viscometer. Since $\mu\alpha$ appears as a product, any errors made in measuring viscosity will result in errors in α. The viscosity, μ, measured in a viscometer is not necessarily the viscosity of the filtrate as it passes through the filter cake and precoat if the filtrate is shear thinning and/or thixotropic.

If filtrate flow rates are to be increased, then either μ or α or both must be reduced.

SLURRY SOLIDS

Slurry solids in coal liquefaction are those constituents which are insoluble when repeatedly washed or extracted with pyridine or tetrahydrofuron (THF) at a particular temperature. They range in size from submicron to greater than 25 mesh. The solids can nominally be classified into three categories: organic solids containing few or no minerals; solids which are mixtures of mineral solids and organic compounds containing high levels of heteroatoms; and solids which are almost exclusively inorganic in nature.

The majority of the solids are mineral solids which are intimately associated with carbonaceous phases through hydrogen bonding or some form of electrostatic attraction. These solids can be made to associate closely with organic constituents, especially preasphaltenes, by addition of non-hydrogen bonding solvents to the slurry or by cooling.

The size distribution of the three types of solids residue is important to filtration. Because the size distribution is very dependent upon temperature and the "liquid" properties, it is very difficult to make a size distribution estimate except at conditions substantially different from actual process conditions. Coulter counters are commonly used to measure size distributions after residue samples are dispersed in an electrolyte.(12,13) Walker reported 90 wt.% of the particles were greater than 1-4 μm in 5 residues by such analyses. When most of the particles are below 1 micron, it is necessary to use a scanning electron microscope for analysis. Stirling examined two residues with a scanning electron microscope and found that 50% of the particles were below 0.15 μm in a residue from the liquefaction of a Pittsburgh seam coal and were below 0.07 μm in a residue from the liquefaction of an Illinois No. 6 coal.(14) Sample preparation is critical as soluble organic molecules can be left as residue particles after the solvent is evaporated.

An estimate of the mean effective particle diameter of particles in a filter cake can be made from the measured specific cake resistance using Carman's relationships for rigid particles.(15)

$$\alpha = \frac{(1 - \varepsilon)\ 5\ S^2}{\varepsilon^2\ \rho_{solids}} \qquad (2)$$

where, ε = cake porosity

S = specific surface area, area of particle per unit volume of solids

ρ_{solids} = density of solids in cake

and $$D_p = \frac{6\ (1-\varepsilon)}{\phi_s\ S} \qquad (3)$$

where, D_p = mean particle diameter

ϕ_s = particle shape factor

When the equivalent to S from Equation (2) is substituted for S in Equation (3) then

$$D_p = \left[\left(\frac{1-\varepsilon^{3}}{\varepsilon}\right) \quad \frac{5 \cdot 36}{\alpha \phi_s^{2} \rho_{solid}} \right]^{1/2} \tag{4}$$

The relationship between mean particle diameter and specific cake resistance is given in Table 1 for conditions considered reasonable for coal liquefaction residues.

FILTRATE AND SLURRY CHARACTERISTICS

The balance of the coal liquefaction filter feed slurry is the filtrate. It can be broadly classified as containing oils, resins, asphaltenes and preasphaltenes. Oils and resins are nominally soluble in pentane, asphaltenes insoluble in pentane but soluble in benzene or toluene and preasphaltenes are benzene-insoluble but soluble in tetrahydrofuran (THF) or pyridine. The relative amounts of oils, resins, asphaltenes and preasphaltenes at any time are important as the viscosity and colloidal properties of the resulting mixture depend upon the concentration of asphaltenes and upon the concentration of preasphaltenes to even a greater extent.

The most striking difference between the three fractions is in the concentration of heteroatoms as tabulated in Table 2. In the coal-derived materials studied the "average" preasphaltene molecule contained 6-7 heteroatoms compared to 0.4-1.2 and 0.2-

TABLE 1.

Estimate of Specific Cake Resistance for Rigid Particles

D_p, μm	0.25	0.50	0.75	1.00	1.25	1.50	2.0
α, m/kg x 10^{-12}	10.28	2.57	1.14	0.64	0.41	0.29	0.16

α from Equation (4) for $\varepsilon = 0.38$; $\phi_s = 0.73$; $\rho_{solids} = 2000$ kg/m^3

TABLE 2.
Analysis of Coal Derived Materials from the
Liquefaction of Illinois No. 6 Coal in the H-Coal Process

Fraction	Wt. %	Mol. Wt[1]	Weight Percent C	Weight Percent H	Weight Percent N	Weight Percent O[2]	Weight Percent S	Atoms in Average Molecule C	Atoms in Average Molecule H	Atoms in Average Molecule N	Atoms in Average Molecule O	Atoms in Average Molecule S
VACUUM STILL BOTTOMS												
Oils-Resins	33.6	369	89.83	8.95	0.42	0.80	---	27.62	33.03	0.11	0.18	---
Asphaltenes	29.2	485	88.72	7.05	1.11	2.69	0.43	35.86	34.19	0.38	0.82	0.07
Preasphaltenes	9.6	980	83.97	6.19	1.73	7.08	1.03	68.58	60.66	1.21	4.34	0.32
Insolubles	27.6	---	-----	----	----	----	----					
PRODUCT OIL[3]												
Oils-Resins	23.7	245	91.31	7.65	0.19	0.85	----	18.64	18.74	0.03	0.13	----
Asphaltenes	63.2	280	90.34	7.23	0.53	1.90	----	21.08	20.24	0.11	0.33	----
Preasphaltenes	3.8	810	79.38	6.51	1.61	11.83	0.67	53.58	52.73	0.93	5.99	0.17
Insolubles	9.3	---	-----	----	----	-----	----					

[1] Molecular Weight by vapor pressure osmometry in pyridine at *75°C*

[2] Oxygen by difference

[3] Product oil is the gross reactor effluent after pressure let-down.

0.3 for asphaltenes and oil and resins, respectively. A majority of the oxygen is present as phenolic oxygen.(16) A substantial portion of the nitrogen occurs as basic nitrogen.

The viscosity, solubility and adsorption characteristics of asphaltenes and preasphaltenes depend almost exclusively upon the heteroatoms through hydrogen bonding and upon π-π bonding. Of the heteroatoms in coal-derived liquids, oxygen as phenolic oxygen is by far the most dominant because it will hydrogen bond with basic nitrogen and will associate with other phenolic oxygen atoms. As the concentration of molecules with two or more heteroatoms increases, the probability of colloidal micellization or agglomeration increases since the intermolecular association of two such molecules does not preclude further association. When an individual molecule is associated to another molecule or solid particle at two or more points, the association can persist at higher temperatures than would normally be expected. Illustrations of π-π and/or hydrogen bonding in model compounds as exhibited by their melting points are shown in Figure 1. The position of the functional group is important because of steric hinderance.

The presence of colloidal particles ($<1\mu m$) in filtration is important since the fine particles can pass into the cake and be captured in the interstitial volume between larger particles. Such capture would increase the specific cake resistance.

Ho has shown by small angle X-ray measurements that collodial particles exist in solution when high molecular weight asphaltene fractions are dispersed in THF or pyridine.(17) His data are presented in Figures 2 and 3. The asphaltene fraction was the 75% pentane/25% toluene-insoluble, toluene-solution fraction from the H-Coal vacuum bottoms from the liquefaction of an Illinois No. 6 coal. This particular asphaltene fraction contained an average of 0.6 nitrogen and 1.2 oxygen atoms per molecule. Gel permeation chromatographic fractionation of this high molecular weight asphaltene fraction revealed that over 20 wt. % of the fraction had mole-

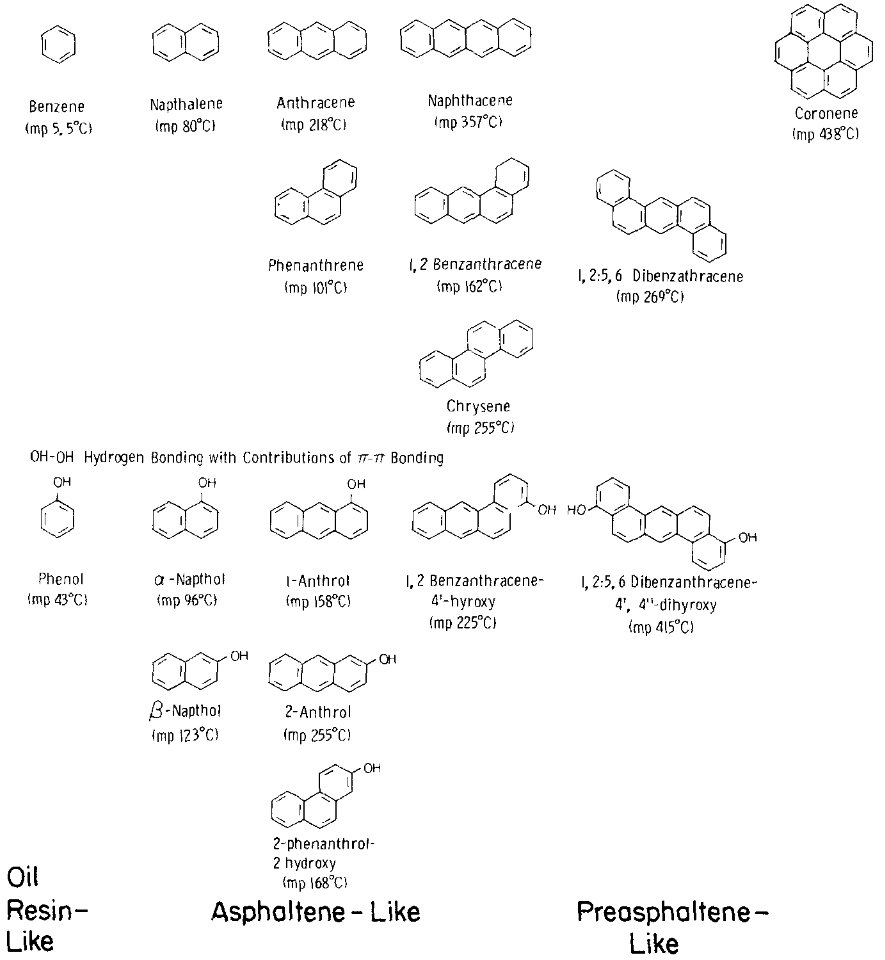

FIGURE 1. Illustrations of the Effects of π-π Bonding and/or Hydrogen Bonding as Evidenced by Their Melting Points.

cules which contained on the average more than 1.3 nitrogen and 5.2 oxygen atoms per molecule and molecular weights above 1200 in pyridine at 75°C. It is very possible that much of this subfraction was associated under the conditions of the molecular weight determination, in this case being vapor pressure osmometry.

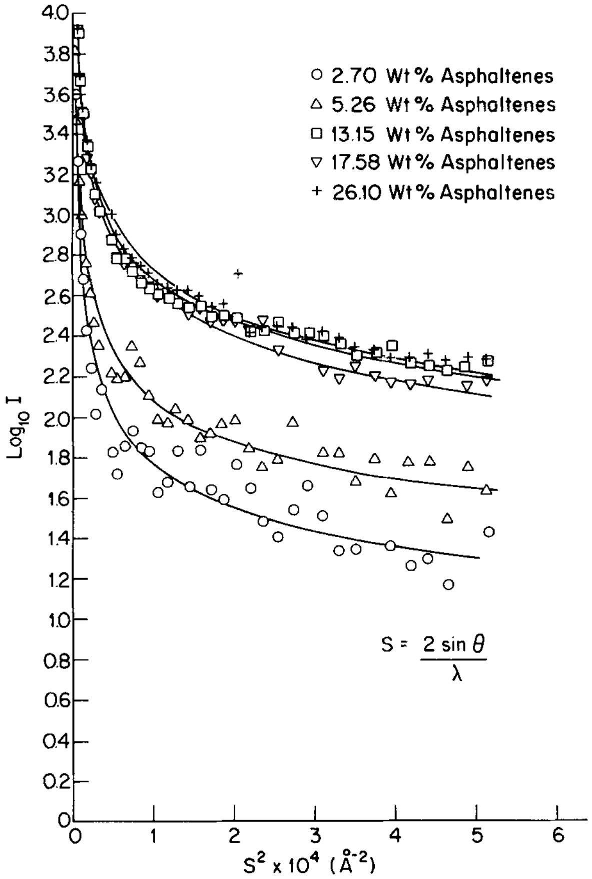

FIGURE 2. Guinier Plot for Small Angle X-Ray Scattering of Asphaltenes in THF at 25°C.

The size distribution of colloidal particles in the samples examined by small angle X-ray scattering data are presented in Table 3. The method of Jellinek et al. was used for the size distribution estimate.(18) From 5-9 wt.% of the asphaltenes have radii of gyration in the 20-65 Å (0.002 - 0.0065 μm) size range and 1-4 wt.% have radii of gyration above 65 Å in the two solvents. There does not seem to be a great difference between the distribution in the two solvents. If anything, there appears to be larger

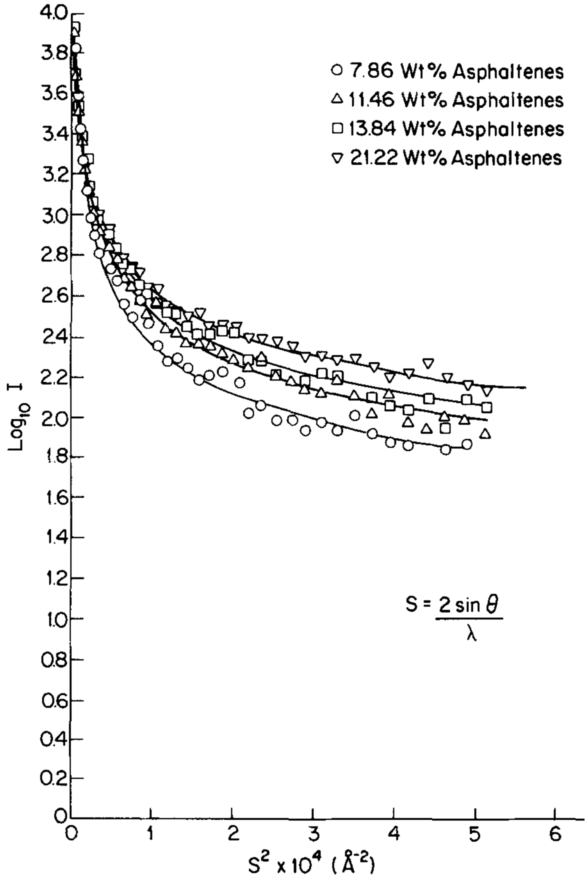

FIGURE 3. Guinier Plot for Small Angle X-Ray Scattering of Asphaltenes in Pyridine at 25°C.

particles in the pyridine solutions, probably because pyridine will associate more strongly with asphaltenes molecules than would THF.

The properties of coal liquefaction slurries can be altered by the addition of association/precipitation-inducing solvents to the slurry. The extent of the change depends upon the type and amount of additive, temperature and shear rate. A typical response is illustrated in Figure 4. With a small amount of additive, as in the region AB of Figure 4, colloidal micelles would exist

TABLE 3.

Correlation of the Particle Size Distribution and the Concentration from Small Angle Scatter Data

Radius of (gyration)	Concentration	Solvent	Weight Fraction
Small size range, Å			
8-10	2.70%	THF	0.906
8-10	5.26%	THF	0.930
8-10	13.15%	THF	0.914
8-10	17.58%	THF	0.895
8-10	26.10%	THF	0.920
8-10	7.86%	pyridine	0.900
8-10	11.46%	pyridine	0.871
8-10	13.84%	pyridine	0.916
8-10	21.22%	pyridine	0.884
Medium size range, Å			
20-65	2.70%	THF	0.056
20-65	5.26%	THF	0.050
20-65	13.15%	THF	0.069
20-65	17.58%	THF	0.095
20-65	26.10%	THF	0.070
20-65	7.86%	pyridine	0.057
20-65	11.46%	pyridine	0.070
20-65	13.84%	pyridine	0.078
20-65	21.22%	pyridine	0.084
Large size range, Å			
> 65	2.70%	THF	0.021
> 65	5.26%	THF	0.020
> 65	13.15%	THF	0.017
> 65	17.58%	THF	0.010
> 65	26.10%	THF	0.010
> 65	7.86%	pyridine	0.043
> 65	11.46%	pyridine	0.029
> 65	13.84%	pyridine	0.006
> 65	21.22%	pyridine	0.032

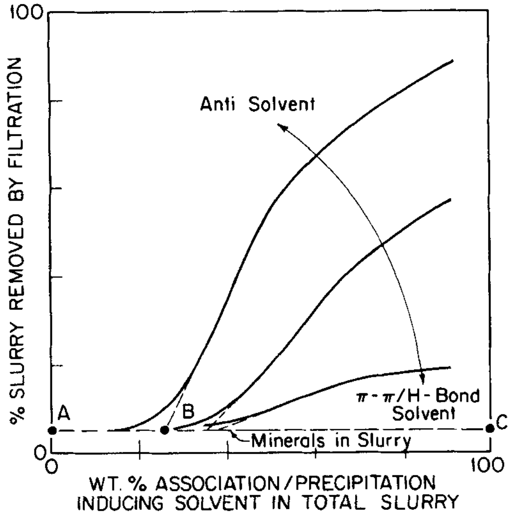

FIGURE 4. Effect of Solvent Additions of Filtration of Coal Liquefaction Slurries.

from the assocation of the multiheteroatom molecules and multiheteroatom molecules would associate with and/or bridge together mineral solids but the amount of material removed by filtration would be only slightly greater than the amount of minerals and undissolved coal in the slurry. As the amount of precipitate-inducing solvent is increased, the micelles associate and extensive precipitation occurs as shown at additive concentrations to the right of point B. The amount of material removed in the region BC depends significantly upon the characteristics of the additive. Solvents such as n-decane can not prevent association except by dilution effects. Strong hydrogen bonding solvents such as THF and pyridine will tend to keep the slurry dispersed by competing with coal-derived heteroatom molecules in intermolecular associations. Ideally, enough precipitate-inducing solvent should be added to cause the highly multiheteroatom asphaltenes and preasphaltenes to bridge together mineral solids and to form large micelles with similar molecules without excessive precipitation or association of the molecules with fewer heteroatoms. If this were done, the specific cake resistance would decrease, the viscosity would decrease and the fraction of highly multiheteroatom material in the filtrate would decrease. The filtrate would then have better properties as a fuel oil and as a feed stock for catalytic upgrading.

VISCOSITY

The viscosity of a coal liquefaction slurry or filtrate depends upon the relative amounts of oils and resins, asphaltenes and preasphaltenes. The mineral solids concentration has a significant effect on the slurry viscosity. The oils and resins behave nominally as a Newtonian fluid. Viscosity testing of asphaltenes by Cameron showed that the asphaltenes are strongly dependent upon shear rate history necessitating a specific system of testing.(19) Cameron used the following procedure. The asphaltenes where subjected to a shear rate greater than 100 sec^{-1} until a constant torque was obtained. The shear rate was then reduced to a lower value (0.05 or 1 sec^{-1}) and the torque measured immediately. This torque was called the torque at zero time. Shearing was next terminated completely for 12 minutes before shearing again at the lower shear rate. The torque was measured immediately and shearing again stopped. The measured torque was compared to that measured at zero time. After the fifth measurement at low shear rate, shearing was continued until a steady torque was reached. In all cases there was a drop-off of torque to a steady value above the torque measured at the lower shear rate at zero time. The results are presented in Figure 5. The increase in torque with time is indicative of intermolecular association. The decrease in torque with time upon steady shearing is indicative of intermolecular association. The decrease in torque with time upon steady shearing is indicative of shear-induced dissociation and the material would be considered thixotropic. The drop-off of torque was more pronounced in the higher shearing case (1.0

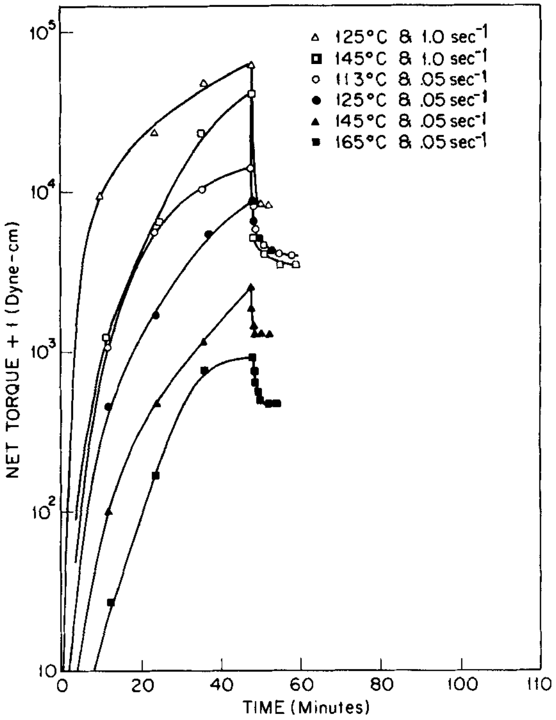

FIGURE 5. Net Torque above Torque at Zero Time After a High Shear Rate History with No Shear between Data Points for Coal-Derived Asphaltenes.

sec^{-1}) indicating that higher shear rates are more effective in breaking up associated material.

Cameron found that if a viscosity determination were made immediately following shearing at 100 sec^{-1} for 1 minute that the viscosity of the asphaltene fraction was nominally independent of shear rate down to a shear rate of 0.01 sec^{-1} . The data are presented in Figure 6. Because of the high shearing prior to each measurement, the asphaltenes were nominally in the same colloidal state for each measurement and the results shown in Figure 6 would indicate that the asphaltenes were only slightly shear thinning under the conditions of the measurements.

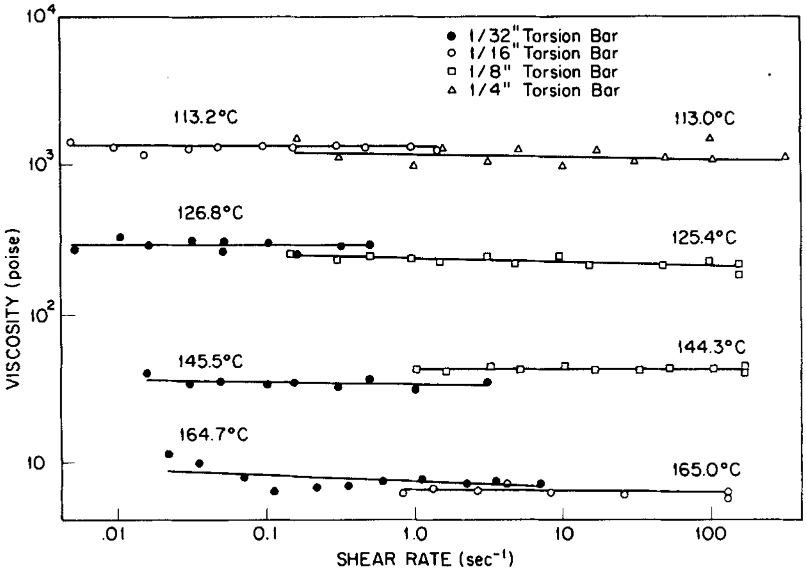

FIGURE 6. Effect of Shear Rate on Viscosity of Coal Derived Asphaltenes with Each Measurement Proceeded by High Shear Rate.

Cameron tested unextracted H-coal vacuum bottoms material consisting of oils and resins, asphaltenes, preasphaltenes, and residual solids for viscosity over about five decades of shear rate and over a temperature range from 90°C to 146°C. Contrary to the asphaltene testing, he found that high-shearing immediately before taking a data point actually caused a higher torque. A period of time on the order of 10 minutes was necessary for the torque to reach a lower equilibrium value. Data were taken from high to low shear rates without interspersing with high-shearing. Thixotropy was evident at all levels of shear rate but equilibrium was reached in a short time. Time-torque studies similar to those for asphaltenes indicated a build up of torque with time but an almost complete rapid recovery to the original torque indicating that the previous shear history was not that important for this material.

Since the viscosity behavior of H-Coal material was similar to some polymer materials, the data were treated by the procedure of time-temperature superposition. Briefly, time-temperature superposition is a method by which viscosity data for different temperatures can be superimposed to obtain a master curve of extended shear rate at the reference temperature. Superposition of three isotherms onto the 90°C isotherm is shown in Figure 7. The superposition appears reasonable with respect to shapematching of curves.

The data in Figure 7 shows evidence of shear-induced dispersion of associated colloidal micelles. At shear rates up to 0.001 sec^{-1}, the structure is reasonably rigid. As the shear rate increases above 0.001 sec^{-1}, the clusters of micelles increasingly disperse until at 1 sec^{-1} only strong intermolecular associations persist.

McKeen measured the effect of precipitate-inducing and hydrogen-bonding solvents on the viscosity of filtered product oil from the liquefaction of an Illinois No. 6 coal in the H-coal process. (20) The filtrate represents the oils, asphaltenes and preasphal-

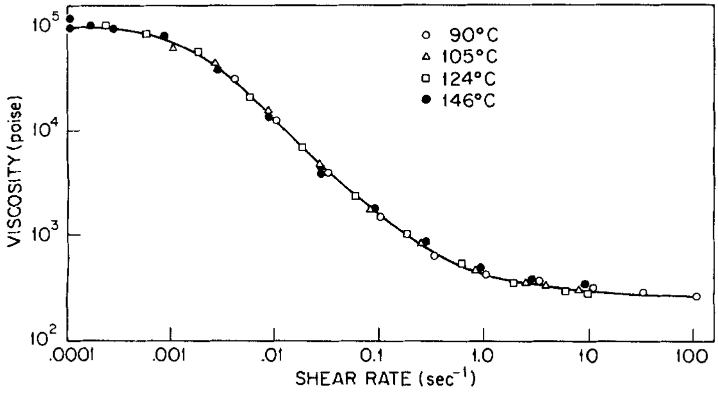

FIGURE 7. Time-Temperature Superposition on 90°C Isotherm of Viscosity Data Taken on H-Coal Vacuum Bottoms Produced by the Liquefaction of an Illinois No. 6 Coal in the H-Coal Product Oil.

tenes in the product oil described in Table 2. The measurements were all made in a helical coil viscometer at approximately the same shear rate. The data are presented in Figures 8 and 9. The viscosity reduction in the case of precipitate-inducing solvents was by a combination of dilution and preasphaltene and asphaltene association. In the case of hydrogen-bonding solvents the reduction was by a combination of dilution and colloidal particle dispersion.

FILTRATION EXPERIMENTS

Stirling conducted a number of special filtration experiments to determine the major contributions to the high specific cake re-

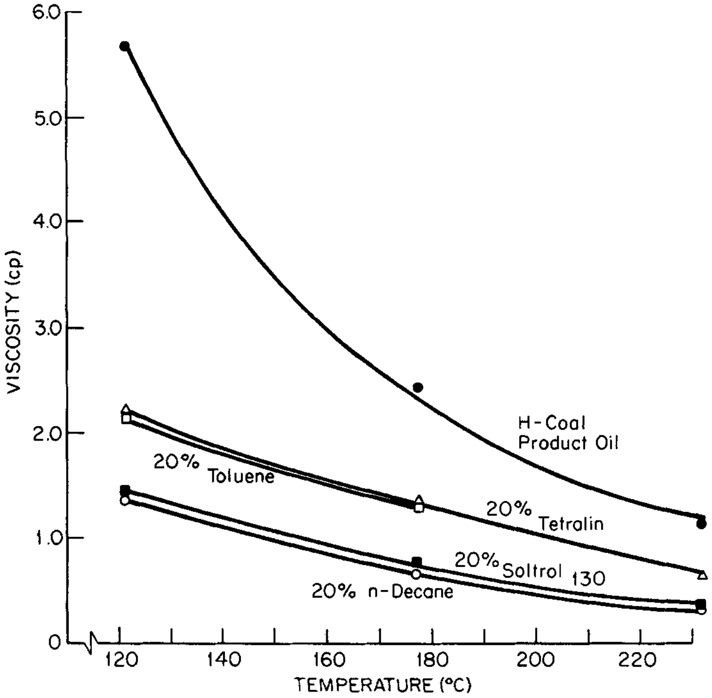

FIGURE 8. Effect of Temperature on the Viscosity of the Filtrate Obtained from the Indicated Slurries of H-Coal Product Oil.

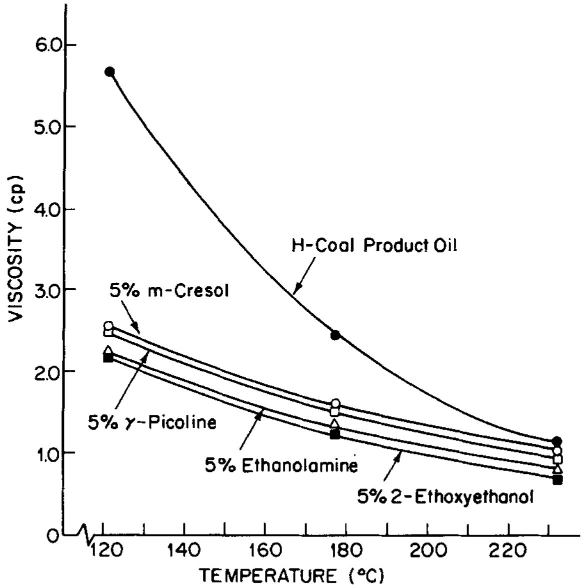

FIGURE 9. Effect of Temperature on the Viscosity of the Filtrate Obtained from the Indicated Slurries of H-Coal Product Oil.

sistance of filter cakes produced when filtering coal-derived slurries.(14) Extentions to his work are reported here.

Filtration of THF - and Pyridine - Extracted Solids

Stirling observed that the specific cake resistance of filter cakes made up of THF-insoluble solid particles increased with temperature especially between 177 and 232°C. He found that between these two temperatures the specific cake resistance increased by approximately 100%. He attributed this increase to softening and plastic flow of undissolved organic material in the filter cake. In order to test this theory, THF insoluble solid particles were exhaustively extracted with pyridine in a Soxhlet extractor. This extraction removed 10 wt.% of the starting THF-insoluble material. These solids were then slurried in tetralin and filtered at temperatures ranging from 121 to 232°C. The results of these filtrations as well as several filtrations of the THF insoluble solid particles are shown in Figure 10.

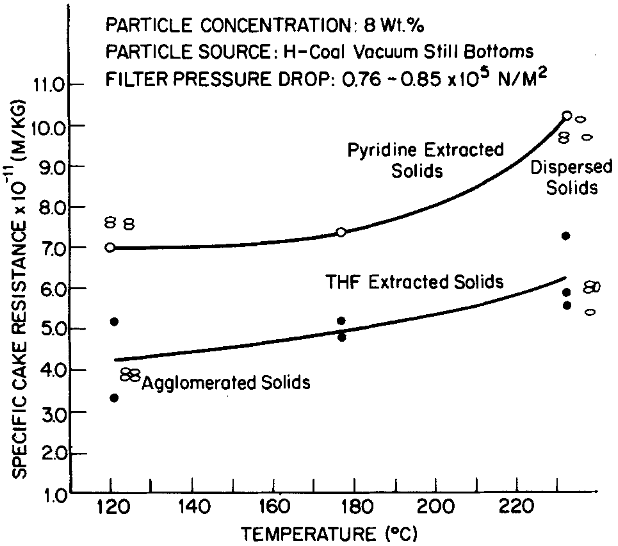

FIGURE 10. Effect of Degree of Organic Material Extraction on the Specific Cake Resistance.

From Figure 10, it can be seen that the specific cake resistance does not change significantly from 121 to 177°C for both the THF-insoluble and the pyridine-insoluble solid particles. However, from 177°C to 232°C the specific cake resistance increases significantly, by 26% for the THF-insoluble solids, and by 40% for the pyridine-insoluble solids. The fact that these increases are roughly of the same magnitude and that they occur over the same temperature range implies that the material dissolved from the THF-insoluble solid particles by pyridine extraction is not solely responsible for the increase in specific cake resistance. Therefore, either increased dispersion of some of the solid particles themselves is causing the increase in specific cake resistance or there is still some residual organic material present in the pyridine extracted solids.

The fact that the specific cake resistance is higher for the pyridine-insoluble solids than for the THF-insoluble solids at every temperature is explained by the removal of organic material by the pyridine extraction. The organic material removed must contain molecules with many heteroatoms per molecule or be molecules of many condensed aromatic rings. Organic material present in the solids serves to bridge some of the smaller particles into larger particles. Filtering slurries of larger particles gives filter cakes that have lower specific cake resistances. The pyridine extraction served to remove most, if not all, of the residual organic material leaving behind unassociated small particles. These smaller particles would give filter cakes of higher specific cake resistance.

Filtration with Precipitate-Inducing Solvents

McKeen conducted a series of filtration experiments with the H-coal product oil described in Table 2.(20) Four solvents were used: tetralin, toluene, n-decane and Soltrol 130 (a mixture of paraffinic hydrocarbons boiling in the range 185-207°C). The results, when the solvents are added to give concentrations of 20 wt.% additive in the slurry and the slurry agitated for 2-hours at the filtration temperature before filtration are given in Figure 11 over a range of temperatures. Each solvent produced a decrease in the apparent specific cake resistance by association or agglomeration of colloidal-size particles into larger particles. The mean particle size of the cake increased. The words apparent specific cake resistance are used here as the viscosities used in the computation were those measured for the filtrate in a helical coil viscometer and are not necessarily those of the liquid passing through the cake. The magnitude of the change in apparent specific cake resistance is inversely related to similarities between the slurry liquid and solvent. The paraffinic hydrocarbons do not restrict intermolecular associations among the coal-derived constituents while tetralin, being hydroaromatic, can weakly inhibit associations through π-π bonding.

Filtration with Hydrogen Bonding Solvents

McKeen also conducted experiments with the H-Coal product oil to which hydrogen-bonding solvents were added.(20) The results

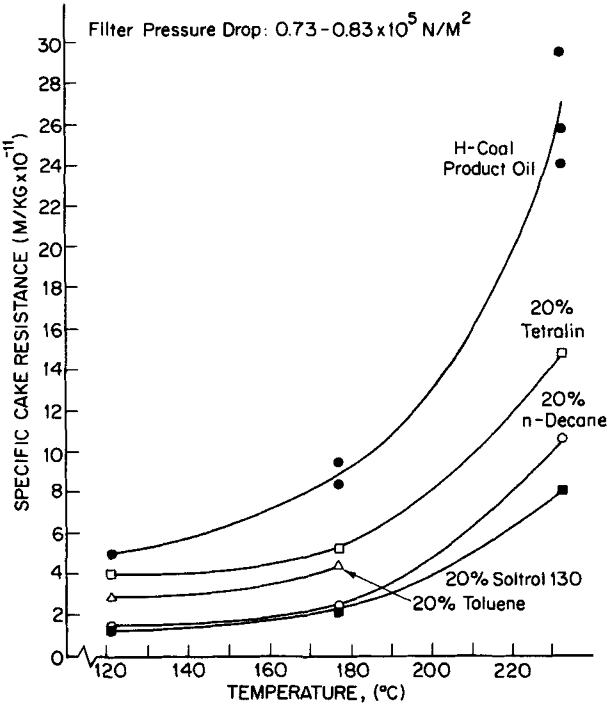

FIGURE 11. Effect of Temperature on the Specific Cake Resistance for Slurries of H-Coal Product Oil Containing 20 Weight Percent Precipitate-Inducing Solvents.

are presented in Figure 12 for additions which give concentrations of 5 wt.% solvent in the slurry. The hydrogen-bonding solvents produce increases in the apparent specific cake resistances by peptization of large colloidal particles into smaller particles and by solubilizing preasphaltene molecules which bridge together small mineral particles. The mean particle size of the cake decreases. The 2-ethoxyethanol is particularly effective in dispersing the associated system. 2-ethoxyethanol contains two oxygen heteroatoms and can hydrogen bond with both acidic and basic fractions. Also being molecular weight 90 compared to 93 and 108 for γ-picoline and m-cresol, respectively, considerably more hydrogen bonding interactions are possible at a concentration 5 wt.% compared to γ-picoline and m-cresol.

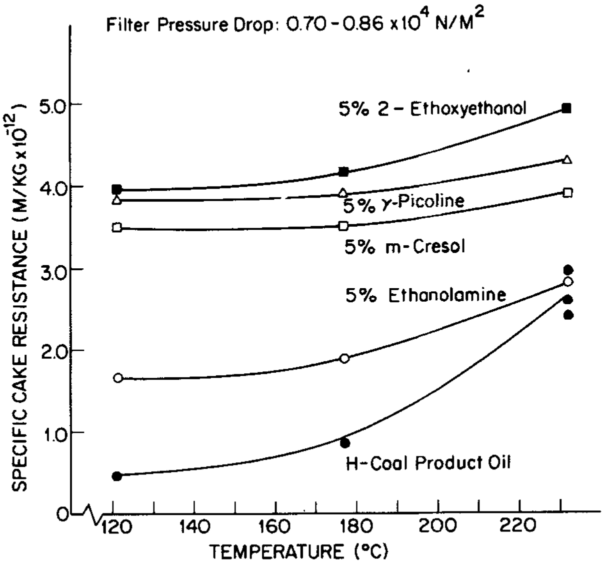

FIGURE 12. Effect of Temperature on the Specific Cake Resistance for Slurries of H-Coal Product Oil Containing 5 Weight Percent Hydrogen Bonding Solvents.

While hydrogen-bonding solvent additions are not appropriate for improving filtration, experiments with filter slurries can reveal the type of solvents which can more readily peptize the colloidal constituents of coal in the pre-heater during coal liquefaction.

Precoat Studies

Islip conducted experiments to determine possible interactions between a filtered product oil and a 75 wt.% Celite 503 diatomaceous earth/25 wt.% Fibra-Flo 7-C asbestos precoat. The H-Coal product oil described in Table 2 was filtered and the filtrate used in the experiments reported in Table 4. In each run the precoat was laid down with tetralin at 232°C and the initial resistance, r_0, determined. The viscosity of tetralin at 232°C was used in the calculations. The slurry was then loaded into the preparation vessel where it was heated to 232°C while being stirred with a

TABLE 4.

Study of the Interaction of Filtered Product Oil on a Precoat Material

Precoat Flow Resistance During Lay-down with Tetralin at 232°C	Pressure Drop	Slurry	Change in Resistance of Precoat when Filtering Slurry at 232°C
r_o	ΔP_{total}	--	Δr
$2.15\text{X}10^{12}\,m^{-1}$	$1.27\text{X}10^{5}\,N/m^{2}$ (18.5 psi)	Filtered Product Oil	$-0.17\text{X}10^{12}\,m^{-1}$
$1.06\text{X}10^{12}\,m^{-1}$	$1.29\text{X}10^{5}\,N/m^{2}$ (18.8 psi)	Filtered Product Oil + Tetralin @ 20% of Slurry	$+0.10\text{X}10^{12}\,m^{-1}$
$2.27\text{X}10\quad m^{-1}$	$3.01\text{X}10^{5}\,N/m^{2}$ (43.75 psi)	Filtered Product + Tetralin @ 20% of Slurry	$-1.0\text{X}10^{-12}\,m^{-1}$

Precoat 75 wt% Celite 503 Diatomaceous Earth
25 wt% Fibra-Flo 7-C Asbestos

Filtered Product Oil was obtained by filtering through the precoat material H-Coal product oil from liquefaction of an Illinois No. 6 Coal in H-Coal process.

Magne-Drive stirrer at 1200 rpm. After being stirred at 232°C for two hours, the slurry was passed into the filter vessel and immediately filtered through the precoat. The filtrate flow rates were measured. The viscosity of the filtrate was measured at 232°C in the helical coil viscometer after filtration and the new resistance computed. Little or no build-up of cake was observed.

The changes in resistance can be explained by viscosity and colloidal effects. The filtrate, being immediately filtered after a high shear history, may have a lower viscosity than measured sometime later in the visometer. When the larger value of viscosity is used in the computation of r, Δr can be negative if there are few colloidal particles that are captured or retarted in the precoat during filtration. When tetralin is added to the starting material some colloidal micellization would be expected and those micelles could be laid down on the precoat as a very thin coat or be captured in the precoat as in deep-bed filtration. In either case the resistance would increase. For experiments conducted at the same pressure drop across the precoat, the change in resistance would be expected to be larger with the tetralin addition. For experiments conducted with tetralin addition at different pressure drops, the viscosity would be reduced by shear thinning effects the most in the highest pressure drop run and the apparent specific cake resistance would be expected to be less. The results suggest that the apparent reduction in viscosity more than compensates for any colloidal particle effects.

CONCLUSION

Coal liquefaction slurries are more complex than most filtration slurries. They contain a wide spectrum of materials which range from small individual molecules to very large mineral solids and agglomerated solids. The mean effective particle size of the solids in the cake and the viscosity are both significantly affected by the presence of preasphaltenes and asphaltenes with multiheteroatoms. Selective addition of association/agglomerationinducing solvents can dramatically increase the filtrate flow rate by decreasing both the viscosity and specific cake resistance. These decreases more than compensate for the need to process slightly more slurry with a slightly increased amount of cake.

In coal liquefaction processes, the solids separation process must be considered in terms of the total process material and energy balances and the total plant capital cost. By selectively removing those fractions which tend to peptize colloidal particles and adding back fractions which promote association, filtration rates can be improved while simultaneously reducing the levels of multiheteroatom species which cause coke deposits on catalysts during subsequent product up-grading.

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