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Enhancement of Rheological Behavior of Indian High Ash Coal−Water Suspension by Using Microwave Pretreatment

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In this paper, rheological characteristics of coal−water slurries (CWS) by using microwave pretreatment have been presented. Detailed experimental investigations were carried out for two high ash Indian coals with 38 and 32% ash content. Experiments were conducted at the 900 W power level and various exposure times of 30, 60, 90, and 120 s. Before and after treatment, the samples were ground for rheological characteristic of CWS in an online Bohlin viscometer. All slurries were found to exhibit pseudoplastic characteristics and results indicate that the rheological properties of microwave-treated coal are better than untreated coal. The density of microwave-treated coal is less than that of untreated coal after grinding. A factorial plan was used, and empirical equations were obtained and found to be encouraging and highly significant.

1. Introduction

In recent years, there has been growing interest and acceptance of microwave heating of coal and minerals. However, it is well-established that the impact of mining and subsequent processing operations must be reduced to meet future sustainability requirements. One such area is the use of microwave heating technologies to improve the efficiency of various mineral processing unit operations, viz. leaching, gold ore grindability, and coal grinding. Microwave energy is a nonionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz.[1] Microwaves can pass through materials like glass, paper, plastic, and ceramic and be absorbed by foods and water; but they are reflected by metals. Microwave energy is derived from electrical energy with a conversion efficiency of approximately 50% for 2450 MHz and 85% for 915 MHz. Microwave-based processing of materials is a new, powerful, and significantly different technology to process materials that may not be amenable to convectional means of processing or to improve the performance characteristics of existing materials. Microwave treatment resulted in differential separation of mineral matter in coal as the dielectric constant of coal and mineral matters are different. This resulted in selective heating of minerals and coal carbonaceous matters being almost transparent. Recently, continuously increasing demands for energy have led scientists to seek for ways of finding new energy sources. Thus, researchers have directed their attention toward various methods of burning coal−water slurries (CWS) for energy generation. Transportation of coal in the form of slurry through a pipeline is gaining importance. Microwave pretreatment of coal has been found to selectively heat the mineral matter based on differences in dielectric properties, thereby causing the pyrite to decompose magnetically susceptible pyrrhotite.

The rheological behavior of solid−liquid suspensions has a great bearing on the power requirements for pumping of solid−liquid suspensions. These rheological properties of suspensions are very much influenced by the nature of the suspending medium, particle size, shape, surface characteristics, and size distribution.[2]

The surface characteristics of microwave-treated coals and that microwave treatment on few Indian high-ash coals resulted in smoothing of surface and conversion of α-silica to β-silica. It was found that microwave-treated coal slurry facilitates enhanced flow characteristics and abates the erosion problem in pipeline transport.[2,3] The rheological properties of coal−oil−water suspensions containing solids of different sizes and the effect of coal particle size distributions on rheology of CWS was studied, and some previous work with respect to slurry rheology in ultrafine grinding was reviewed.[4−6] Different chemicals were used as the dispersing agent and stabilizer and have a significant effect on the stability and viscosity of coal−water slurries.[7] The properties, settling rates, and the rheology of coal−water mixtures (CWM) made up from different coals were investigated.[8] Literature survey reveals that a detailed study of coal−water slurry rheology has been carried out using 20 coals of different origins having ash contents 2.6−37.8% by weight. They have generalized the flow behavior into three categories based on carbon content of coal.[9] They derived the following expression for spherical particles in liquid.

$$\mu_{sl} = (1 + 2.5X_v + 7.17X_v^2 + 16.2X_v^3)\mu \qquad (1)$$

The irregular particles of rigid spheres in Newtonian fluids at low and moderate concentrations of solids are reported in the literature.[10] Both rheological and hindered-settling characteristics of small particle size suspensions of 10−50 μm with particles of thorium oxide in water, methanol in titian kaolin, and alumina and graphite in water were studied, and the following expression was proposed for suspensions of high solid concentration:[11]

$$\frac{\mu_{sl}}{\mu_l} = 1 + 2.5X_v + 10.05X_v^2 + 0.062\exp^{[(1-0.875X_v)/(1-1.595X_v)]} \qquad (2)$$

Literature review reveals that the viscosity of a suspension depends on the nature of the solid particles, shape, particle size distribution, nature of suspending medium, solid concentration, additives, pressure, and temperature. The rheology of coal slurries has been studied mostly with low-ash coals. Since Indian coals are high in ash content and moreover the nature of low-ash coal is completely different from those found elsewhere, it

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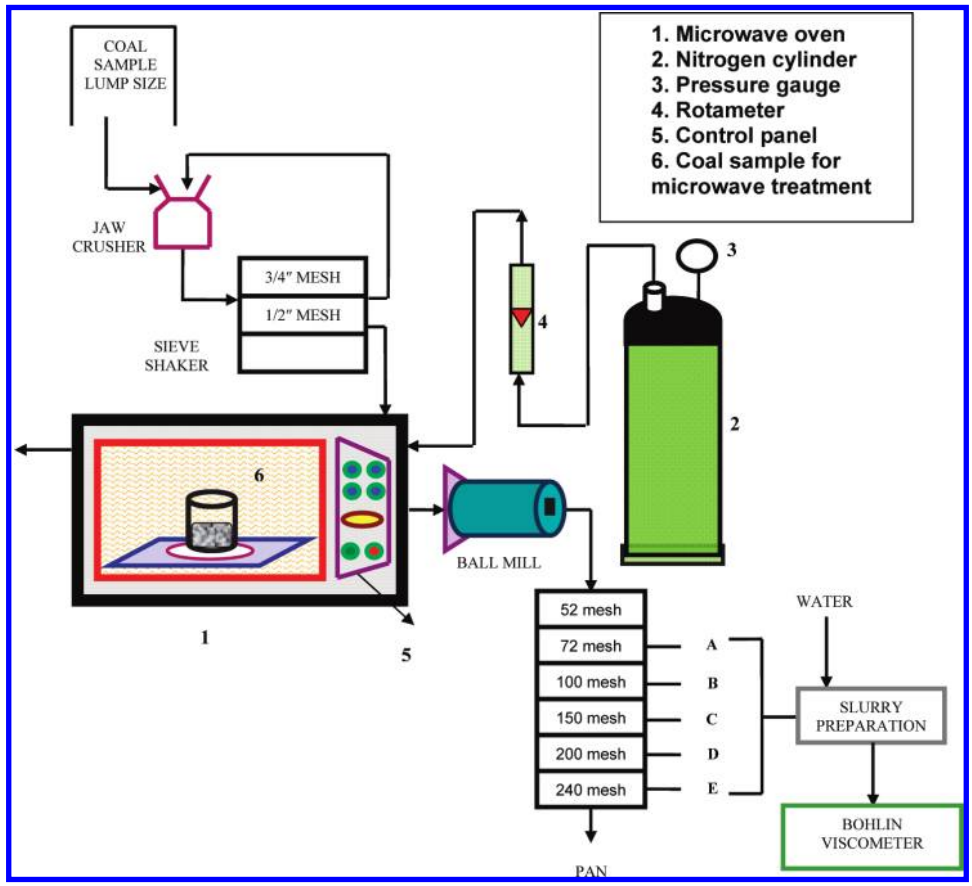

Figure 1. Schematic of experimental setup for microwave treatment of coal.

is very much essential to measure rheological characteristics. In addition, after microwave treatment, the rheological behavior of pretreated coal should show different behavior from that of untreated coal. However, detailed rheological characteristics of microwave-treated coal−water suspensions are very limited. In the present investigation, therefore an attempt has been made to investigate the properties of microwave untreated and treated Indian high ash coals for rheological characterization.

2. Experimental Setup and Technique

The schematic diagram of the experimental setup is shown in Figure 1. Microwave treatment was carried out with a 900 W experimental prototype microwave oven with variable power at 2.45 GHz. The experimental setup mainly consists of a microwave oven, which was used for microwave pretreatment of coal and was a LG MC-808WAR model. The microwave oven consists of a door handle, microwave radiation-proof oven window, stirrer fan cover, control panel, and oven cavity light. Nitrogen gas at a controlled flow rate was maintained within the oven as an inert atmosphere. Hot air, steam, and vapors were generated within the oven cavity during treatment. An air vent was provided to expel these vapors and other gases during operation. Two types of coal were chosen, viz. Jamadoba washery (TATA STEEL) referred to as coal-X (38% ash content) and West Bokaro washery referred to as coal-Y (32% ash content). The original coal sample of 15 kg was crushed in a jaw crusher, which gives −3/4 in. +1/2 in. mesh sieves. The coal samples of −3/4 in. +1/2 in. (19.05−12.7 mm) fractions were taken in a glass container of around 0.5 kg capacity such that the height of the coal bed was approximately equal to the diameter of the container. The container containing the coal samples was placed on the floor of the oven in the revolving tray. Then nitrogen gas was purged at controlled rate through a rotameter in order to create an inert atmosphere for about 5 min, and then, the door was closed carefully. Then the oven was set at a power level of 900 W and a programmable time of 30, 60, 90, or 120 s. The temperature of the coal in the actual experiment was measured by a digital thermometer (range 0−200 °C, type Pt-100). Then, the microwave-treated coal samples were collected for different sized fractions by ball mill. The microwave-treated coal of −3/4 in. +1/2 in. (19.05−12.7 mm) fractions was grinded in a ball mill for 20 min. After every 5 min, the sample was taken out and sieved with the help of a sieve shaker for 5 min to get the five cut fractions (253, 182, 127, 90, and 60 μm). Each of the fractions was collected and weighed. The oversize material (>295 μm) was returned to the mill for further grinding in successive intervals of 5 min for a total time of 20 min. The similar procedure was followed for coal-X and coal-Y.

The Bohlin Visco 88 BV was used to measure rheological properties of the suspension with a concentric cylindrical geometry with a rotating inner cylinder and stationary outer cylinder. The spindle is driven by a synchronous motor through a calibrated spring, and the deflection of the spring is displayed by the viscometer. The exact volume of sample was placed in the annular space between the spindle and the cup, and measurements were taken at different rotational speeds of the spindle. The concentric cylinder can be configured in to eight different measurement systems. The torque developed on the inner cylinder due to sample is directly related to the viscosity and should be in a range of 0.5−9.5 mN m for the accurateness of the measurement. The gap between the inner bob/vane and outer cylinder is thin so that there is almost a constant gradient of shear over it. A 40% by weight sample was placed in the

Table 1. Proximate Analysis of Coal Sample-X

constituents (%) (adb)	253 μm	182 μm	127 μm	90 μm
untreated coal				
fixed carbon	38.86	42.7	45.68	43.75
volatile matter	20.6	18	16	16.4
moisture	2.04	1.1	1.15	2.45
ash	38.50	38.20	37.17	37.40
microwave treated coal (900 W, 30 s)				
fixed carbon	40.85	42.59	44.3	42.9
volatile matter	19.9	18.3	18.3	19
moisture	1.15	1.3	1.2	1.2
ash	38.10	37.81	36.20	36.9
microwave treated coal (900 W, 60 s)				
fixed carbon	40	41.27	44.13	43.9
volatile matter	21	20	19	20
moisture	1	1.5	1.05	1.1
ash	38	37.23	35.82	35
microwave treated coal (900 W, 90 s)				
fixed carbon	39.25	41.55	43.4	43.75
volatile matter	21.9	20.3	19.7	19.6
moisture	1.15	1.25	1.3	1.05
ash	37.7	36.9	35.6	35.6
microwave treated coal (900 W, 120 s)				
fixed carbon	43.56	41.5	44.7	44.45
volatile matter	18.4	21.1	18.5	19.4
moisture	1.04	1	1.5	1.45
ash	37	36.4	35.3	34.7

cylinder of the viscometer after placing the bob/vane properly with in the cylinder. A whole viscosity measurement need about 120 s. On the basis of the computer program, when the recorded numerical values under different shear rates were input to the computer, the apparent viscosity at a 224.60 s^{-1} shear rate would be obtained. All rheological measurements of the samples were done in a shear rate range of 0−300 s^{-1}. In all the tests, the pH value of the slurry varied between 7.05 and 7.25, and the temperature was kept constant at 25 ± 2 °C. Plots of shear rate and shear stress were obtained directly. The same procedure was repeated for all the samples. In this study, all samples were measured by the use of a special cone, C14 system which has a gap width of 0.7 mm between the inner and outer cylinders.

3. Results and Discussion

Proximate and Ultimate Analysis. The proximate analysis of the microwave pretreated and untreated coals (coal-X and Y) for different sizes are carried out and typical values for coal-X are presented in Table 1. Table 2 shows the ultimate analysis of the microwave pretreated and untreated coals of 90 μm (both coal-X and Y). The contents of C, H, N, and S of the coal samples were measured using a LECO CHNS 932 Elemental Analyzer. The oxygen contents of samples were calculated by difference. Results show that the ash content of microwave-treated coals is lower than that of original untreated samples. This lowering of ash content due to microwave treatment may be due to the partial removal of mineral matter finer than 90 μm by grinding and screening. Due to microwave treatment, mineral matter is heated faster than coal; as a result, the binding force in minerals is weakened and the fines form while being ground in a ball mill. Also, the ash content increases as the particle size increases. This is a feature of this coal, as ash tends to concentrate in large fractions with Indian coals. The ash content of coal-X is more than that of coal-Y, as it contains more mineral matter. However, the moisture content of microwave-treated coals was found to be lower than that of untreated coal. The same trend has been observed for coals of different particle sizes. The volatile matter and fixed carbon of microwave-treated coals are found to be more than those of untreated coals for both coal-X and Y, which is quite obvious, as discussed earlier, due to partial removal of mineral matter.

Particle Size Analysis. The particle size distributions of the samples were determined by Mastersizer 2000. Water was used as the dispersion medium. The automatic particle size analysis can be obtained in the measuring range 0.1−1000 μm in suspension. The data recording/result presentations are obtained by an MS Windows program. A comparison of the particle size distribution and some size parameters of untreated and microwave treated coal of 60 μm particles are shown in Figure 2. The particle size is corresponding to the accumulative mass percentage of particles that pass the size on the distribution curve. D_{10}, D_{50}, and D_{90} refer to the particle sizes that 10, 50, and 90% of coal particles by weight can exactly pass. As seen from Figure 2 data, the microwave-treated coal sample contains significant amount of finer particles than microwave untreated coal sample. Also, it has found that the surface area of microwave treated coal is more than that of untreated coal. It has concluded that the particle size distribution of coal after microwave treatment is more depended on the coal grinding process than before microwave treatment. Almost the same trend of size distribution has been observed for other samples.

Density Measurement. Densities of different coal particles for both microwave-treated and untreated coals have been measured. A typical value for 60 μm particles was found to be 1.6 for untreated X coal and 1.48 g/cm^3 for MW-treated coal for a 2 min duration. It has been found that the density of microwave-treated particles was found to be less than that of untreated particles for the same size. For coals, density gradually

Table 2. Ultimate Analysis of Coal-X and Y (90 μm)

Coal-X			
constituents (%) w/w, dry basis	untreated coal	microwave treated coal (900 W, 1 min)	microwave treated coal (900 W, 2 min)
carbon	59.54	60.52	57.25
hydrogen	3.86	3.78	3.59
nitrogen	2.60	2.65	2.56
sulfur	0.52	0.48	0.43
oxygen	33.48	32.57	36.17
Coal-Y			
constituents (%) w/w, dry basis	untreated coal	microwave treated coal (900 W, 1 min)	microwave treated coal (900 W, 2 min)
carbon	58.41	61.48	59.22
hydrogen	3.69	3.87	3.45
nitrogen	2.49	2.79	2.48
sulfur	0.58	0.47	0.47
oxygen	34.83	31.39	34.38

increases with increased particle diameter. It may be due to the effect of microwave treatment of mineral matter resulting in the decomposition of pyrite and possibly the conversion of α-silica to β-silica, which may reduce the erosion considerably.

Effect of Duration of Microwave Treatment on the Temperature of Coal. It has been observed that the coal temperature increases with the duration of microwave treatment, which was measured by a thermocuple. The rise in temperature of coal-X for the same treatment period is greater than that of coal-Y as shown in Figure 3, which is obvious, as the percentage of mineral matter in coal-X is higher than in coal-Y and microwave energy absorption of minerals is 2−3 times faster than that of carbonaceous matter. Also, the ash content of a material is 1.5 times that of the mineral matter.

Rheological Characteristics of Coal−Water Slurries (CWS). Effect of Shear Rate on Shear Stress for CWS. The effect of the shear rate on shear stress for microwave-treated and untreated coal-X of 60 μm, 50% solid concentration, has been investigated. All suspensions have been found to follow pseudoplastic behavior, which is higher at higher concentrations. The shear stress for untreated coal has been found higher than that of microwave-treated coal. This may be due to changing surface characteristics of microwave-treated coal and the effects of microwave treatment on the physicochemical properties and microstructures of coal−water slurries. It has also been found that MW-treated and untreated coal suspensions of 30 and 40% concentration of coal-X and Y of different fractions show pseudoplastic behavior. But, the pseudoplastic behavior for 90 and 60 μm coals is more regular than that for the 253 μm particles.

Effect of Shear Rate on Apparent Viscosity. Figure 4 shows the effect of slurry concentration on apparent viscosity (point viscosity) for untreated and microwave-treated coals. There is an exponential decrease in viscosity with increased shear rate for all types of coal samples. The behavior of 60 μm particle size for 50% slurry is more regular than of 253 μm of 30%. It can be seen that the viscosity of microwave-treated coal is

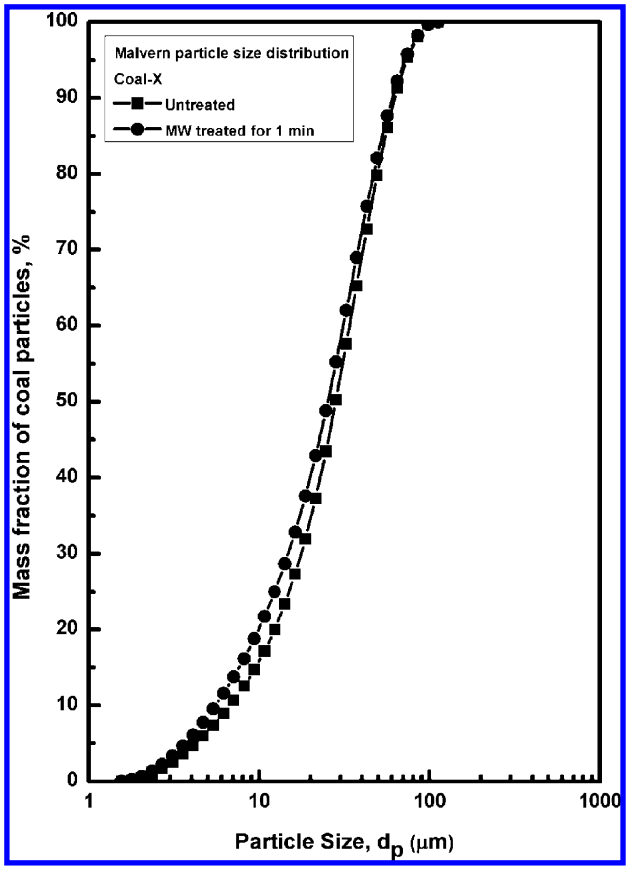

Figure 2. Malvern particle size distribution of coal-X (60 μm) before MW treatment and after MW treatment [900 W, 1 min].

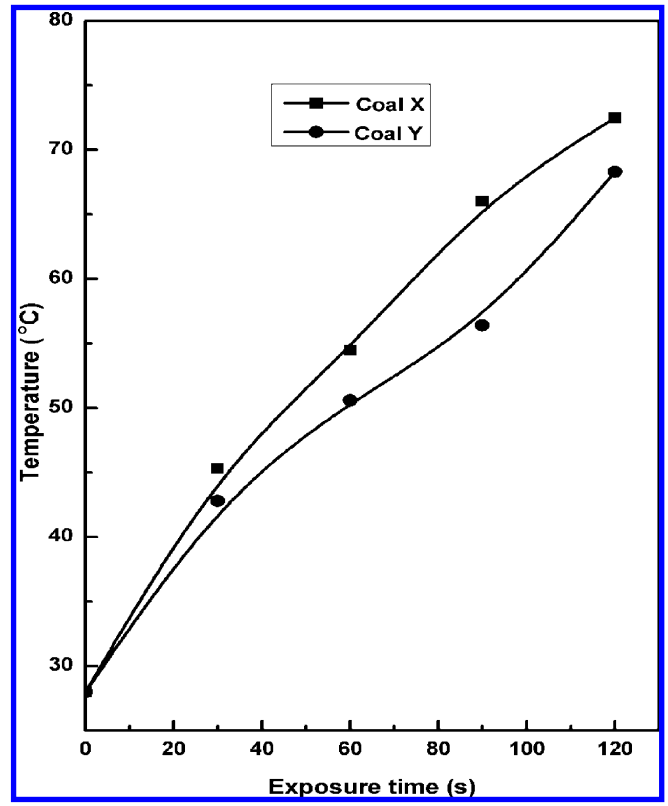

Figure 3. Effect of duration of MW treatment on the temperature of coal.

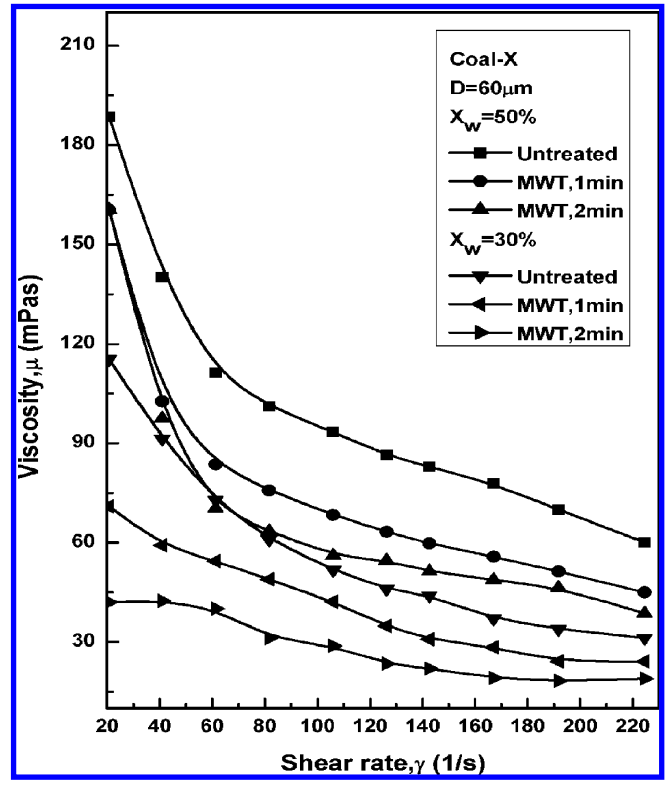

Figure 4. Effect of slurry concentration on apparent viscosity for MW treated and untreated coal.

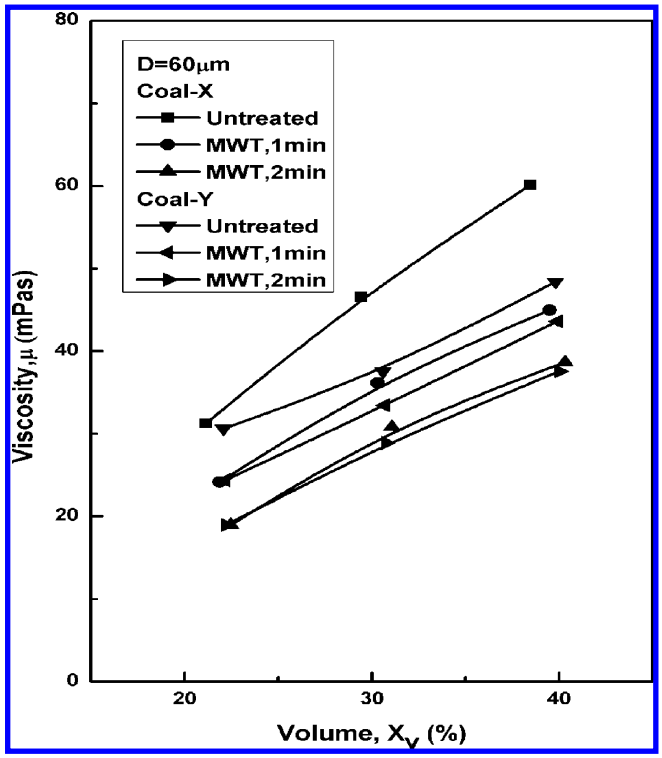

Figure 5. Effect of slurry concentration on viscosity of MW-treated and untreated coal.

always lower than that of untreated coals. The decrease in viscosity of microwave-treated coals might be due to the decrease in irregularity of the particles after grinding and surface smoothing of treated samples. The viscosity is always higher for 50% both treated and untreated slurries than that of 30% slurries at a constant shear rate.

Effect of MW Treatment and Slurry Concentration on Viscosity. Figure 5 shows the effect of slurry concentration on viscosity for microwave-treated and untreated coal-X and Y of 60 μm particles. It has found that the viscosity at infinite shear rate (viscosity calculated at constant and relatively high shear rate, i.e., 224.60 s^{-1}) increases with increased slurry concentration. Generally, shear stress increases with increased shear rate up to a certain point, after which it becomes almost constant. The viscosity at this constant shear stress (infinite shear rate, 224.60 s^{-1}) is termed as μ_∞. From the figure, it has been found that the viscosity of microwave-treated coals was lower than that of the untreated coals and it increases with the particle concentration.

Figure 6 show the effect of slurry concentration on average viscosity for microwave untreated coal-X and Y of different fractions. There is an increase in viscosity with decreased particle sizes and increased particle concentration. It indicates that large surface area or higher particle concentration lead to corresponding decreases in fluidity of the system. The increase in viscosity with particle concentration can be attributed to an increase in particle interaction of a hydrodynamic and/or colloidal nature. The viscosity increases with decreased particle size can be explained by the fact that the smaller particles have a larger surface area, with the result that more liquid is required to wet the surface and hence available fluid for flow is reduced and viscosity is increased. It has also been observed that the viscosity of coal-X was more than that of coal-Y. This may be due to the greater content of mineral matter.

Variation of Shear Rate with Shear Stress in a log−log Plot. A log−log plot of shear stress vs shear rate was done to find the values of constants "*k*" and "*n*". It has been found that the value of *n* varies from 0.31−0.64 and *k* varies from 0.16−0.82. It has been also observed that 30% suspensions by weight showed a higher value of *n* than 50% suspensions. The developed correlation is applicable to the microwave-treated

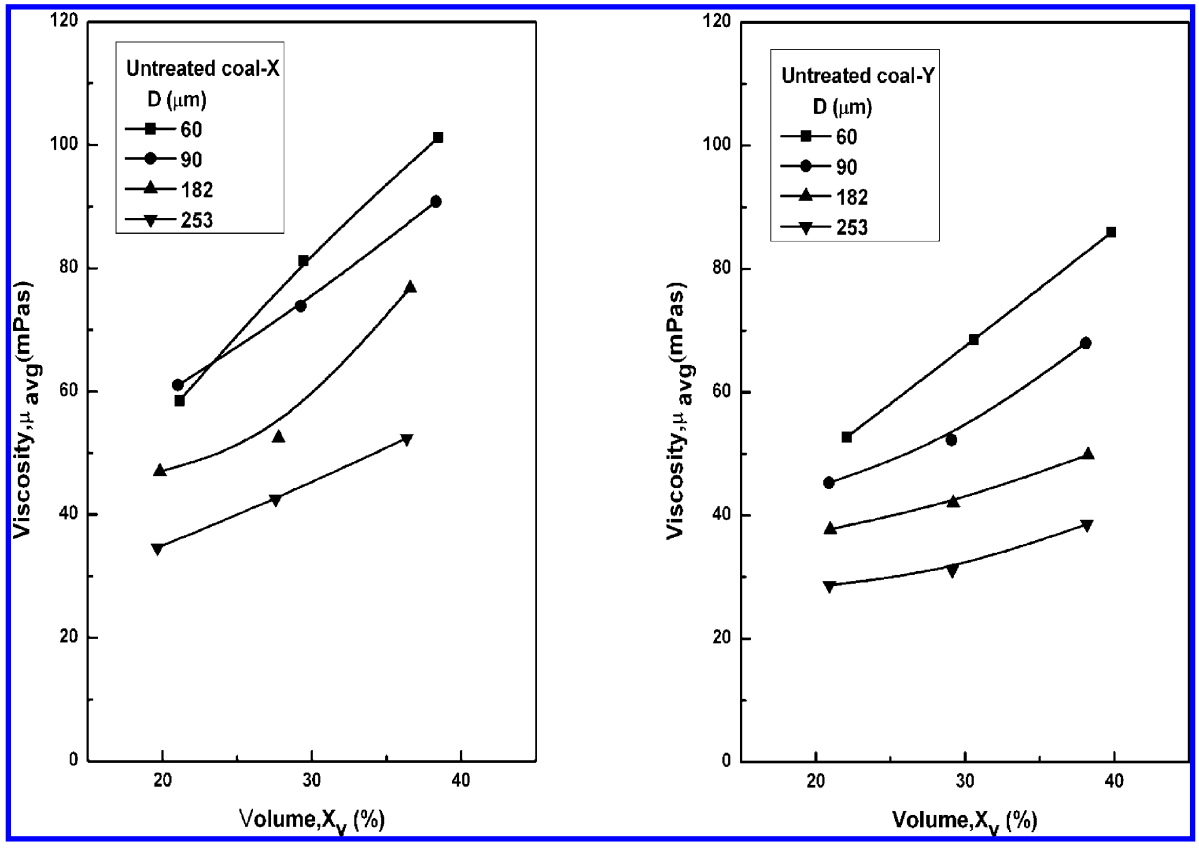

Figure 6. Effect of slurry concentration on average viscosity for untreated coal-X and Y.

Table 3. Scope of the Factors for Apparent Viscosity of Coal-X and Y

Sl no.	name of the variable	variable (General symbol)	factorial design symbol	min. level (−1)	max level (+1)	magnitude of variables
1	particle diameter (μm)	D	A	60	253	60, 90, 127, 182, 253
2	solid concentration (%)	X_w	B	0.30	0.50	0.30, 0.40, 0.50
3	shear rate (1/s)	y	C	20.72	224.60	20.72, 41.03, 61.33, 81.71, 106, 126.38, 142.61, 167.13, 191.76, 224.60

coal-X and Y for the 60 to 253 μm coal particles and at a slurry concentration of 30−50% by weight. The terms k and n are rheological constants, referred to as fluid consistency coefficient and flow behavior index, respectively. The higher the value of k, the more viscous the slurry. The departure of n from unity indicates the degree of deviation from Newtonian behavior. From experimental findings, it has found that almost all the slurries are pseudoplastic in nature and the 50% slurry behaves longer as a non-Newtonian fluid than the 30% slurry. The value of n obtained for coal-X seems to be less than that for coal-Y.

Development of Correlation. For Average Viscosity ($\mu_{avg.}$). A correlation based on factorial design analysis[12] has been developed for the average viscosity. The method of factorial design analysis bring out the interaction effects of variables, which would not be found otherwise by conventional experimentation and to explicitly find out the effect of each of the variables quantitatively on the response. The scope of the factors considered for factorial experimentation are for particle diameter (D), solid concentration (X_w) with a factorial symbol (A) with a minimum level (−1) of 60 and 0.3 and at maximum level (+1) of 253 and 0.50, respectively. The magnitude of variables for particle size are 60, 90, 127, 182, and 253, whereas for solid concentration, they are 0.3, 0.4, and 0.5, respectively. The variables, which affect average viscosity are particle diameter and solid concentration. Thus total numbers of experiments required at two levels for the two variables is four for average viscosity.

The model equations are assumed to be linear, and the equations take the general form

$$Y = (b_0 + b_1A + b_2B + b_3C + ... + b_{12}AB + b_{13}AC + ... + b_{123}ABC) \quad (3)$$

(i) Coefficients are calculated by the Yates technique

$$b_i = \sum \frac{\alpha_i Y_i}{N} \quad (4)$$

Where, b_i is the coefficient, Y_i is the response, α_i is the level of the variable, and N is the total number of treatments.

(ii) Calculations of the level of variables for average viscosity

(A) Level for particle diameter = (particle diameter −142.4)/96.5

(A) Level for solid concentration = (solid concentration − 0.4)/0.1 The following equation has been obtained:

$$Y = (0.062 - 0.018A + 0.015B - 0.006AB) \quad \text{for coal-X} \quad (5)$$

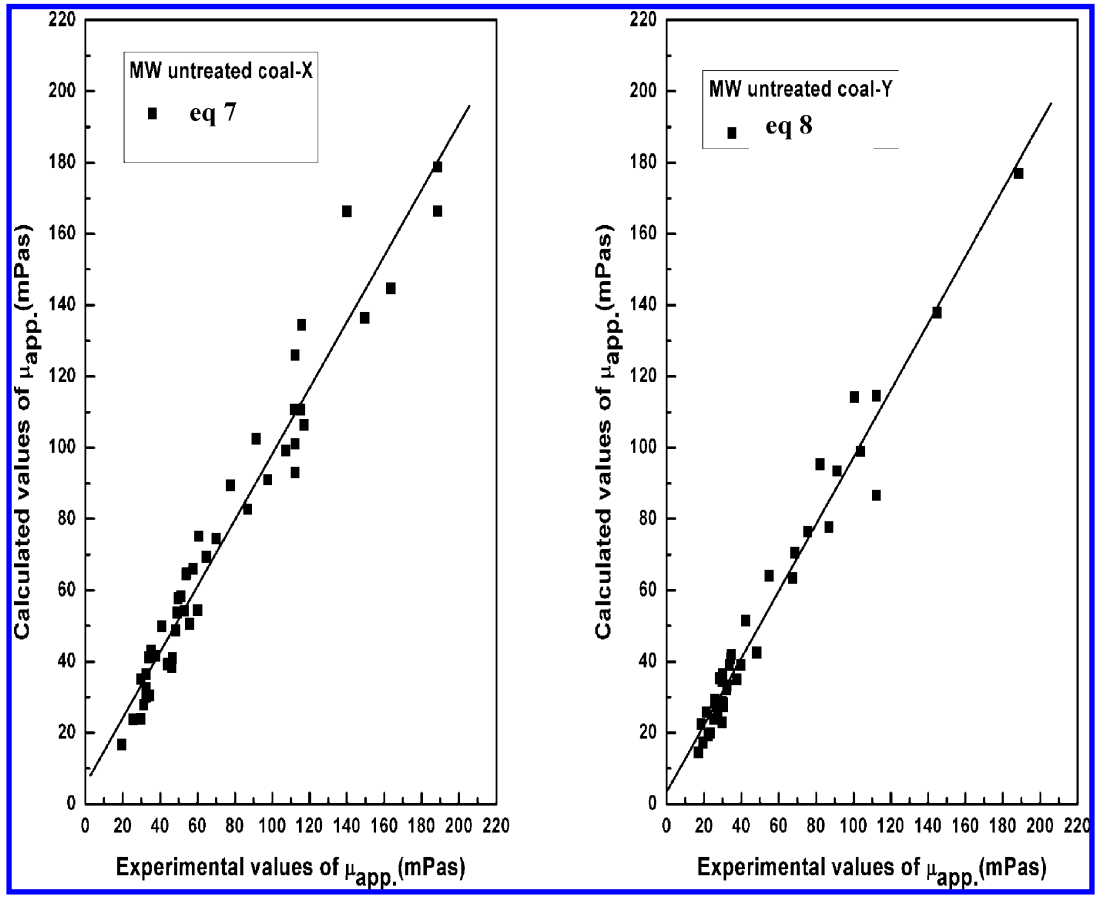

Figure 7. Comparison of experimental values of apparent viscosity with those calculated from eqs 7 and 8.

$$Y = (0.052 - 0.018A + 0.011B - 0.006AB) \quad \text{for coal-Y} \tag{6}$$

The value of the coefficients indicates the magnitude of the effect of the variables, and the sign of the coefficient gives the direction of the effect of the variable. That is, a positive coefficient indicates an increasing in the value of the responses with increase in the value of the variable and a negative coefficient shows that the response decreases with increase in the value of the variable. The calculated values of average viscosity from eqs 5 and 6 are compared with experimental data taken at conditions other than those used for development of correlations, and they are found to agree within a standard deviation of ±10%. The comparison of data for average viscosity for coal-X and Y is presented in the Figure 6.

Apparent Viscosity (μ_{app}). The scope of the factors considered for factorial experimentation is presented in Table 3. The variables which affect apparent viscosity are particle diameter, solid concentration, and shear rate. Thus total numbers of experiments required at two levels for the three variables is eight for apparent viscosity.

Calculations of the level of variables for apparent viscosity

(A) Level for particle diameter = (particle diameter − 142.4)/96.5

(B) Level for solid concentration = (solid concentration − 0.4)/0.1

(C) Level for shear rate = (shear rate − 116.33)/101.94 The following equation has been obtained:

$$Y = (0.081 - 0.018A + 0.017B - 0.044C - 0.008AB + 0.009AC - 0.006BC + 0.005ABC) \quad \text{for coal-X} \tag{7}$$

$$Y = (0.071 - 0.022A + 0.016B - 0.041C - 0.01AB + 0.013AC - 0.01BC + 0.007ABC) \quad \text{for coal-Y} \tag{8}$$

The comparison of data for apparent viscosity for coal-X and Y is presented in Figure 7.

4. Conclusions

Detailed experimental investigations have been carried out for high ash Indian coals for rheological characteristics of coal slurries. Proximate and ultimate analyses show that ash contents increase with increases in particle size. Fixed carbon and volatile matter for microwave treated coal were found to be more than that of untreated coal. Coal temperature increases with the duration of microwave treatment. The temperature of high ash coal was found to be more than low ash coal subjected to the exposure of microwave energy. Rheological characteristics for coal slurries were found to exhibit shear-thinning behavior with an apparent viscosity decreasing with increasing shear rate, which is higher at higher concentrations. It can be seen that the viscosity of microwave-treated coal is always lower than that of untreated coals. The value of n obtained for coal-X seems to be less than that for coal-Y. It has been found that the value of n varies from 0.31−0.64, and k varies from 0.16−0.82. A factorial plan was used, and empirical equations were obtained which correlate rheological characteristics with the particle diameter and mass fractions of the slurry components. The correlations have been found to be encouraging and highly significant.

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Nomenclature

b_i = coefficient
C_1, C_2 = constants related to a measuring system
D = particle diameter (μm)
k = constant in power law or consistency index
M = shear stress related torque (mN m)
N = total number of treatments
n = viscosity index
V = rotational speed of a rotor (Bohlin viscometer) (rpm)
X_v = volume fraction of solid in suspension (%)
X_w = weight percentage of coal in slurry (%)
Y_i = response

Greek Letters

γ = shear rate (s^{-1})
τ = shear stress (Pa)
τ_0 = yield stress (Pa)
μ = viscosity (Pa s)
μ_∞ = slurry viscosity at infinite shear rate (Pa s)
μ_{app} = apparent viscosity (Pa s)
μ_l = viscosity of suspending medium (Pa s)
μ_{sl} = slurry viscosity (Pa s)
α = one form of silica in mineral matter of coal, dimensionless
α_i = level of the variable
β = another form of silica in mineral matter of coal, dimensionless
ρ = density (kg/m^3)

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