Influence of solid concentration on rheological characteristics of fly ash-water suspension

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Abstract. Coal is the primary source of energy in almost every nation. As the energy demand is increasing, a greater amount of ash is generated as a result of increased coal consumption. The present study is aimed to investigate the physical, chemical and rheological properties of fly ash and its suspension with water for the design of a slurry transport system. The mineralogical composition of the fly ash shows large fraction silica and alumina. The rheological analysis shows that at lower concentrations (10-30%), the slurry behaves like a Newtonian fluid whereas it behaves like a non-newtonian fluid at higher concentrations (40-60%). The rheological characteristics can further used for the prediction of flow characteristics of the slurry in a horizontal pipe.

1. Introduction
Coal is the only solid fuel on the Earth, which has the highest energy density and this is because of the process of its occurrence in the Nature. In spite of the uneven distribution of coal throughout the world, it is the primary source of energy in every nation in the world. In India, as per the official report,[1] around 59.2% of energy demands are met by the different coal based power stations. The Indian coal reserves are mostly young and possess low quality high ash coal (~40% ash). Many research groups in India are working on the development of efficient techniques to utilize the ash as much possible. The rheology is defined as the study of deformation and flow of matter under the action of shear forces. The solid-liquid slurry flow behaviour in the slurry pipeline depends upon the rheological characteristics of the slurry suspension.[2] The rheological characteristics of the slurry suspension play a very important role in slurry transportation system design. Many investigators [3–5] have studied the rheological behaviour of slurry suspensions of different materials. In most of the cases, it has been found that there is a great change in the rheological characteristics of the fly ash slurry when the solid concentration increases beyond 30%.[6–10] There is a great variation in the properties of coal with the variation in its place of occurrence and hence there is also a variation in the properties of the ash at different places.[11] Presently, there is no empirical correlation, by which the rheological characteristics of the ash-water suspension can be estimated. This is because the rheological characteristics are dependent on the physical properties such particle size, particle shape, wettability, settling nature, etc. and also on the chemical composition and pH of the slurry. The head loss can be calculated on the basis of flow rate-pressure drop relationship of the pipeline for solid–liquid mixture flow.[12–15] So, the aim of this study is provide an authentic set of data related to the physical chemical and rheological characteristics of the fly ash and its suspension in order to design a better slurry transport system.

2. Experimental procedure:
The sample of fly ash is collected from the ash disposal unit of a Nabha Power, Rajpura. The physical properties of fly ash are studied using standard test setups. The particle size distribution (PSD) is
measured using standard sieve shaker with sieve sizes ranging from 1000 micron to 53 micron. For this, a measured quantity of sample is put into the top sieve and is closed with a lid. After running the shaker for 20 minutes, the sieves are disassembled and the weight of the sample retained by each sieve is measured. From the PSD, the median diameter or d50 is calculated to be a particle size, such that 50% (by weight) particles are finer or coarser than it.

In order to calculate the static settling concentration of the fly ash, the gravitational settling method is used. The test is performed using a 100ml graduated cylinder. The slurry is so prepared that the concentration of fly ash to water is 30:70 by weight and the total volume of the slurry is 100ml. The initial volume of slurry in the present case is 100ml. The volume of settled slurry is measured at regular intervals for 15 minutes and afterwards this interval is increased by multiple times.

The JOEL JSM-6510LV model of SEM (Scanning Electron Microscope) is used to see the shape of the ash particles, which uses a high resolution microscope. The beam of electrons scattered by the particles, is captured by the low vacuum electron detector and an image of the same is generated. The process is carried out in very sophisticated environments like vacuum, so that no foreign particle interfere the analysis. Due to non-conducting nature of the fly ash particles, they are initially coated with gold. For the present analysis, the particles are visualized to a magnification of x500. To study the chemical and mineralogical composition of the fly ash, the EDS (Energy dispersive X-ray spectroscopy) is used. The different components of the fly ash reflect the incident X-rays with different frequencies and hence different energies. The spectroscope is pre-calibrated with, the energy corresponding to a known pure compound. For the present analysis, a very small sample of fly ash is used to know its composition. The pH test is done to know the chemical nature of the slurry with respect to the variation in the solid concentration. The pH of the slurry solution is measured using a standard pH meter, which is initially calibrated with water, assuming the pH of water as 7. The calibrated pH meter is then used to measure the pH of the fly ash slurry, which is displayed on the digital screen of the pH meter.

In order to investigate the rheological characteristics of fly ash water suspension, the fly ash sample having particle size distribution as indicate in Table 1 is used. The required quantity of ash is mixed in 100 ml of water with the help of a glass rod for the preparation of the slurry. The stirring is carried out gently in order to avoid particle attrition. The concentration of the slurry, Cw varied from 10-60% by weight. The rheological tests are performed using Anton-Paar RheolabQC rheometer, which works on Searle principle. For rheological characteristics, the shear rate is varied from 0 to 500 s⁻¹ and corresponding shear stress is measured. The rotating bob and cup assembly is used for the present investigation. The prepared slurry is poured into the cylindrical cup and then the cup is coupled to the bob. The rheological study is carried out at a constant temperature of 33°C. In order to avoid the error in the analysis due to the settling of the slurry, the test is performed for a total of 3 minutes and 10 seconds including a waiting time of 10 seconds.

3. Results and discussion
3.1 Physical and chemical properties of fly ash
Table 1 shows the particle distribution for fly ash. It has been found that around 20.7% particles are finer that 53µm and 61.2% particles are finer than 75µm. Nearly 25% of the particles lie in the size range of 75-150 µm and almost 14% particles are coarser than 150 µm. The largest particle is coarser than 250µm but, only a small fraction (1.4%) of such particles is present in the sample. The median diameter or d50 for the sample is calculated to be 68.1µm. The static settling concentration test is conducted for a total of 1200 minutes as the settling beyond this time is negligible. The results of the settling concentration with time are presented in Table 2. Initially, due to higher potential energy of the particles, the settling rate is higher. The potential energy of the particles decreases, as the particles move down and hence the velocity. Due to this the settling rate decreases with the passage of time. For the optimal condition of slurry transportation, the solid concentration can be 5-10% lower than the maximum value of the static settled concentration. From Table 2, it is found that the final maximum static settled concentration Cw of the fly ash slurry is found to be 60.2 %. The results provided by the SEM analysis (Figure 1) at x500 magnification show that the ash particles are
spherical in shape. The SEM micrograph shows that there is a small fraction of hollow spheres, known as cenospheres. These cenospheres are mostly present in the form of agglomerates with the coarser particles. EDS (Energy dispersive X-ray spectroscopy) is used to know the mineralogical composition of fly ash. The data from the EDS study (Figure 2) indicates a high fraction of silica (SiO$_2$) and alumina (Al$_2$O$_3$) in the fly ash. Silica and alumina together account for ~81% of the ash mass. Loss of ignition (LOI) fraction for the unburnt carbon is estimated as per ASTM-311 standard, which is found to be 1.32%. The detailed mineralogical composition of the sample is shown in Table 3 and Figure 3. The pH of the slurry is tested using electronic pH meter. The pH values depicted in Table 4 and Figure 4 show that the fly ash slurry is almost neutral in nature for all the tested solid concentrations. The maximum and minimum values of pH are observed to be 6.67 and 6.15 for 10% and 60% solid concentration respectively. This indicates that the ash contains a greater fraction of acidic (weak) minerals.

Figure 1 SEM micrograph of fly ash

Figure 2 EDS spectrum of fly ash sample

Figure 3 Chemical composition of fly ash

Figure 4 pH analysis of the fly ash slurry

In the previous investigation by [16], it is observed that the acidic nature of a slurry directly relates to the hydrophobic nature of the suspended particles. The hydrophobic nature of ash particles does not allow water particles to enter into its pores, so most of water lies around the particle. This leads to the overall reduction in the viscosity of the slurry. In the present case, the pH of the slurry is considerably constant and almost close to the neutral value, so the pH of the slurry will not influence the rheological characteristics of the suspension.

3.2 Rheological characteristics

The rheological characteristics for the different concentrations of the fly ash are analysed for different values of the shear rate. In the case of lower concentrations such as 10%, 20% and 30%, the slurry exhibits a Newtonian behaviour. The shear stress shows an increasing trend with the increase in the concentration of the solids.
**Table 1** Particle size distribution

| Particle size (micron) | 355 | 250 | 150 | 106 | 75 | 53 |
|------------------------|-----|-----|-----|-----|----|----|
| % finer                | 100 | 98.6| 86.1| 79.6| 61.2| 20.7|

**Table 2** Static settling concentration at different time intervals

| Time (Min) | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 15 | 20 | 30 | 60 | 180 | 1200 |
|------------|---|---|---|---|---|---|---|---|---|----|----|----|----|----|----|----|-----|------|
| Css % (by weight) | 30 | 31.1 | 32.4 | 33.3 | 34.5 | 35.9 | 37.1 | 39.9 | 40.7 | 41.2 | 41.7 | 42.1 | 42.6 | 44.2 | 46.4 | 51.1 | 57.4 | 59.3 | 60.2 |

**Table 3** Chemical composition of fly ash

| Component | SiO₂ | Al₂O₃ | FeO | CaO | CO₂ | MgO | CuO | LOI | Others |
|-----------|------|-------|-----|-----|-----|-----|-----|-----|--------|
| % age     | 45.2 | 36.2  | 5.41| 3.11| 2.76| 2.3 | 1.6 | 1.32| 2.04   |

**Table 4** pH of the slurry at different concentrations

| Cw % | 10 | 20 | 30 | 40 | 50 | 60 |
|------|----|----|----|----|----|----|
| pH   | 6.67| 6.6 | 6.51| 6.4 | 6.28| 6.15|
In Figure 5, it is clearly represented that as the concentration is increased beyond 30%, there is a great change in the rheological behaviour of the slurry, i.e. the Newtonian behaviour is no longer exhibited by the slurry. This is because at higher concentration, there is more drag in two adjacent particle layers due to the increased adhesiveness of the solid particles. The rheological behaviour of the slurry can be called as Bingham plastic type Non-Newtonian fluid.

Figure 5 Shear stress vs. shear strain curves for different solid concentration of fly ash slurry.

![Shear Stress vs. Shear Strain Curves](image)

This behaviour can be represented by the following equation:

\[ \tau = \tau_y + \eta \frac{du}{dy} \]

Where \( \tau \) - Shear stress, \( \tau_y \) - Bingham yield stress, \( \eta \) - Bingham viscosity and \( \frac{du}{dy} \) - Shear rate

Figure 6 The fitted trend lines for the experimental data of the fly ash water slurry

Figure 7 Viscosity (\( \eta \)) vs. shear rate curves for the fly ash slurry at different solid concentration

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**Table 5** Yield stress and Bingham viscosity values for different solid concentration

| Cw % | \( \eta \)  | \( \tau_y \)  | \( \tau = \tau_y + \eta \frac{du}{dy} \) | \( R^2 \) |
|------|------------|-------------|---------------------------------|--------|
| 40   | 0.0131     | 1.6546      | \( \tau = 1.6546 + 0.0131 \frac{du}{dy} \) | 0.9946 |
| 50   | 0.0201     | 3.8114      | \( \tau = 3.8114 + 0.0201 \frac{du}{dy} \) | 0.9902 |
| 60   | 0.0314     | 6.6465      | \( \tau = 6.6465 + 0.0314 \frac{du}{dy} \) | 0.992  |
In case the above equation is to be used for a Newtonian fluid, the value to yield stress is taken zero. In Bingham plastics, it is found that with the increase in the shear rate, the viscosity of the fluid shows a decreasing trend. So, it can be interpreted that the increase in flow velocity will lead to greater shear, which will result into the reduction in the viscosity of the slurry and hence a lesser rise in the pressure drop. The values of Bingham yield stress are found to be different for different solid concentration, as shown in Table 5. Different linear equations can be fitted for different concentration in order to predict the results at greater values of shear rate. In the previous investigations by Kumar et al. 2014 [17] Kumar et al. 2016 [13], similar trends of the shear stress vs. shear strain have been reported. Figure 6 shows the trend lines fitted for the above data, each having R² (correlation coefficient) value ≥0.99. Figure 7 represents the variation of viscosity with respect to the shear rate. In the case of lower solid concentration, due to the Newtonian behaviour, the viscosity is almost constant at the shear rates >60 s⁻¹. Whereas at higher concentrations, due to Non-Newtonian behaviour, the value of viscosity is quiet high, which shows a decreasing trend with increase in the shear rate. The initial value of the viscosity is high because the static high density slurry has a large amount of adhesive (attractive) force, in order to overcome this force, large amount of shear stress is required, which in other words is known as the yield stress or the Bingham yield stress. In the present case, it has been found that the value of yield stress also increases with increase in the solid concentration. The viscosity of the slurry has also shown an increasing trend with increase in the solid concentration.

4. Conclusions

Present work highlights various properties and characteristics of fly ash and fly ash slurry, for the design of a hydraulic transport system. Following conclusions were drawn on the basis of the present study:

- The maximum settled concentration of fly ash slurry is 59.34%.
- pH analysis of the slurry at all concentrations shows a neutral behaviour.
- The fly ash samples are mainly constituted of silica (SiO₂), alumina (Al₂O₃) with low amounts of Fe₂O₃ and little amounts of CaO, MgO, and MnO etc. The presence of high proportion of silica and alumina improves the strength and CaO improves the cementing property which tends the bulk utilization of fly ash in the field of civil engineering, geotechnical, cement materials and stowing etc.
- The fly ash slurry behaves like a Newtonian fluid at low solid concentrations (<30%), whereas it shows a Non-Newtonian behaviour at high solid concentrations (>30%).
- The maximum viscosity is found to be 0.2471 Pa s for the sample containing 60% solid concentration.
- The maximum yield stress of 6.6465 Pa is observed in the case of 60% solid concentration.

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