Physico-chemical characteristics of fly ash samples from thermal power plants of Orissa

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Some physico-chemical characteristics of three fly ash samples have been studied with an objective to create a data base for different fly ash samples in the country. It has been observed that relevant physical properties of the samples show systematic variation with particle size distribution. Thus, while apparent density and optical or geometrical area increase with fineness of the particles from sample 1 to 3, true density, voidage and void volume exhibit opposite behaviour. Batch leaching studies indicate that whereas for samples 1 and 2 most of the trace metals (except Cu) leach out rapidly from the surface of the ash particles in the pH range 6.4-7.5, under acidic conditions (pH ~ 4.5) the rates of leaching of most trace metals, except Co and Cd, are relatively slow for sample 3. The pH values of zero charge of the three major oxide constituents namely, SiO₂, Fe₂O₃ and Al₂O₃, as determined by the solid addition method, conform more or less to the literature values of 2.5, 7.0 and 9.5 respectively. Viscosity measurements of aqueous suspension indicates that sample 3 is least hydrated in water and therefore shows low viscosity value even at high concentration of slurry.

Fly ash is the product of combustion of coal in boiler furnace of the thermal power plants. Because of the abundant reserve of coal in India, thermal power contributes more than 75% of the total power generation in the country. As more and more thermal power plants are being set up in the country to meet the ever increasing demand of power, it is estimated that 115-120 million tonnes of fly ash will be generated annually in the country even before the end of this century¹. Unless a substantial portion of this fly ash finds some use, this will lead to a very serious environmental crisis.

However, during the last several years, the R&D efforts on the utilization of fly ash has gained considerable momentum and several processes leading to the bulk use of fly ash are likely to be put into commercial exploitation¹². Some of the important possible uses of fly ash are in the production of building materials (bricks, cement, concrete etc), land and mine fill, construction of roads and highways, embankments etc. For any of the above possible uses of fly ash, the material has to be characterized and standardized scrupulously and all the fly ash produced in the country must be categorized according to their physico-chemical properties. It is therefore imperative to create a detailed data base for the characteristics of fly ash generated in all the major thermal power plants in the country. The present work is a part of the ongoing in-house project of the laboratory on the characterization and utilization of fly ash from different thermal power plants in the country. In the first instant, the work was initiated with fly ash samples from two thermal power plants in Orissa, namely, Talcher and National Aluminium Company (NALCO), situated in the district of Angul, about 130 km from Bhubaneswar. Both the plants use coal from Talcher Coal Field, but the technology of combustion used in the two plants is probably different. The results of characterization studies carried out on fly ash samples of these two thermal power plants are presented here.

Experimental Procedure

Materials

Altogether three samples have been used in the present work. Two of these have been collected from Talcher thermal power plant and are: (i) sample collected from hopper below the electrostatic precipitator (ESP), before coming in contact with water jet injection; (ii) ash sample collected from the settling pond. The third sample

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(sample 3) was from the thermal power plant of the National Aluminium Company (NALCO). This sample was also collected under dry condition from the hopper below the ESP. The physico-chemical properties of the ashes obtained from coal combustion are preserved in such sample, unlike pond ash, where its prolonged contact with water may lead to variation in the properties.

Methods

The three samples have been characterized by the following methods.

Chemical analysis—While the major constituents such as SiO₂, Al₂O₃, CaO, MgO, Fe₂O₃ etc were determined by the procedure recommended by both Indian (IS: 3812) and Japanese standards (JISA 6210), the minor constituents and trace elements were determined by either atomic absorption spectrophotometry (AAS) or inductively coupled plasma emission spectrometry (ICPES).

Particle size—The particle size distribution pattern was determined with the help of a MALVERN particle size analyzer (UK, model 3600).

Surface area—The particle size analyzer also provides specific surface area per unit volume from which area per unit mass also can be calculated using the density data. Surface area determined by this method is essentially outer or geometric area and is closely similar to ‘Blaine area’ obtained by air permeability method.

Density—True densities of the sample were determined by the displacement of toluene in a 25 mL pycnometer at 30°C. The apparent density values were determined by packing 5 g of bulk sample in a 20 mL graduated cylinder after 15-20 times of gentle and uniform tapping and noting the volume with an accuracy of ±0.2 cm³. The mean value of triplicate measurement was taken. The suspension densifies for different solid concentrations in water were determined in a 25 mL pycnometer at 30°C.

Viscosity—Viscosity values of different concentrations of solids in water were determined by a Brookfield viscometer at 30°C and 60 rpm of spindle rotation.

Microscopy—The fly ash samples were examined under transmitted light in a Leitz polarizing microscope (Germany). The samples were also examined under a scanning electron microscope of JEOL make (Japan).

X-ray diffraction—X-ray diffraction pattern of the samples were taken in a Philips diffractometer using CuKα radiation.

Point of zero charge—Points of zero charge (pHₚzc) of the samples were determined by the solid addition method as follows:

In a series of 100 mL plastic bottles, 45 mL of NaClO₄ solution (10⁻¹ or 10⁻⁴ M) was transferred. The pH values of the suspensions were approximately varied from 3.0 to 10.0 by adding known volume of either 0.1 N HClO₄ or 0.1 N NaOH. The total volume of the suspension in each bottle was made up to 50 mL by adding the balance electrolyte solution. The pH values were again noted and designated as initial pH or pHᵢ. 0.5 g of accurately weighed sample was added to each bottle which was immediately closed to avoid the contact with atmospheric CO₂. The suspensions were allowed to equilibrate for 72 h with intermittent shaking. The final pH values were then

Table 1—Physical properties of fly ash samples

<table>
<thead>
<tr>
<th>Properties</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>True density, g cm⁻³</td>
<td>2.09</td>
<td>2.09</td>
<td>1.58</td>
</tr>
<tr>
<td>Apparent or bulk density, g cm⁻³</td>
<td>0.941</td>
<td>0.950</td>
<td>0.975</td>
</tr>
<tr>
<td>Voidage (-)</td>
<td>0.556</td>
<td>0.546</td>
<td>0.383</td>
</tr>
<tr>
<td>Void volume, cm³ g⁻¹</td>
<td>0.584</td>
<td>0.574</td>
<td>0.393</td>
</tr>
<tr>
<td>Geometric surface area, cm² g⁻¹</td>
<td>1754</td>
<td>4400</td>
<td>12186</td>
</tr>
<tr>
<td>Particle size distribution</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(a) 50% finer than, µm</td>
<td>30</td>
<td>9</td>
<td>6</td>
</tr>
<tr>
<td>(b) 80% finer than, µm</td>
<td>56</td>
<td>17</td>
<td>10</td>
</tr>
</tbody>
</table>
Table 2—Chemical composition of the three fly ash samples

<table>
<thead>
<tr>
<th>Constituents (%)</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>57.00</td>
<td>58.92</td>
<td>56.00</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>29.48</td>
<td>28.90</td>
<td>33.07</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>6.20</td>
<td>5.00</td>
<td>4.84</td>
</tr>
<tr>
<td>CaO</td>
<td>1.00</td>
<td>1.10</td>
<td>0.27</td>
</tr>
<tr>
<td>MgO</td>
<td>0.35</td>
<td>0.32</td>
<td>2.10</td>
</tr>
<tr>
<td>TiO₂</td>
<td>1.90</td>
<td>2.04</td>
<td>1.10</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.15</td>
<td>0.24</td>
<td>0.08</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.41</td>
<td>0.40</td>
<td>0.12</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.30</td>
<td>0.30</td>
<td>0.09</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.064</td>
<td>0.000</td>
<td>0.065</td>
</tr>
<tr>
<td>LOI</td>
<td>3.00</td>
<td>2.15</td>
<td>1.60</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>99.854</td>
<td>99.37</td>
<td>99.335</td>
</tr>
</tbody>
</table>

Indian specification (IS-3812) the Blaine area of fly ash should be higher than 2800 cm²/g. Therefore, both particle size distribution and surface area data corroborate each other on the fineness of the samples.

Density measurement—The above observations are also corroborated from density measurements. While the true or particle density value of samples 1 and 2 is same, i.e. 2.09 g cm⁻³, sample 3 shows a very low value of about 1.58 g cm⁻³.

Both particle size and microscopic observation (see below) reveal that sample 3 consists of very fine particles, most of which tend to float when sprinkled on water. All these suggest that the presence of hollow cenospheres is more widespread in this sample than in the samples 1 and 2, thereby showing low value of particle density. The bulk density values of the three samples follow the similar pattern of variation as with particle size distribution, the sample of coarsest size showing lowest value of bulk or apparent density because of larger interparticle void space. The results are shown in Table 1.

Since fly ash is the product of high temperature molten phase of aluminosilicate compounds (glass), the internal porosity and pore volume are very small in such materials. Therefore, it is more appropriate to use the terms voidage (ε) and void volume (ψ), instead of porosity and pore volume. The ψ and ε values can be estimated from the relations:

\[ \psi = \frac{1}{\rho_b} - \frac{1}{\rho_{true}} \]  \hspace{1cm} (1)

\[ \varepsilon = 1 - \frac{\rho_b}{\rho_{true}} \]  \hspace{1cm} (2)
Fig. 3—SEM photographs of sample 1 showing—(a) A typical spherical particle of glassy material. (b) Agglomeration of several spheres to form “pleospheres” or “cenospheres”. (c), (d) Relatively low melting (temperature) phase surrounded by partially molten high temperature phase. Fine particles of refractory oxides or iron oxides are seen adhered to the outer surface of large spheres.

where \( \rho_b \) and \( \rho_{true} \) are the bulk and true (or particle) densities respectively in g cm\(^{-3}\). The results given in Table 1 indicate that \( \gamma \) and \( \varepsilon \) values decrease with decrease in average particle size from sample 1 to 3.

**Microscopic observation**—Extensive microscopic observation under the transmitted light show that all the fly ash samples contain fine unburnt carbon particles of irregular shape and occurs to the extent of about 3-4% by weight (Fig. 2). The non-carbon ash consists of non-crystalline glassy materials, generally spherical in shape due to surface tension and other hydrodynamic effect of the environment. Scanning electron micrographs show that all the three samples commonly consist of glassy spheres of assorted size range with very small quantity of crystalline materials. Fig. 3a illustrates the SEM photograph of a typical spherical particle constituting the fly ash sample 1 from Talcher thermal power plant. The size range of such spheres, however, varies widely from 1 to 100 \( \mu m \). The two important features that can be noted from such spheres are—(i) the presence of certain granular deposits possibly of some refractory oxides or iron oxide or even unburnt carbon, (ii) the presence of holes or vesicles as identified by black spots on the surface\(^6\). These vesicles or holes are believed to be formed by the differential cooling when a low melting phase (\( \text{Na}_2\text{O}/\text{K}_2\text{O}/\text{CaO}-\text{SiO}_2-\text{Al}_2\text{O}_3 \)) solidifies earlier.
than the overall molten droplets (SiO$_2$-Al$_2$O$_3$). Fig. 3b shows a typical agglomerated spheres (cenosphere). Fig. 3c and 3d demonstrates the partial melting of a high temperature agglomerated phase. Relatively low temperature phase therein cools and solidifies earlier forming spheroidal particles and holes in the major matrix.

On the other hand the fly ash sample 3 consists of very fine spherical particles ranging from 1 to 30 μm in size. These spheres generally reveals relatively smooth surfaces, though, some also contain vesicles or holes. Fig. 4a is an unusually large particle having a layered structure. This probably represents a partially converted aluminosilicate mineral rich in iron. The lathe-like structure in Fig. 4b possibly represents a partially decomposed pyrite grain dislodged from coal. But, the occurrence of such material is rather rare in fly ash.

**Fig. 4—SEM photographs of sample 3 showing—**(a) Layered structure of a large aluminosilicate mineral possibly containing high iron, (b) Lathe-like structure of a decomposed pyrite grain dislodged from coal.

**Fig. 5—X-ray diffraction patterns of the three fly ash samples along with the diffraction patterns of some typical high temperature minerals.**

**X-ray diffraction**—Mineral matters present in coal directly control the mineralogy of fly ash. However, during the combustion of coal, its mineral matters undergo considerable change. Though XRD is the most frequently used method to determine the mineralogical characteristics of fly ash, quite a number of reflections are difficult to be assigned unambiguously to any distinct mineral. This is because during the ash fusion a variety of aluminosilicate compounds are formed for which standard literature references are not available. The problem becomes further complicated as many of these compounds are non-crystalline (glass) for which no XRD pattern can be obtained. Fig. 5 suggests that the major minerals present in Talcher fly ash are corundum (α-Al$_2$O$_3$), mullite, sillimanite, tridymite and small amounts of magnetite and fayalite. But those minerals which are present in 5% or less by weight in the sample are difficult to be identified by XRD method. For identifying such minerals chemical analysis data combined with careful microscopic observations are employed.
Table 3—Analysis of trace elements in the three fly ash samples

<table>
<thead>
<tr>
<th>Constituents ppm</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>40(18-76)*</td>
<td>42</td>
<td>49</td>
</tr>
<tr>
<td>Cu</td>
<td>21</td>
<td>20</td>
<td>26</td>
</tr>
<tr>
<td>Mn</td>
<td>75</td>
<td>77</td>
<td>36</td>
</tr>
<tr>
<td>Co</td>
<td>82(23)</td>
<td>88</td>
<td>49</td>
</tr>
<tr>
<td>Pb</td>
<td>75(63.7)*</td>
<td>70</td>
<td>86</td>
</tr>
<tr>
<td>Zn</td>
<td>48</td>
<td>50</td>
<td>53</td>
</tr>
<tr>
<td>Cd</td>
<td>5</td>
<td>4</td>
<td>9</td>
</tr>
<tr>
<td>Cr</td>
<td>67(45-160)*</td>
<td>135</td>
<td>62</td>
</tr>
<tr>
<td>Hg</td>
<td>0.1</td>
<td>0.06</td>
<td>0.08</td>
</tr>
</tbody>
</table>

*Values reported in ref. 9.

Chemical Analysis
Initially spectrographic measurements were carried out to detect the presence of various trace elements in the samples. The results indicate that while Cr, Ni, Co and V occur in appreciable amount (~80-100 ppm), Be, B, Cu, Ga, Se etc are present in trace quantities (~20 ppm). Besides these, there are many elements which are present in minute traces.

Tables 2 and 3 depict the major and minor constituents of the fly ash samples respectively. It can be seen from the data that the three major constituents namely, SiO2, Al2O3 and Fe2O3 are closely similar within the acceptable (IS-3812) range and are typical of the ash samples collected from different parts of India. This is because almost all the thermal power plants use high ash (35-40%) coals in their boiler. However, sample 3 contains higher Al2O3 content but little CaO content, suggesting that the ash sample is more of refractory and abrasive in nature. Such type of ash may create erosion problem not only in the plant components, but also during its transport through pipe line.

From the building material point of view these three major constituents are represented as “modulus of silica” or SiO2/(Al2O3+Fe2O3) ratio. The value of this ratio should lie between 1.3 and 2.5 (Ref. 7). For the three present samples the values are 1.60, 1.74 and 1.47 respectively suggesting that chemical composition-wise these samples can be used for making building materials. Another interesting aspect for the sample 3 is that while its total alkali content is rather low, its MgO content is higher. Possibly, excess Al and Mg contents occur in the form of spinel ferrite8 i.e. (Mg, Fe)(Fe, Al)2O4.

Leaching Characteristics
Leaching of trace elements from fly ash in aqueous medium is an important aspect from the point of view of environmental contamination. Recently, this aspect of flyash disposal problem has been excellently reviewed by Prasad et al.10. There are various methods available in literature to determine the leachability of various trace elements from fly ash and their comparative evaluation has also been reviewed10. In the present work, the batch leaching method has been used because of the simplicity of its operation and minimum interference of extraneous factors.

As far as trace elements are concerned the three samples have almost similar composition, except high chromium content in sample 2. Some of the trace metal contents are within the range of values given by Sahu9 for Talcher fly ash. However, Hg and Cd contents are low in all the samples.
ions in solution with the resultant rise\(^2\) in pH.

\[
\begin{align*}
M_2O + H_2O & \rightarrow 2 M^+ + 2 OH^- \\
MeO + H_2O & \rightarrow Me^+++2 OH^- 
\end{align*}
\]

where M=Na or K and Me=Ca or Mg.

In the case of pond ash (sample 2) these metal ions probably occur within the matrix as most of the surface concentration possibly had got depleted, thereby showing slower rate of release compared to that of sample 1. But other factors such as long-term hydrolysis of dissolved iron and aluminium may release H\(^+\) thereby restricting the rise in pH level\(^2\).

The acidic pH of sample 3, is, however, due to the release of the oxides of sulphur (SO\(_2\) or SO\(_3\)) adsorbed on highly reactive and extremely fine ash particles, producing H\(_2\)SO\(_3\) or H\(_2\)SO\(_4\) in solution\(^1\). The low alkali and alkaline earth metal contents, but high dissolved SO\(_4^{2-}\) content provide adequate evidences for the acidic character of the sample. The released H\(^+\) slowly attacks iron oxides and even some alumino-silicate minerals, resulting in the slow increase in pH value was demonstrated in Fig. 6.

On the basis of pH variation with time, the leaching characteristics of trace metals may be discussed. The leaching behaviour of six trace metals from samples 1 and 2 (Figs 7 and 8) are essentially similar. The initial rates of leaching of Ni, Co and Pb are more rapid than those of Hg and Cr. While Ni and Co concentrations attain their constant values after 7-8 days of leaching, the concentrations of Hg and Pb continue to increase slowly up to 30 days and possibly beyond this period. From environmental point of view, the steady release of these two toxic metal ions is a matter of concern, as this may lead to possible contamination of both ground and surface water. Figs 7 and 8 also indicate that leaching of Cu from samples 1 and 2 is very slow. It is interesting to note that no Cd was found in measurable quantity in the leachates of these two samples.

The leaching characteristics of various trace metals (except Ni and Cu) from sample 3 are different from those of samples 1 and 2 as demonstrated in Fig. 9. After an initial rapid dissolution up to 4 days, though the concentration of Ni tends to attain an almost constant value, the concentration of Co continues to increase steadily up to 30 days. Another notable difference is that Cd begins to leach out from this samples right from the beginning. Acidic medium accelerates\(^14,15\) the leaching of Co and Cd. Fig. 9, however, shows that the rate of leaching of Pb, Hg and Cr is considerably slowed down in acidic medium.

**Mechanism of Leaching**—It is now well-established that most of the trace elements occur on the surface of fine particles of fly ash, some of which are in soluble forms.

The initial rapid dissolution of certain quantity of Ni and Co in the first few days followed by an almost plateau region up to 30 days suggest that certain fractions of these two elements occur in soluble form, possibly as sulphates. Indeed, many metal oxides including nickel and cobalt oxides get sulphatized at temperature around 800°C by SO\(_2\) present in the flue gas\(^16\). A small portion of the
sulphates of Ni and Co might have escaped decomposition at higher temperature when got trapped within the glassy/siliceous materials. On the other hand, Hg, Pb etc. are probably leached out from the surface of the glassy spheres, possibly as hydroxy species below their solubility products. The formation of the hydroxy species of these metals including Cu is favoured with increase in pH. At pH below 5.5 Cu does not occur as soluble hydroxy species, but above 6.0 not only Cu but also Pb and Hg occur as CuOH\(^+\), PbOH\(^+\) and HgOH\(^+\) respectively. However, above pH 7.0, CuOH\(^+\) tend to get precipitated as Cu(OH)\(_2\), while the other two positively charged hydroxy species are stable even up to pH 8.5. Therefore, the release of Pb and Hg increases with the period of leaching but Cu tends to attain a plateau region.

**Interfacial Properties in Aqueous Suspension**

Many of the uses of fly ash involve processing of the material in presence of water. In aqueous suspension the major constituents such as silica, aluminosilicates, iron oxides etc. in fly ash undergo surface hydration, hydroxylation and finally acquire surface charge by releasing either H\(^+\) or OH\(^-\) ions. Silica and aluminosilicate minerals have points of zero charge (PZC) at pH 2.2-3.5 (ref. 19), whereas oxides of iron have PZC value at pH around 7.0 (ref. 4, 19). The PZC value of Al\(_2\)O\(_3\), on the other hand, lies at pH 9.5-10.0 (ref. 19). The change in pH value (ΔpH=pH\(_i\)-pH\(_f\)) observed by the addition of fly ash into aqueous solution of an indifferent 1-1 electrolyte clearly demonstrates the changes that take place on the surfaces of the three different major constituents of fly ash (Fig. 10). It can be seen that there is no change in ΔpH in the pH range 3-4. Therefore, in this pH range the interfacial behaviour of the material is predominantly controlled by silica or aluminosilicate minerals.

As the pH value is increased from 4.0 to 5.5 the final pH value of the suspension becomes higher than that of the initial value as the silicate mineral surface becomes more and more negatively charged. In such a situation H\(^+\) will be concentrated close to the surface forming the counter-ion layer and OH\(^-\) will tend to form diffuse double layer resulting in the increase in pH\(_i\). The suspensions are expected to remain in dispersed state at the minimum value of ΔpH i.e., maximum value of final pH when the fly ash particles are likely to experience mutual repulsion due to high negative surface charge.

As pH\(_i\) value is increased above 5.5, pH\(_f\) value decreases gradually up to a pH\(_i\) value of 6-7 when ΔpH becomes zero. Here the oxides of iron probably play an important role in controlling the interfacial behaviour of fly ash. Since the amount of iron oxide phase is much lower than that of either silicate or alumina, a mutual interaction of the three major constituents lower the cross-over point in some cases below the usual pH\(_{pzc}\) of iron oxide. At pH\(_i\) > 7.0 the equilibrium or final pH
It has been observed by several investigators\textsuperscript{22-24} that both relative and specific viscosity of suspension of solid particles in water is related not only to the true volume fraction of solid but also to the volume fraction of "free liquid" which is defined as the difference between apparent (measured) and true volume fractions of solid ($\phi_t$) in a suspension. The true volume fraction of a solid is the volume fraction of solid in a closest packed sediment and is obtained from voidage ($\varepsilon$) value given in Table 1. The apparent volume fraction ($\phi_a$) may be estimated experimentally from the relationship\textsuperscript{25}

$$\phi = \frac{\rho_s - \rho_m}{\rho_c - \rho_m} \quad \ldots(3)$$

where $\rho_s$, $\rho_m$, $\rho_c$ are the densities of solid (true), medium and suspension respectively. Since the ratio ($S'$) between apparent and true volume fraction of solid gives the relative volume of solid in a suspension, the apparent volume fraction of solid is $S' \phi$ the volume fraction of free liquid is $1- S' \phi$. The physical significance of the value of $S'$ is, that it represents the degree of hydration or the volume of bound water per unit volume of solid.

As stated above, the specific viscosity ($\eta_{SP}$) is directly proportional to the volume fraction of solid and inversely to the volume fraction of 'free liquid'. Combining these two variables Robinson\textsuperscript{24} obtained Eq.(4)

$$\eta_r - 1 = \eta_{SP} = \frac{K \phi}{1 - S' \phi} \quad \ldots(4)$$
where, \( K \) is the overall constant

Rearranging,

\[
\frac{\phi}{\eta_s} = \frac{1}{\eta_p} \left( \frac{1}{K} \right)^{m}
\]

Some authors\(^{22,23} \) have, however, preferred to use logarithmic plot of relative viscosity versus volume fraction of solid or free liquid to explain the exponential rise in viscosity with concentration of solid. A typical form of equation is given\(^{23} \)

\[
\eta_r = \left( 1 - \frac{\phi}{\phi_c} \right)^{-m}
\]

when \( m \) is related to the shape factor. For spherical particles its value is 2.5

The results of viscosity measurement of the aqueous suspension of the three fly ash samples may be explained on the basis of above correlations.

Fig. 11 depicts the plot of relative viscosity versus volume fraction of fly ash slurry at 30±0.5°C. It can be seen that Newtonian behaviour follows up to 40% slurry, above which viscosity increases almost exponentially for all the three samples. Fig. 12 shows the plot of \( \phi/\eta_p \) versus \( \phi \) for aqueous suspension of the samples up to 65% solid (w/v). It can be seen that the linear behaviour follows better at high solid concentration. The values of \( S' \) and \( K \) were determined from the slope and intercept values respectively. It can be seen from the figure that while the bound water for samples 1 and 2 is about 3 times the true volume of solid, for sample 3 the value is 1.5 suggesting that the suspension of sample 3 is less hydrated, possibly due to low alkali or alkaline earth metal contents. The physical significance of \( K \) is generally attributed to the frictional coefficient which is influenced by factors such as particle roughness and the adsorbed layer of water around the particles\(^{24} \).

Fig. 13 shows the plot of log \( \eta_r \) against log (1-\( \phi/\phi_c \)). Unlike Fig. 12, a common straight line with a distinct intercept value, can describe the Eq. (6) for all the three fly ash samples. The intercept value is related to yield stress of the suspensions. The slope value of –2.6 is closely similar to –2.5 as suggested by Roscoe\(^{23} \) for suspensions of spherical particles.

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