Separation of Cenospheres from Fly Ashes by Floatation Method
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Abstract
Fly-ashes are non-combustible mineral residues which are produced from coal in thermal power plants. Four different types of fly ashes were collected from different power station in Gujarat. Characterization through SEM shows that fly ash contains cenosphere i.e. gas bubble containing ceramic particle independent of their bulk density. Floatation technique was used for the separation of cenosphere from fly ash. Two solvents with extremely different densities were used for the separation of cenospheres. All methods gave approximately yield of less than 1 % cenosphere in fly ash. Color of cenospheres varied from gray to almost white and the value of density range from 0.4 – 0.8 g/cc. Further, chemical composition analysis revealed that cenospheres do not contain any high concentration of hazardous elements.

Introduction
Cenospheres are hollow ceramic microspheres present in fly ash, a by-product of coal burning power plants. Cenospheres are the lightest particles in the fly ash [1, 6]. The wet microspheres are dried, processed to size specifications and packaged to meet customer requirements [7].

Not all cenospheres are alike. In fact, these can be quite different. The properties of cenospheres depend on the consistency of the coal used and the operating parameters of the power plant. As long as these two factors remain constant, the cenospheres will be quite consistent. Since the collection procedure for cenospheres are as simple as flotation in water, the collected samples may not necessarily be complete cenospheres, but broken plate-like particles. [8]

Cenospheres are light weight, inert, hollow spheres of Silica and Alumina filled with gas which naturally occurs in the combustion of coal. The properties are same as manufactured sphere products. Thus it is also called Glass beads, hollow ceramic sphere or microspheres.

These cenospheres can be used as reinforcements for polymer matrix composites, can be added in paints, etc.

Due to their unique combination of low specific gravity, spherical shape, controlled size, high compression, good thermal and acoustical insulation property, these are and can be used in many high value applications.

Single liquid floatation like water may not give separation of all cenospheres. Therefore, in the present studies two liquids with wide difference in densities were used for separation of cenospheres.

Experimental

Raw Materials Used
Four samples of fly ashes from different power plants were used in the separation tests. Fly ashes were collected from the different sources in GUJARAT, INDIA (Fig 1).

Cenospheres were separated from different fly ashes using float and sink method. Acetone (density-0.789 g/cc.) and water (density-1 g/cc.) were used as medium for separation in float and sink method. The collected cenospheres were characterized for different properties.

Characterization Techniques

Particle Size Distribution was determined using Sieve Shaker. Sieves of mesh sizes 75 µm, 106 µm, 150 µm, 212 µm, 300 µm were used and the cenospheres had sizes; less than 75, 75 – 106, 106 – 150, 150 – 212, 212 –300 in microns. Moisture Content for cenosphere was calculated by drying these in an oven at 110 °C for a dwell time of 10 hour.
Carbon content for various cenospheres was studied. Approximately 1 gm of Cenospheres was weighed in platinum crucible and placed in furnace and temperature was increased to 1000 °C & kept at hold for 2 hour. After ignition, the sample was cooled in desiccators and weighed.

Packing density for various cenospheres was measured by taking these in measuring cylinder having known volume. After filling the cylinders to a marked volume level, these were tapped to occupy maximum space and minimum volume. Ultimate density was measured through mass per volume.

Surface morphology was studied using Scanning Electron Microscope (HITACHI S-3000N). Different elements present in cenosphere ware estimated using EDAX. Crystallinity of the cenosphere was tested by XRD (Phillips X-pert X-ray).

**Results and discussion**

**Separation of cenosphere from fly ash**

Degrees of separation in the recovery of cenospheres from coal fly ash have been estimated using float and sink method. Different liquids viz. water (1 g/cc) and acetone (0.789 g/cc) were used for separation of cenosphere from fly ash. Fly ash is taken in a vessel to which is added water. Complete mass is stirred for four hours and is then allowed to settle for ten hours. All cenospheres having density less than 1 g/cc float up and are separated. Same process is used for separation using acetone (density-0.789 g/cc). Water separation should had resulted more cenospheres than acetone. But the experimental findings were different. Cenospheres separated using acetone were more than using water. Because fly ash has cementitious property. Cementitious property is more in water than in acetone. So more wt. % of cenosphere are separate in acetone compared to water. About 80-85 % of the cenospheres could be recovered. Careful washing of the recipient was done to avoid solid particles. After density separation, these fly ash fractions were weighed. These are presented fig. 2. Fig. 2 reveals that fly ash from GEB Gandhinagar has higher % of cenosphere.

**Particle Size Distribution**

Figure 3 shows the relationship between weight % of cenosphere and the particle size. Maximum of cenosphere particles had size less than 75 micron.
 Moisture Content

Moisture content of the cenosphere particles was found to be 0.0233 %. As seen from table 1, the moisture content is independent of the cenosphere size.

| NO. | Size (µm) | % Moisture |
|-----|-----------|------------|
| 1   | All       | 0.0346     |
| 2   | 212-106   | 0.0311     |
| 3   | 106-75    | 0.0139     |
| 4   | >50       | 0.0134     |
|     | Moisture Content(Avg.) | 0.0233 %   |

Carbon content

The properties of cenospheres depend on the elements present in the raw coal and the operating parameters of the power plant. In all different GEB power plant use coal is different. So the values of carbon content are different. In GEB Sikka, carbon content value is high compared to another and In GEB Gandhinagar, carbon content value is Low as shown in fig. 4.

Packing Density

Density By Cylinder (With Tapping)

Bulk density of the cenospheres was determined by placing the cenospheres in cylinder and tapped it. The measuring cylinder was filled with powder and tapped to provide proper compaction arrangement of the cenospheres particles in cylinder without application of any external pressure. Table 2 shows the results of densities of various cenospheres. Density of the Cenospheres was calculated from the

\[ \rho = \frac{M}{V} \]

Where M = mass of powder,

V = vol. of tapped powder in cylinder.

| Different Thermal Power Station | D gm/ml |
|-------------------------------|---------|
| GEB Gandhinagar               | 0.48    |
| GEB Sikka                     | 0.47    |
| GEB Vanakbori                 | 0.46    |
| Torrent Power Sabarmati       | 0.48    |
| D-AVERAGE0.47 gm/ml           |         |

EDAX

EDAX analysis of cenosphere shows (table 3 & Fig. 5) the presence of Al and Si elements in the cenosphere particles. Both side of the surface of each particle were examined. Also, different peak heights (wt %) was observed indicating the non uniformity of weight percent of the elements.
Table 3
Elemental Composition

| ELEMENT | CENO (FACE I) WT% | CENO (FACEII) WT% |
|---------|------------------|------------------|
| C       | 18.48            | 22.65            |
| O       | 46.99            | 43.02            |
| Mg      | 00.38            | 00.49            |
| Al      | 13.90            | 11.13            |
| Si      | 17.11            | 17.42            |
| K       | 00.99            | 01.72            |
| Ti      | 00.88            | 00.61            |
| Fe      | 01.26            | 02.97            |
| Total   | 100              | 100              |

SEM

SEM analysis unfolds the morphological characteristics of cenospheres. The cenospheres are mostly open-pore type. The size of the cenospheres was also measured from the SEM micrograph.

Average size of the cenosphere particles was found to be $82 \pm 4 \mu m$. At higher magnification, it could be noticed that the majority of the cenosphere particles have smooth surface with a few porosities.

From fig. 6 it is observed that as the particle size of cenosphere increases, the surface roughness of cenosphere increases. Also there is an increase in voids on the surface of the cenosphere. The probability of cracked particles are seen more in larger particle sizes as compare to smaller particles. Also, the voids were seen on the surface of the cracked region of the cenosphere particles (Fig. 6 D). The small sized cenospheres are seen in the inner region of the larger cracked cenosphere particles (fig. 6 B).

It also depicts that within the same cenosphere the shell thickness varies at different locations. The shell thickness of the cenospheres was digitized from 100 such shells and the average shell thickness was noted to be $6.5 \pm 0.2 \mu m$. 
**XRD analysis**

Figure 7 shows XRD pattern of Cenospheres. The Cenospheres consist of quartz and mullite as crystalline phases as indicated by X-ray patterns and some quantity of glassy phase. Also, it is found that as the cenosphere particle size increases the carbon content also increases.
Conclusion

Cenospheres mainly contain alumino-silicate, mullite & quartz phases and are not subjected to any phase transformation up to 900 °C.

The cenospheres are almost spherical in nature and some of the cenospheres are associated with tiny cenosphere on their surfaces. The shells of the cenospheres are porous in nature and of varying thickness.

As size of the cenosphere increases, the surface roughness of cenosphere increases. The probability of occurrence of cracks particles are more in larger mesh size cenospheres as compared to smaller mesh size particle.

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