Summary

Fly ash as a waste byproduct is produced during combustion of bituminous / subbituminous pulverized coal by thermal power plant and at present, it is a serious ecological problem associated with their storage and disposal. Therefore the development of suitable scientific, technical and economic solutions for use of fly ash is of prime importance.

The spherical shaped materials blown out from the furnace during combustion of coal in thermal power plants make up 75% of ash generated. The cenospheres comprises 0.5-1.0 weight percentage of the fly ash individual particles and are chemically fairly homogeneous, but the pronounced compositional variation exists among particles with similar physical and structural attributes. Cenospheres are composed of nearly stoichiometric mullite (3Al₂O₃. 2SiO₂) needles bonded by alluminosilicate of similar composition that offers excellent thermal, elastic and mechanical characteristics for use in thermo-structural applications. Unburnt organic matter during combustion of coal leaves carbon in three different forms such as inertinite particles, isotropic coke and anisotropic coke in the fly ash. Fly ash also contains toxic elements like Co, Pb, Ni, Cd, Cu, Fe, Zn, etc. in traces.

Various developments have been made to explore the possibility of utilizing fly ash as raw materials for value added products. Fly ash is used as raw materials for different applications such as heat insulating bricks, glazed floor and wall tiles, mineral wool, building distemper, light weight refractory, ceramic fibers, high wear resistance tiles/pipe lining, fire abatement applications, sintered lightweight aggregates, high performance concrete, zeolites, cement, glass ceramics, building bricks, etc.
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Fly ash can be extremely attractive material, applicable for the synthesis of the composites also. The very unique and inexpensive composite powder resource, unlike those synthetically produced in a favourable spherical form with wide range of size and density, may be suitable in various structural applications.

Particles filled polymer matrix composites (PMCs) are more attractive because of their wide applications and low cost. Polymers such as nylon 6, styrene butadiene rubber, natural rubber, epoxy, polystyrene, polyester, polypropylene, poly methyl methacrylate, high density polyethylene and polyaniline are used as matrix. By incorporating inorganic mineral fillers into plastic resin, improves various physical properties of the materials such as mechanical strength, modulus and heat distortion temperature as compared to neat resin components. In general the mechanical properties of particulate filled composites depend strongly on size, shape and distribution of filler particles in the matrix and good adhesion at the interface surface.

The demand for the light weight materials for engineering applications had led to the development of fly ash based thermosetting resins. In cenosphere-epoxy based composites the addition of cenosphere can lead to reduction of the density and may increase strength of the composites. At present, epoxy resins are widely used in various engineering and structural applications. In order to improve their processing, product performance and cost effectiveness - various flyash and cenospheres are incorporated into the resins during processing. Furthermore addition of glass fiber as reinforcement can play a major role in maintaining strength, stiffness, thermal stability and frictional properties of the composite materials.

This thesis, reports the processing of composites with different formulations and characterization carried out on epoxy-flyash composites. The work incorporated in the thesis has been divided into six chapters.
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Chapter-I include the introduction to fly ash, glimpse of utilization of fly ash in various fields, separation of cenospheres from fly ash and various types of fly ash and cenosphere based composites with detailed literature survey carried out.

Chapter-II contains experimental methods and instrumentation used for characterizations of fly ash, cenospheres and other raw materials. The processing methods used for fabrication of the composites and characterization techniques used for these composites, is given in this chapter. This chapter also contains information about various instruments used in the present studies.

Fly ash was characterized for chemical composition like silica, alumina, iron oxide, carbon content, etc. Physical properties like bulk density, pH, Electrolyte conductivity and particle size distribution were also carried out. Particle size distribution was carried out by sieve analyzer and laser diffraction based particle size analyzer. Density and porosity of the composites were calculated according to ASTM C-838-91 and ASTM C-20-92 methods respectively. The Epoxy- Fly ash composites were characterized for their physical, mechanical, thermal and morphological properties.

The micro-structural studies of worn surfaces and unworn surfaces of composites were observed by Optical microscope (LABOR LUX 12 POL S) and Scanning Electron Microscope (Hitachi S-3000 N). Thermal properties were measured by TGA, DSC on Mettler TG50 and DSC20 respectively. Mechanical properties like compressive and flexural strength, Hardness, impact and Vickers Hardness were measured on UTM (INSTRON), Rockwell Hardness Testing Machine TRSDM and Omni Tech Vickers micro hardness respectively. Tribological performance of each composite were evaluated at linear speed (20 cm$^{-1}$.s$^{-1}$), load conditions (1N – 5N) and sliding distance (50 – 300 m). All the tests were performed
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at room temperature (without external heating of samples) in atmosphere. The friction force (and thus friction coefficient) was determined from the output of a strain gauge mounted on the arm carrying the pin. The temperature rise due to friction which was measured using an Al–Cr thermocouple placed at a distance of 50 mm from one of the samples and 3 mm above the counter-face.

Chapter III includes the study on the effect of four different fly ash samples from different power plants namely GEB Gandhinagar, Gujarat (GEBG), GEB Sikka, Gujarat (GEBS), GEB Vanakbori, Gujarat (GEBV) and Torrent Power Sabarmati, Gujarat (TPS). All collected fly ash were characterized for their specific gravity, surface area and LOI. Separation of cenosphere from fly ash was also carried out. Packing density was found to be maximum in fly ash collected from Torrent Power Sabarmati i.e. 2.1 g/cc and minimum value 1.7 g/cc was found in fly ash collected from GEB Vanakbori power station. This data also indicated that fly ash density varies strongly with various power stations and found to be in the range of 1.7–2.1 g/cc. One would expect that such variations are due to variation in origin of coal, coal mineralogy, and the combustion conditions in the furnace. The main constituent of the fly ash consist of silica (SiO₂), Alumina (Al₂O₃), Fe₂O₃, CaO, and MgO. The other components present were alkali (Na₂O, K₂O) & TiO₂, which remain fly ash and cenosphere in very less amount (<3%). Fly ash (GEB Gandhinagar) has more LOI value as compared to other. The Blain’s surface area remains in between 0.92 – 2.185 m²/g for Fly ash and between 1.82 – 2.1 m²/g for cenosphere. It was observed that the specific surface area of cenosphere were higher as compared to that of Fly ash. The degree of separation in the recovery of cenospheres from coal fly ash was estimated using float sink method. Different solvents were used for separation of cenosphere from fly ash. The solvents used were water (1 g/cc), methanol (0.791 g/cc), acetone
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(0.789 g/cc), ammonia (0.91 g/cc) and mixture of water and methanol (10:1) (0.973 g/cc).

Particle Size Distribution of fly ash was determined by mechanical sieve Shaker. Sieves of different ASTM mesh sizes i.e. 75µm, 106µm, 150µm, 212µm and 300µm were used. Density of cenosphere was determined by float & sink method. The moisture content of fly ash was found to be 0.023%. SEM analysis reveals that the cenospheres are almost spherical in nature & some of the cenospheres were associated with tiny cenosphere on their surfaces. The spot analysis of surface of cenospheres was done using EDAX. It shows various elements such as Si, Al, Fe, K, Ti, O and C present in varying percentage in Cenospheres. X-ray analysis confirms that Cenospheres consist of different phases of quartz (SiO₂), mullite (Al₄SiO₈)₁.₂, mullite-synthetic (Al₄.₇₅Si₁.₂₅O₉.₆₃), graphite(C), carbon, sillimanite (Al₂SiO₅), as crystalline phases and some quantity of glassy phase. The characteristic XRD peaks of mullite and mullite-synthetic were observed at 26.19° (20), 16.41° (20), 25.97° (20), 40.83° (20), for sillimanite (Aluminium Silicate) at 26.03° (20), 26.43° (20), 40.91° (20) and for Graphite (Carbon) at 26.61° (20), 43.46° (20). TGA analysis was done in the presence of the oxygen atmosphere. Weight loss occurs at different range of temperature mainly due to unburnt carbon and some minor constituents present in the cenospheres.

Chapter IV elucidates detailed study of epoxy-cenosphere based composites and their chatcteristics. Bulk Hardness & Micro hardness test were performed for epoxy rigid block & cenosphere reinforced epoxy composites. The bulk hardness data reveals that as the percentage of cenosphere increases, the hardness of composites decreases. Vicker’s Micro hardness in the region of epoxy matrix is more as compared to cenospheres. Compressive strength of the fabricated composites is low in
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high volume cenosphere composites. Also the density of composite decreases with addition of more weight percentage cenospheres. Porosity of composites increases with increase in particle size, aspect ratio, and volume fraction of reinforcement material.

Optical studies were done for EF composites. Fly ash is found to be evenly distributed in epoxy matrix. Some of the particles show a hollow type structure. These are light weight cenosphere particles. Tribological measurement and tribological study was carried out for as such epoxy, EF composite with different percentage of fly ash with variation in load condition. Load is varied from 1N, 2N and 5N. Coefficient of friction of neat cured epoxy block decreases with increase in load. It was found to increase with increase in percentage of fly ash. Fly ash generally consists of SiO$_2$ and Al$_2$O$_3$ particles which are hard ceramic particles. Owing to hard fly ash particles, there is change in friction pattern as well as the surfaces of rubbed samples as observed under optical microscope.

The studies on composites made of Epoxy resin (Matrix), cenosphere (Filler) & glass fiber (Reinforcement) are summarized in Chapter V. The number of glass fabric plies was kept fixed as 20 and the different weight percentage of cenospheres from 2.5 to 20 were added to epoxy resin to make the composites. Results showed that with increase in the percentage of cenospheres, porosity of the epoxy-fly ash composites increases. Fiber volume content was found to be 32%. The composites exhibit increasing trend in flexural strength with addition of 2.5% to 15% cenospheres. With higher amount of cenospheres, there is decrement in value of strength. Further study also reveals that as cenospheres size is smaller the strength is higher. The maximum compressive strength value of 234.92 MPa was found in composites with 7.5 % of cenospheres and further addition of cenospheres decreases the value of compressive
strength. Rockwell hardness value decreases from HRL 101.14 to 100.85 as the percentage of cenosphere increases from 0 to 17.5. The impact strength is found to increase with percentage of cenospheres.

Chapter VI summarizes all results obtained and the conclusions drawn. Present studies illustrate the processing of epoxy-flyash composites, epoxy-cenosphere composites and epoxy-glass fabric-fly ash composites. Fly ash, cenosphere and glass fabrics show good compatibility with the epoxy matrix. Composites prepared with glass fabric showed better physical properties as compared to epoxy fly ash and epoxy cenosphere composites. In an overall conclusion, density and porosity of the composites is found to depend on processing of composites, percentage of fly ash and cenosphere and on curing time to some extent. Densities of the composites prepared with mixed glass fabrics and fly ash as reinforcements are higher compared to other composites. Microstructure analysis show well distribution and bonding of fly ash particles, cenosphere and glass fabric in the epoxy matrix. Thermal studies were carried out for epoxy-fly ash composites. Rockwell hardness of the composites made with fly ash is found to be higher as compared to other composites. Mechanical properties of the epoxy-glass fabric-fly ash composites are somewhat better than other composites. Glass fabric as an additional reinforcement improves the compressive strength of the composites. Tribological properties of epoxy fly ash composites were also carried out. It was found that as the amount of fly ash increases coefficient of friction increases. Also, with increase in load during measurement from 1N to 5N, coefficient of friction decreases. In case of epoxy-fly ash composites the friction value was found to be 0.129 - 0.263 (5N).