IJARSCT

ISSN (Online) 2581-9429

International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)

International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal

Impact Factor: 7.53

Volume 4, Issue 4, March 2024

SEM, XRD and FTIR Analysis of Fly Ash Polymer Composite and Investigate the Micro Structural Changes, Physical, Mechanical, Thermal Conductivity and Wear Behavior at Different Proportions

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Abstract: *Industrial waste such as fly ash poses environmental problems, yet it is utilized as an inexpensive and readily available construction material. However, these bricks have low strength. Thus, extensive research aims to increase the strength of these bricks. The current study develops a new systematic procedure to produce fly ash composite bricks with higher compressive strength. Here, fly ash is blended with cold-setting resin at varying proportions and water treated at different temperatures to find a solution for the brick industry. Under optimal test conditions, the obtained fly ash-resin powder bricks have a compressive strength, hardness, water absorption, density and thermal conductivity of 11.24MPa, 47.37HV, 19.09%, 1.68 g/cm3, and 0.055 W/mK respectively. The sliding wear behavior is also examined. X-ray diffraction, FTIR analysis and scanning electron microscopy are used to study the structure-property correlation of these composites.*

Keywords: Industrial waste

I. INTRODUCTION

The production of FA in India is now estimated to be around 180 MT annually; this is expected to rise to approximately 320 MT annually by 2017 and 1000 MT annually by 2032 [3].Indian coal undoubtedly has a poor heat value and a high ash content.An rising number of coal-based thermal power plants have been built to fulfill the demanding needs. This led to the production of a massive amount of burned residue in the form of fly ash (80%) and bottom ash (20%). The burned coal's finely divided particles are released into the flue gases, which are mechanically separated from one another by separators and electrostatic precipitators before being gathered in hopper fields. The rate of FA production is high and continues to rise increasing year after year. It is estimated that China, India, and the US produce 275 million metric tons of FA annually. However, less than half of this is used in different contexts. The largest issue facing the manufacturing and processing sectors is how to properly dispose of leftover garbage. Because of the detrimental effects on the environment, fly ash must be dumped appropriately, and full use of FA when practical is justified. Waste materials that are often flammable, reactive, corrosive, or poisonous have a negative impact on the environment. The disposal of the remaining industrial waste products is a major challenge that needs to be addressed in a cost-effective, environmentally-friendly way. The challenge of safely disposing of ash without damaging the environment, destroying ecological equilibrium and the huge storage space required are major challenges and obstacles for the safe and sustainable growth of the country. Therefore, continuous efforts are required by making strict regulations by the Government to make full use of the ash. Currently, only 50% of fly ash is being used in a profitable way in India[4]. The most popular and feasible ways to use these industrial wastes products are to build roads, highways, embankments, etc. The problem of environmental pollution can be significantly reduced if these waste products are effectively used in the construction of roadways, highways, etc. However, not enough soil of the required quality is available. Therefore, not only are these industrial wastes being used as an alternative for natural soil in the construction process, but it also solves the issues of disposal and environmental pollution

About Fly Ash: Overview

FA is an Industrial waste which is accepted as an environmental pollutant, generated during the combustion of coal for energy production. When the coal is fired inside the grate of a boiler, Carbon and volatiles materials completely burnt off. But still, some inorganic impurities of earth elements (sand, Feldspars etc.) are bonded together and are discharged out through flue gases. When these fused materials are allowed to solidify, it results in the formation of fine and spherical particles called Fly ash. These FA particles are tiny spheres enclosed in a big sphere called plerospheres. Hollow spheres are also called cenospheres. The morphology of FA particles is sphere due to the bonding which takes place during suspension of released flue gases from chimneyor boiler. These fine particles mainly consist of oxides of silicon, aluminum and iron. Some elements like P, Mg, K, Ca, with small traces of Cu, Zn, Mn, Fe, B, and Mo are also found. The properties of FA vary from different sources, from the same source but with time and with the techniques used for handling, storage and variation in load generation

A. Fly Ash Bricks

Bricks has been used as a major construction and building material. Since long Aluminous –silicateand silica bricks are chosen as refractory materials in many industrial applications, due to their high wear resistance, long lasting, sturdy and load bearing capacity at high temperatures [8]. Dueto the limitation of clay resources, china has partially restricted the use of conventional fired bricks produced from clay [9]. Therefore the ultimate aim is to find raw materials for brick production alternative to clay. These days energy savings has become a very important environmental and economic issue. The consumption of energy from buildings comprises about one third of the totalconsumption, with nearly half of its energy lost through the walls [10].One of the effective approaches to reduce energy consumption is to decrease the thermal conductivity of wall material, such as brick. Organic residues such as saw dust, polystyrene, paper sludge, coal, coke and inorganic products are commonly used to decrease the thermal conductivity of the brick. These residues used as a pore forming additives to obtain highly porous bricks. Numerous studies have been conducted on fired brick made of Fly ash [11, 12]

The bricks made of FA are three times resilient and stronger than conventionalbricks with constant strength. Due to the presence of free lime the strength of compacts is accelerated at high rate. Hence these bricks are perfectly fit for internal and external loadbearing and non-load bearing walls. To determine the compressive strength and microstructure of the cracked samples, compacts of Fly ash and cold setting resin along with hardener with various percentages are prepared and treated in water at 1100 C - 180oC for 24 hours.

Salient features of FA bricks:-

1) Practically no damage can be seen during transport and use, due to their highstrength.
2) Owing to uniform size of bricks mortar required for joints and plaster reduces byalmost 50%.
3) The seepage of water through bricks substantially reduces due to its low waterpenetration.
4) Like conventional clay made bricks, FA bricks are not soaked in water for 24 hours before use. Only sprinkling of water is sufficient.
5) No need of Plastering.

B. Cold Setting Resin: - An Overview

Cold mounting compound resin is used as a binder material to provide inter particle bond betweenthe FA particles and to increase their strengthening effect. They are good resistance to atmosphericand chemical degradation. Resin powder cannot shows its effect alone until it is mixed with hardener (or accelerator) to provide the mounting compound, and then the polymerization processtakes place to form the desired block. This process sometimes generates heat but this generation can be minimized by the use of cool air or cooling water. These compounds can be ideally chosenfor those materials which show sensitivity towards heat or pressure. This cold setting resin offersbetter properties for Fly ash compacts. Improved mechanical strength and hardness, resistance to atmospheric and chemical degradation, reduced thermal conductivity, eliminates porosities and cavities, fast curing of compacts are some of the common properties. The setting compound and the hardener were supplied by Geosyn pvt. Ltd. Kolkata.

C. Objective of the Present work

The aim of the present work is to fabricate Fly ash polymer composite at different proportions of polymer and to study physico mechanical, thermal conductivity and wear behavior. In present project an attempt was made to increase the density and hardness of the water cured cylindrical samples. SEM, XRD and FTIR analysis were also done to investigate the microstructural changes.

II. LITERATURE REVIEW

A. Introduction

Porbaha et al. (2000) estimated that around 41% offly ash was utilized for the production of landfills in Japan. Utilization of fly ash has got a great contribution in the field of construction of ridges and fills. The increased use of fly ash in the fieldof construction and fills has gained a major acceptance in technology demonstration projects.

Vittal (2001) stated that few embankments have already been constructed using pond ash in India.According to IRC, 2001 (a working body of Indian govt.) has proposed strategies to use fly ash inroad embankments [14]. Flyash shows self - hardening behavior and can be utilized in construction over wide range. This property is due to the availability of free lime .The properties of it depends on various characteristics out of which some are characterization of coal, fineness of pulverization,furnace type and temperature of firing

B. Classification of Fly ash

Based on the amount of lime present, Fly ashes can be classified as Class F and Class C accordingto ASTM C618 [15], And on the basis of lime reactivity FA are categorized as Grade I and GradeII according to I.S. 3812 [16]. The type of coal burned and the amount of ash content relates the classes of Fly ash. Class F Fly ash contains low lime and ash being greater than 70 Wt. % of Sio_2+Al_2o_3+Fe_2o_3 .While On the other hand, the ash content between 50-70 Wt. percent Sio_2+Al_2o_3+Fe_2o_3 and high lime content are grouped in Class C. Class F fly ash can be produced fromanthracite of bituminous coal while sub bituminous or lignite coal produces class C type fly ash. Since anthracite coal is not used for power generation so class F type Fly ash can easily be derived from this coal. Characteristics of Class F type Fly ash shows low calcium ashes having lime percentage 6, so these are not self - hardened. But normally this class exhibit pozzolanic features.The ash content from this class covers more than 2% unburned carbon which can be ascertained by (LOI) test. The major crystalline phases present in the form of Quartz, mullite and hematite identified fly ashes, derived from bituminous coal.

C. Fly Ash Properties

Making out desirable application of FA demands the study of fly ash characterization in terms of structural morphology, interfaces between phases and its susceptibility to chemical change (reactivity) is of great significance. FA is characterized by its physical, chemical and mineralogicalproperties that are greatly reliant on the essence of the parent coal, the combustion conditions, various emission control devices and the storage and treatment methods [17].

- Physical Properties:.
- Chemical Properties

D. Uses of Fly ash

Fly ash finds application in various sectors. In broad, the use of FA can be band together in to three categories.

Huge amount of FA is utilized in various areas which are of little significance in terms of cost. Some of the common uses are inbrick industry, filling of mines, ridges, surfacing and recovery of fallow land etc. A lot of efforts have been made for manufacturing of bricks by different agencies using lime, different types of resins, gypsum and clay. These binders are mixed with FA at different ratio and a range of products are available in the marketplace. Bricks made of FA are more highlighted since it saves the valuable top layer of soil. Similarly, exhaustion of river sands for mine stowing in underground collieries would lead to use of ash in large scale for filling up of mine excavations. The study of Leaching action with FA is essentially required before use.

FA are commonly used as cement stabilizer, light weight filler materials for prestressed structures ,wall slates and roofing tiles, for insulating blocks ,in paints and enamels and asherbicide in agricultural science to destroy unwanted vegetation. These all comes under thecategory of medium cost values.

Recovery of various Magnetic oxides, Aluminum oxides (Al_2O_3), and different trace elements , synthesis of Zeolites for industrial applications and making of inorganic wools.Removal of bleaches and organic compounds from waste water, mercury from the flue gases, adsorbents for cleaning of flue gases (So_x and No_x emissions). These are grouped under high cost values.

E. FA Disposal –curse to environment

Large amount of solid wastes in the form of Fly ash have been generated from thermal power plants. These wastes are widely utilized in various construction materials and other sectors. Apartfrom fulfilling the needs, the disposal of FA is the burning problemand creates hindrance in developing a pollution free nation. Hence it's a matter of great concern. Some of the problem regarding FA disposal are mentioned below:-[27]

Fly ash particles are available in both the dry and wet state. These ashes are disposed in bulk which occupies thousands of hectares of land and destroys the fertility of top soil.

Handling of FA particles in dry condition is a tough job. Since these ashes are very fine and dispersive in nature. The fine tinybits of FA destroy the structural shells and affects cultivation.

It hampers the ecosystem through various modes of pollution i.e. soil, air and water.

Since FA are disposed in open atmosphere which ultimately results in various air bornediseases due to long intake of air.

The biological features of soil and overall yield of crops also get hampered when FA isdisposed in the nearby areas before anytreatment.

F. Reviews on FA

A lot of investigators have worked on the coal ash properties to evaluate its importance in various fields. Some of them are mentioned below:-

1. Sherwood and Ryley stated that Fly ash possesses self-hardening features due to the presence offree lime in the form of calciumoxide or calcium hydroxide.
2. McLaren and Digioiab presented that the specific gravity of Fly ash is relatively lower than thatof soils .The density of the ash fills gets reduced which is a major advantage in terms of its use asvarious filler materials. Now these fillers can be used in spongy walls and ridges particularly whenthe foundation is weak.
3. Sridharan et al., studies the micrographs of FA particles through SEM. These particles are mostlysolid spheres with glassy appearance, hollow spheres with smooth-edged porous grains, asymmetrical agglomerates and irregular absorbent scraps of unburnt carbon. Presence of iron particles which are dark grey in color can be identified as pointed grains.
4. According to Mohini Saxena and P.Asokan a lot of multidisciplinary tests on coal ash have beenconducted at various lab centers. Regional Research laboratory, Bhopal has worked a lot on FA and enhanced the various methodologies for pilot scale demonstration. They cultivated Crops, vegetables and cereals and reported that the yield increases greater than before by FA utilization with no toxicity. They also developed paints using FA and epoxy systems for safety and embellishment. These FA paints has improved resistance to rust, abrasion and wear.
5. Mitchell and Brown said that the soil, FA and lime displays unique behavior and are much more dependent on the physicochemical properties of the fly ash and soil like porosity, segregation ,lime content ,time and pressure applied during compaction.
6. Sevelius et al., studied the utilization of Fly ash and Bottom ash in bricks manufacture and in refractory products. He also studied that there is a remarkable increase in the consumption of FA as a basic raw material.

G. Reviews on Fly Ash Bricks

Fly ash based bricks offer exciting advantages over traditional clay bricks. Aggressive research isbeing carried out worldwide on fly ash based geo polymers to improve functional properties. Thischapter outlines some of the recent reports published in literature on fly ash based geo polymers, its utilization for making bricks and its mechanical properties. Fly ash bricks have created prodigious attention and awareness among materials experts and engineers in current years due to the considerations of developing an environmental friendly, high strength material and partially switching currently used clay bricks.

Obada Kayali studied the properties of Fly ash and clay made bricks and concluded that the mechanical properties of fly ash bricks have exceeded to those of standard load bearing clay bricks.Compressive strength was 24% better than good quality clay bricks and tensile strength was nearlythree times the value for standard clay bricks. The bond strength of the fly ash bricks is 44% higherthan the normal clay bricks. The density of fly ash bricks is 28% less than that of standard clay brick. The reduction ina weight of bricks results in a great deal of savings in terms of raw materialsand transportation costs. Fly ash brick can easily soak

up mercury from normal air which is in contact with it and thus makes it cleaner for berating. There is also a process named carbonation in which fly ash absorbs carbon dioxide from normal atmosphere due to which carbon sequestration occurs and the amount of carbon gets reduced in atmospheric air which helps in minimizing global warming.

Sunil Kumar has presented an extensive review in reported work on fly ash bricks. He investigated the flexural strength, water absorption test, density, porosity and stability of these solid bricks and hollow blocks. He witnessed that these bricks and blockshave enough strength for their usage in low rate housing growth. Tests were conducted to determine the compressive strength and hardening effects and to analyze the effects of curing with time. The compacts treatedin hot water shows better strength andhardening effects as compared to normal water cured compacts. Initially the strength of these blocks and bricks increases with higher rate and then at a comparatively lower rate. There is a direct relationship between FA and water absorption. As the content of FA increases water absorption also increases .And on the other hand water absorption decreases with increase in density of the FA compacts. These FA bricks and blocks with proper phosphogypsum extent have improved resistance to robustsulfate environment.

Ball MC & Carroll RA has studied the various bricks manufacturing methods and understands the reason behind the strengthening effects of these autoclaved FA bricks. The FA bricks becomeshard mainly due to the formation of calcium silicatehydrate and calcium aluminate silicate hydrate. The hydrothermal reaction takes place between silica ,alumina and water when the compacts areallowed to cure under the steam bath normally at 110^0C -180^0 C. The presence of Tobermorite phase also helpsin enhancing the hardenability of the Fly ash bricks.

Cultrone G, Sebastian E, and Elert K, studied the permeability of FA bricks and co-relates its effects on various chemical and mineralogical configuration of Fly ash particles. FA bricks also depend on the temperature of firing resulting in to more vitrified dense structure and phenomenalchange in shape and size

III. EXPERIMENTAL WORK AND METHODOLOGY

A. Introduction

Fly ash has been used in various architectural and industrial applications on large scale. Hence Consumption of this huge amount of fly ash greatly reduces the difficulties met by coal based TPPs for its dumping. Analysis on the performance of FA at various states is essentially requiredbefore its usage. So to understand the characteristics features of FA, experiments cannot be performed on fielddomain. There is no any alternate option except research laboratory test to assess its importance. The research conducted in laboratory provides a calculative approach to govern several parameters that come across during practice.

Brief description of the types of material used, sample preparation and its characterization through SEM, XRD, and FTIR, Mechanical and surface properties like Compressive strength, Hardness and wear resistance, Thermal conductivity measurementand others are outlined in this section.

B. Materials Used

Fly ash: The Fly ash used in this project was collected from electrostatic precipitators of the captive powerplant (CPP-II) in dry condition. The fine powders were oven dried at 110^oC-160^oC and kept in airtight bottle for later use

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International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
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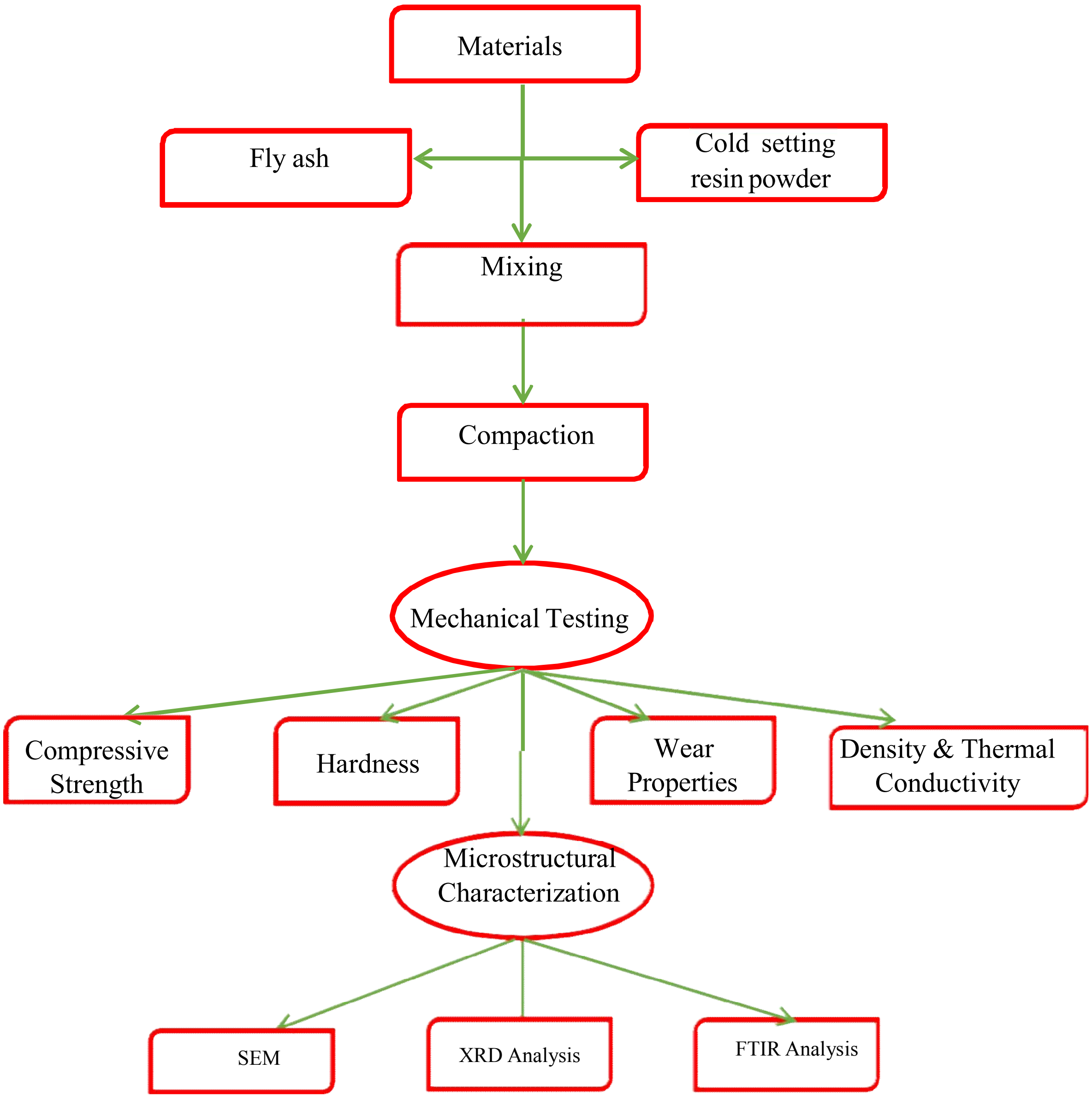
Impact Factor: 7.53
Volume 4, Issue 4, March 2024

Cold setting Resin and Binder: The resin powder and hardener used in the present study was supplied by Geosyn private Ltd. Kolkata.

Flow chart of experimental procedure



C. Experimental Methods

Preparation of Samples: The samples were prepared by Powder metallurgy route.

Mixing: Three different weight percentages of Fly ash and resin powder with (75%, 80% and 85%) and (25%, 20% and 15%) were taken respectively. These compositions were mixed thoroughly by a mechanical vibrator (Abrasion Tester Model PEI- 300), to get a homogenous mixture. Different compositions of Fly ash along with resin powder were kept in three different small size bottles. Around 6-10 small steels balls are kept inside for proper mixing. Mixing was done till the vibratorshows 1000 revolutions which almost took five hours

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Compaction: The compaction experiments were executed to make cylindrical FA compacts. Cylindrical die andpunch having 15 mm diameter made of stainless steel was used to make cylindrical Fly ash compacts .Mixture of approximately 5 gm. was taken for each composition. Then the punch & diewas cleaned with cotton followed by acetone so that all the dust is removed from theinside surfaceof the die and outside surface of the punch. Then greasing was done to avoid sticking. The mixtureprepared earlier was poured inside carefully. During the packing slight shaking was done to accommodate the maximum possible amount of material. Finally the whole system was subjectedto hydraulic seal valve made tight, mounting was done coaxially. Maximum of6tons of load wasapplied on it very slowly. Once the maximum load was achieved, the apparatus was powered off.The whole system was relaxed for 5 minutes which then followed by unloading. Compact was ejected from the Die in the same direction as the compression and was kept in normal atmospherefor 1 day. The cold setting liquid (hardener) was applied on the surface of the compacted sampleswith the help of a dropper, so as to harden the newly made compacts. The amount of Hardener used was 1/6th or 1/4th of the mixture. Hence in this way twelve samples for each composition weremade. All the samples were dried in open atmosphere for 2 days.

Water Treatment: Three samples from each composition were cured in water at 110^{o}C -180 ^{o}C for 48 hours.

D. Determination of Mechanical Properties

Hardness: Vickers hardness tester (LECO, LM 248AT) as shown in Figure 3.1 ,was used to find the hardness values of all the dry and wet samples using 20 gf Load for a dwell time of 15 seconds. At least eight measurements were taken at different position for each sample in order to get constant results.

Fig. 3.1- Leco, LM 248AT Micro indentation Hardness Tester

Compressive Strength: In order to measure the compressive strength of dry and wet samples INSTRON 1196. Prior to test, gauge length and gauge diameter of the dry and wet specimens were measured individually by the aid of Vernier caliper .The tests were carried out at room temperature (300 K) with a constant crosshead speed of 1mm/min and the full scale range load of 50 kN. This computer integrated machine gives the Load vs displacement signals directly when the specimens were subjected to tests

Wear resistance and Friction: In this study computerized Ball on Plate Wear Tester (TR-208-M1) as shown in Figure - 3.2 was usedto evaluate the wear performance and sliding contact resistance of the Fly ash compacts. The experiment was carried out with the help of 4 mm diamond indenter keeping the different track radiusof 4 and 8 mm respectively. Prior to wear, constant normal load of 10 and 20N was applied. The indenter rotates on fly ash compact with a constant speed of 20 rpm for different time period of 600s. At the end of each test, loss in weight of the samples was noted. Results obtained have been expressedin terms of wear depth, and friction co-efficient.

Fig.3.2- Ball-On-Plate Wear Tester (TR-208 M1)

Thermal Conductivity: To measure the thermal conductivity of Fly ash and resin powder mixture, KD2 Pro analyzer as shown in figure 3.3 was used and it follows ASTM Standard D5334-08 [28]. It comprises of a handheld controller and a various sensors that operator can embed into very nearly any material. Single probe of 6cm long and 0.127 mm diameter was inserted ina small plastic bottle filled with FA & resin powder to find the conductivity value .At least ten values of each composition was recorded to get the appropriate result. KD2 Pro uses the transient line heat source mechanism to evaluate the conductivity and diffusivity of the given mixture. A restrictive calculation fits time and temperature information with exponential functions via nonlinear least squares technique.

Water Absorption

Fig. 3.3 KD2 Pro analyzer

The cylindrical compacts were tested for water absorption according to ASTM C642. The weightsof all the samples were taken .Thecompacts were first dried in an oven at 100^0C -120 ^{0}C ensuring removal of moisture and hence allowed it to cool at room temperature. The weights were taken after drying and the variation in weight was less than 5%, considered it as dry. Now the compactsof different composition was immersed in a beaker filled with water and was kept in an oven at 110^0C-180^0C for 48 hours. Compacts were surface dried after removal and final weight was measured. The amount of water absorbed (%) was calculated using equation-1.

$$\frac{M2 - M1}{M1} * 100 \quad (1)$$

where

M1 and M2 are the mass of dry and wet sample respectively.

Density: On the basis of Water absorption test, the density of dry and wet compacts was calculated.

Microstructural Characterization

SEM Study: In present study, A JEOL 6480 LV Scanning Electron Microscope (Fig. 3.4) was used for the characterization of microstructural changes (pits, cavities, and porosity), determination of particlesize and morphology of FA compacts. To get the better image resolution, secondary electron imaging with accelerating voltage of 15 KV was used.

Fig. 3.4 Scanning Electron Microscopy (JEOL JSM-6480LV)

XRD Study: The mineralogical composition of Fly ash and the different phases present was determined by XRD analysis in a Philips X-pert multipurpose x-ray diffractometer (shown in figure.3.5) using Cu Kα(λ=1.5418A^o) radiation. The patterns were examined by comparing the positions of peak and intensities of the samples with those in the (JCPDS) data files. The diffraction patterns were recorded in the scanning range of 20^0-80^o with a step size of 2^0 C per minute

Fig.3.5 Philips X-pert multipurpose x-ray diffractometer

FTIR Study: FTIR spectroscopic technique is used to understand the chemistry of surface for fly ash in thermally active state along with different state of mineral phases, H_2O and –OH group on silica and alumina. Fourier transforms infrared radiation (FITR) spectrometer (shown in figure.3.8) is used to calculate the transmission percentage of infrared. In order to prepare pellet little quantity of potassium bromide (KBr) was segregated with powder sample and after that pressing of mixture was done. Analysis of that pellet was done using FITR by keeping the pellet in sample holder.

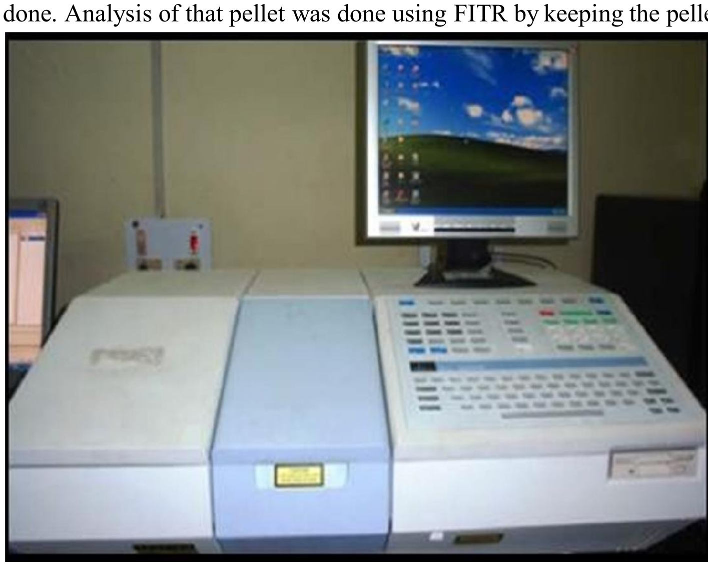

Fig.3.6 Perkin-Elmer Spectrum RXI, (FTIR) Spectrometer

IV. RESULTS AND DISCUSSION

Composition of Fly ash

FA mainly consists Silica (Sio2), Alumina (Al2o3), Calcium Oxide (CaO), and Iron Oxide (Fe2O3).The chemical composition ofFly ash is tabulated in table 4.1.

Compounds	SiO_2	Al_2O_3	CaO	Mgo	P_2O_5	Fe2o3	SO_3	K_2O	LOI
Composition (%)	54.5	26.5	2.1	0.57	0.6	-	-	-	14.18

Table 4.1 Compositional analysis of Fly ash

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International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
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Volume 4, Issue 4, March 2024

Water Absorption Test

Table 4.2 shows the amount of water absorbed corresponding to different FA composition. The water absorption values of FA composites lies in the range of 15.55 % to 19.09%. It can be seen that all the composition met the absorption criteria set by several developing countries. India permits the maximum of 20 % water absorption when compacts are immersed for 24 hours.

Mix Composition(Wt. %)	Weight (gm)		Water Absorption(%)	Average Water Absorption Value(%)
	Dry	Wet		
(FA)75%+ (RP)25%	4.579	5.302	15.78	15.55
	4.630	5.340	15.33	
(FA)80%+ (RP)20%	4.452	5.151	15.70	16.61
	4.642	5.456	17.53	
(FA)85%+ (RP)15%	4.502	5.356	18.96	19.09
	4.329	5.162	19.23	

Table 4.2 Percentage (%) water absorbed by various FA polymer compacts

Density Measurement

Density of the samples was calculated before and after treatment. From Fig. 4.2 we can say that density of dry compacts decreases with increase in weight percentage of FA. As the dry compacts are immersed in water at 110^0C -180^0C, then through capillary action voids are filled and it becomes hard and the porosity is eliminated. As a result of which the compacts become dense and finally the density increases with increase in FA content

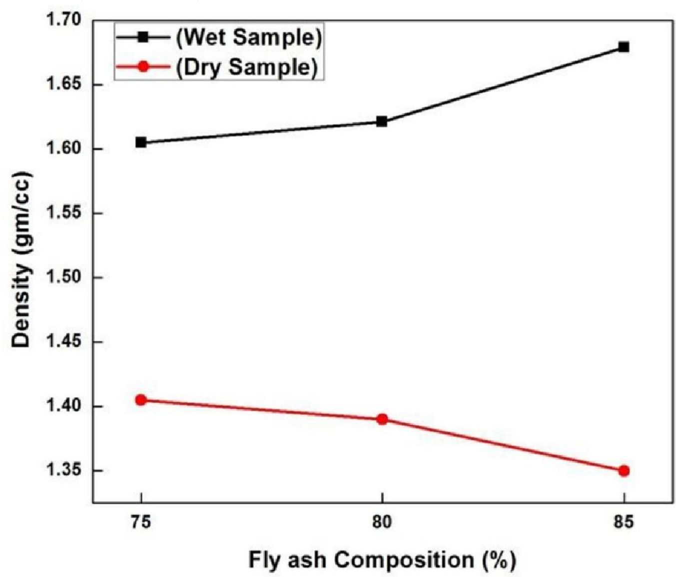

Fig.4.2 Variation in dry and wet density w.r.t FA composition

Mix Composition (Wt. %)	Density (g/cm^3)	
	Dry	Wet
(FA)75%+ (RP)25%	1.40	1.60
(FA)80%+ (RP)20%	1.38	1.62
(FA)85%+ (RP)15%	1.35	1.67

Table 4.3 Density value of dry and wet FA polymer compacts

DOI: 10.48175/IJARSCT-15965

IJARSCT

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Hardness Measurement

Hardness values of all the Fly ash polymer composite of different compositions, both in dry and wet state, were measured by the help of LECO, LM 248AT Vickers hardness tester. The Hardness values as obtained are shown in Table 4.4. The values of hardnessare in the range of 32.93 HV – 44.08 HV for dry composites and 39.78 HV – 47.37 HV for wet FA composites respectively.

S.NO	Mix Composition (Wt. %)	Micro hardness value (HV)	
		Dry	Wet
1	(FA)75%+ (RP)25%	32.93	39.78
2	(FA)80%+ (RP)20%	38.26	43.04
3	(FA)85%+ (RP)15%	44.08	47.37

Table 4.4 Hardness values of various FA resin mix compacts

XRD Analysis

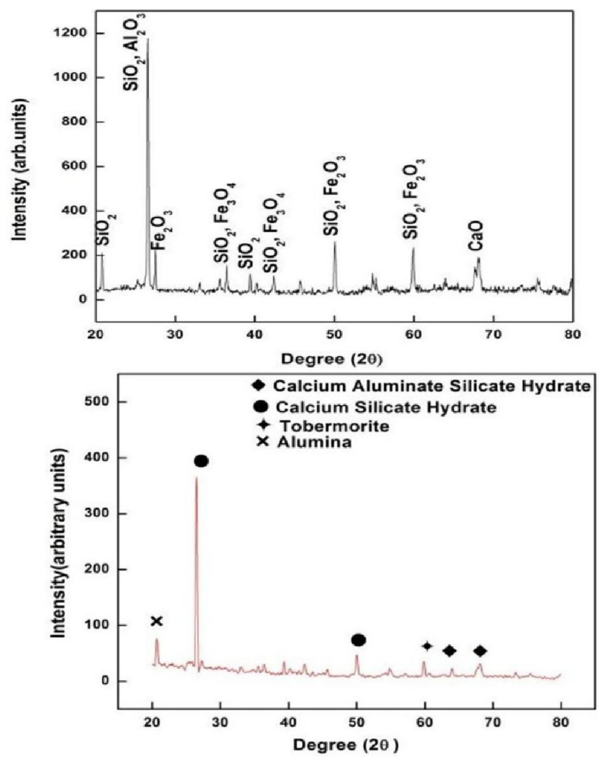

Fig.4.4 (a) XRD analysis of Fly ash and Fig.4.4 (b) XRD analysis of water cured compact

Fig.4.4 (a) shows that Fly ash particles primarily consists of Silica and Alumina. Fig 4.4 (b) Showsthe XRD analysis of water treated compacts. It has been found that in the presence of moisture, pozzolanic reaction occurs that leads to the formation of new phase i.e. calcium silicate hydrate (CSH) and calcium aluminate silicate hydrate (CASH) .These phases are responsible for solidification of unfired compacts and hence creating strong structures, excellent inter particle bonding with improved mechanical properties like hardness etc. CSH and CASH are considered to be an initial reaction product which changes in to a semi crystalline solid phase called Tobermorite ($C_5S_6H_5$).

FTIR Analysis

Fig: 4.5 shows the Fourier transforms infrared radiation (FTIR) spectrometer plot of 100 % FA along with 80% FA + 20 % RP mix. It can be seen that for 80 % FA mix the (%) transmittance is getting decreased with respect to 100% FA .With comparison of FTIRspectrum phase transformation of FA and FA mix can be recognized. The most characteristic difference between the FTIR spectrums of these two is the shifting of band attributed to the asymmetric vibrations ofSi-O-Si and Al-O-Si. The broadness in band appeared to be around 1250 cm-1 in the FTIR spectrum, which became sharper as compared to FA mix. . Then after these bands starts shifting towards low frequencies at around (950 cm-1) indicating the formation of a gel like phase named alumino silicate which is connected with the suspension of fly ash in the strong alkaline activating solutions. Stretching vibration of Si-O-Al appeared at around 600 cm-1. The wide band groups showed up in both IR spectra in the area of 3500 cm-1 are assigned to stretching (-OH) and bending (H-O-H) vibrations of bound water atoms, which are surface consumed or entangled in the huge depressions of the polymeric skeleton [30, 31]. This broadness indicates the presence of strong hydrogen bonding [32].

As a conclusion, water content is a crucial synthesis parameter that affects their mechanical strength. Peaks appeared around 2400 cm-1 attributed to O-H stretching. The gradual decrement in the intensity and broadness in the band confirms the loss of water. Peak at 3000 cm^{-1} – 2000 cm^{-1} could be assigned to C-H stretching vibration of organic contaminants which may be introduced during sample handling or some hydrocarbon present in fly ash [33].

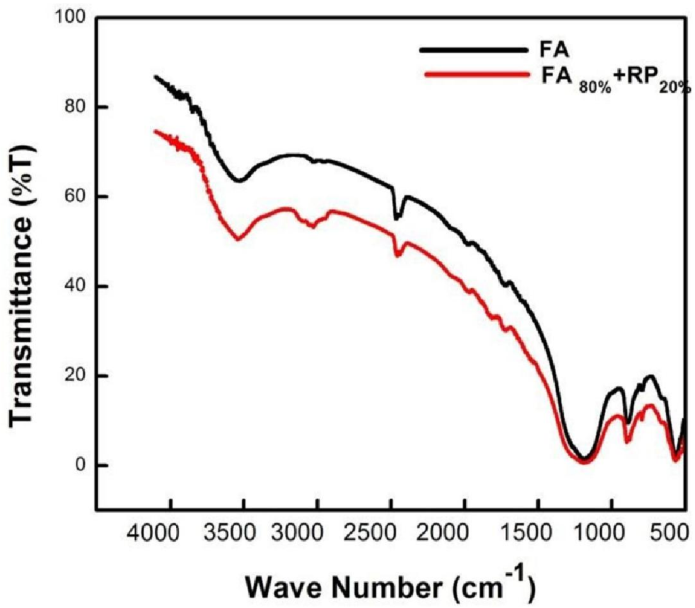

Fig.4.5 IR spectra of the FA and FA resin Powder mix

Determination of Compressive Strength

The compressive strength measurement of the cylindrical samples was done as per standard practiced. Test was conducted on the three samples of each composition and the average value of all is evaluated. Table 4.5 shows the strength values of different compositions of FA, both in dry and wet state. For dry composites, the Compressive strength value lies in the range of 6.5 to 11.28 MPa. 85 wt. % FA compositions have got the highest strength value while the lowest strength value of 6.5 MPa was gained by 75 wt. % FA composition

S.NO	Mix Composition (Wt. %)	Compressive Strength (MPa)	
		Dry	Wet
1	(FA)75%+ (RP)25%	6.5	5.52
2	(FA)80%+ (RP)20%	8.73	7.98
3	(FA)85%+ (RP)15%	11.28	9.43

Table 4.5 Compressive strength values of different FA resin mix compacts

It can be seen from Figure.4.6 that the composition of (FA) 85%+ (RP) 15% has higher compressive strength than other two compositions. It is found that with decrease in the resin percent with fly ash mix has increased the compressive strength. As it is evident from SEM micrographs that 75 wt. % FA mix composite possesses cracks which leads to

IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
Impact Factor: 7.53
Volume 4, Issue 4, March 2024

decrement in compressive strength. As the percentage of FA is increased there is a good bonding between the interfaces which leads to improvement in strength of the compacts. These observations confirm that addition of cold setting resin powder in excess to fly ash may not be beneficial. Here resin powder is only used as a bindingagent. Water treatment shows a little bit negative impact on the strength of composite.

Thermal Conductivity Measurement

Thermal Conductivity is the property of a material depicting its capacity to exchange heat .The Thermal Conductivity of the Fly ash–Resin Powder mix were determined through Hot wire method using KD2 pro analyzer .It was reported that the conductivity of FA- resin powder mix decreases with increase in FA content.75 wt. % of FA composition displayed the maximum thermal conductivity value with an average of 0.0552 W/mK. Only FA powder shows a conductivity value higher than other three compositions.

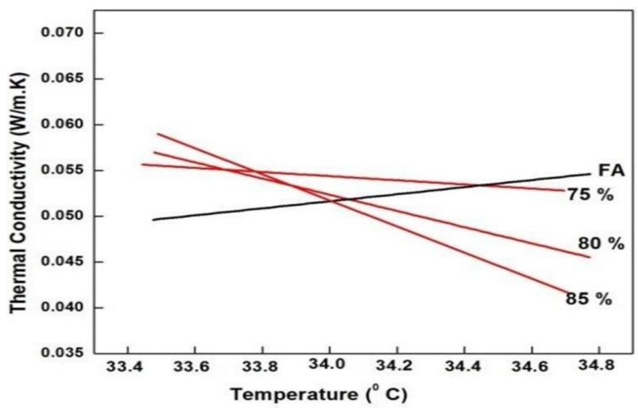

Fig.4.7 Thermal conductivity of FA –Resin powder Mix at different compositions

The thermal conductivity of clay is around 0.82 W/mK which is very much higher than FA. Henceit can be concluded that FA can be used as a substitute material instead of clay. An important result can be drawn from the graph that by adding the resin powder, conductivity value decreases to some extent. The low thermal conductivity of FA is desirable for making several electronic insulating devices. Tests were carried out to assess the insulation capability of the resin added FA mix, shown in Fig 4.7. It is found that the thermal conductivity of FA increases with increase in temperature, whereas in case of resin powder FA mixes, the conductivity of composite decreases with increase in temperature.

Wear Resistance and Friction study

Wear Study: Wear characteristics of FA polymer composites were carried out at different loads of 10N and 20N. Fig.4.8 (a) shows the plot between wear depth (µm) and time (s) of dry composite at load of10N. It can be seen that the wear resistance of 75 wt. % of FA composition is less as compared to the other two composites. It can also be correlated from the results of hardness value mentioned in Table 4.4. For dry compacts, 75 wt. % FA possesses less hardness value than the other two. Similartrend in wear behavior was observed in both the cases. Fig.4.8 (b) shows a plot between wear depth (µm) and time (s) of wet compacts at 10N load. In this case too, 75 wt. % FA compact is less resistant than other two. Since the hardness value of this is lower than the other two compositions. The only difference is that the wet compacts became harder in presence of moisture which in turn the wear depth decreases to a value of (275-250 µm) and is lower than dry compacts(350-280 µm). Fig.4.8 (c) and (d) shows the plot between wear depth (µm) and time (s) of dry and wet compacts at load of 20 N respectively. From figure (c), it can be seen that by applying load of 20 N the wear depth increases to a value of 350-450 µm which is comparatively higher than that of 10 N weared dry compacts.

IJARSCT

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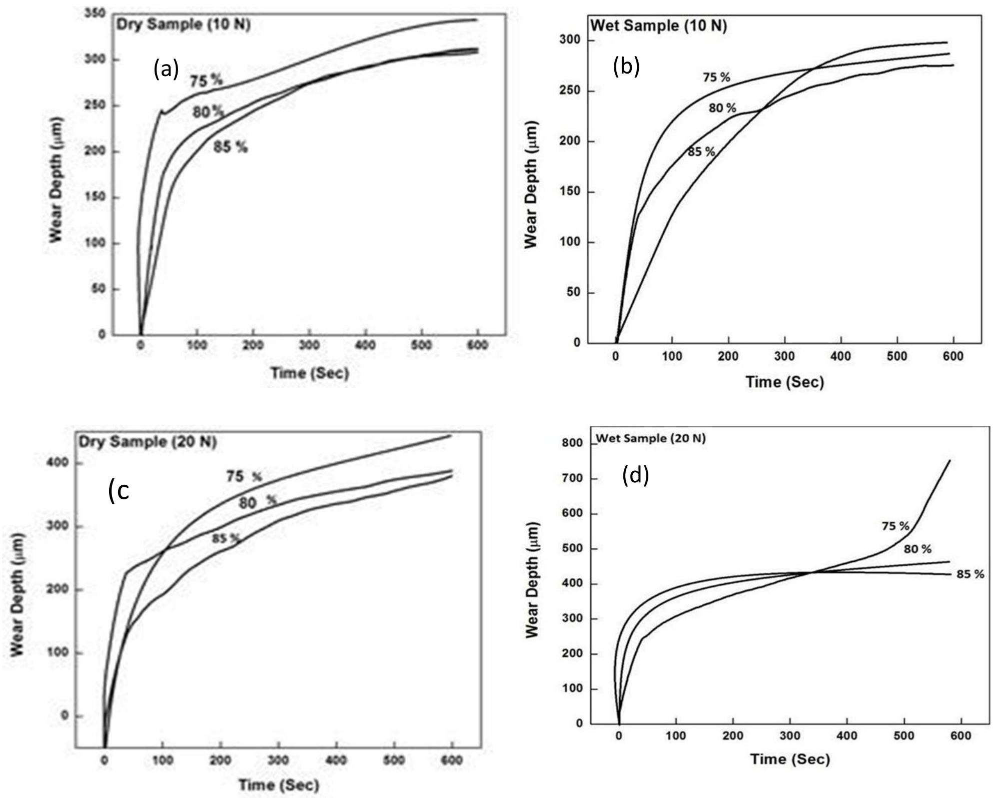
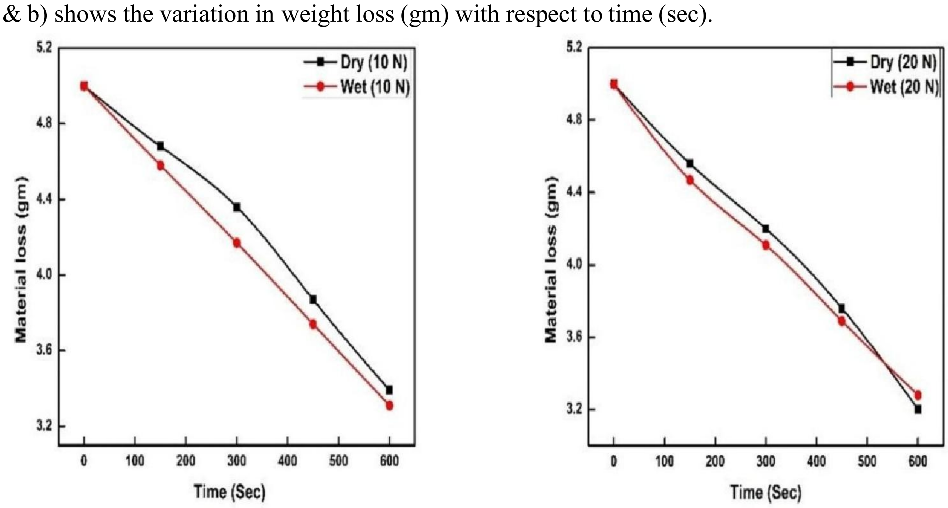


Figure 4.8 Wear behavior of FA compacts at different loads

Since wet compacts are very much harder than the dry one hence by the application of 20 N load smooth curves appears as shown infig. (d) .The wear depth value in this case decreases little bit and this curve follows the trend of steady state. It can also be observed that for the 20 N applied load at a time of about 300 sec the wear depth becomes same irrespective of FA composition. At first it reaches to a value of 400 µm and then a follows constant horizontal saturated line. Moreoverwear behavior can also be co-related with the help of wet density.

Figure 4.9 (a & b) shows the variation in material loss (gm) vs time (sec).It is found that materialloss decreases with increase in sliding time implies the reduction in wear rate with time. The weightloss is more in case of dry samples than water treated samples. There is not much variation with applied load.

Fig.4.9 (a & b) shows the variation in weight loss (gm) with respect to time (sec).



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Volume 4, Issue 4, March 2024

Friction Study: The frictional behavior of Fly ash polymer composites has been shown in Fig.4.10.The average co-efficient of friction (μ) value of all the composites has been reported to be (1.1-1.4). Chapmanet.al, Suggested that spines in the frictional behavior of the composites could be related with the flaw generation from the edges of the material. They estimated that spines in the value were accompanied with an elevated pitched noise [29]. From our studyas shown in Figure 4.10 (a & b), it is observed that initially there is a slight increaseof co-efficient of friction (μ) , but just after few seconds the μ value follows linear trend throughoutthe further time of testing. It can also be seen that co-efficient of friction decreases with an increasein FA composition.

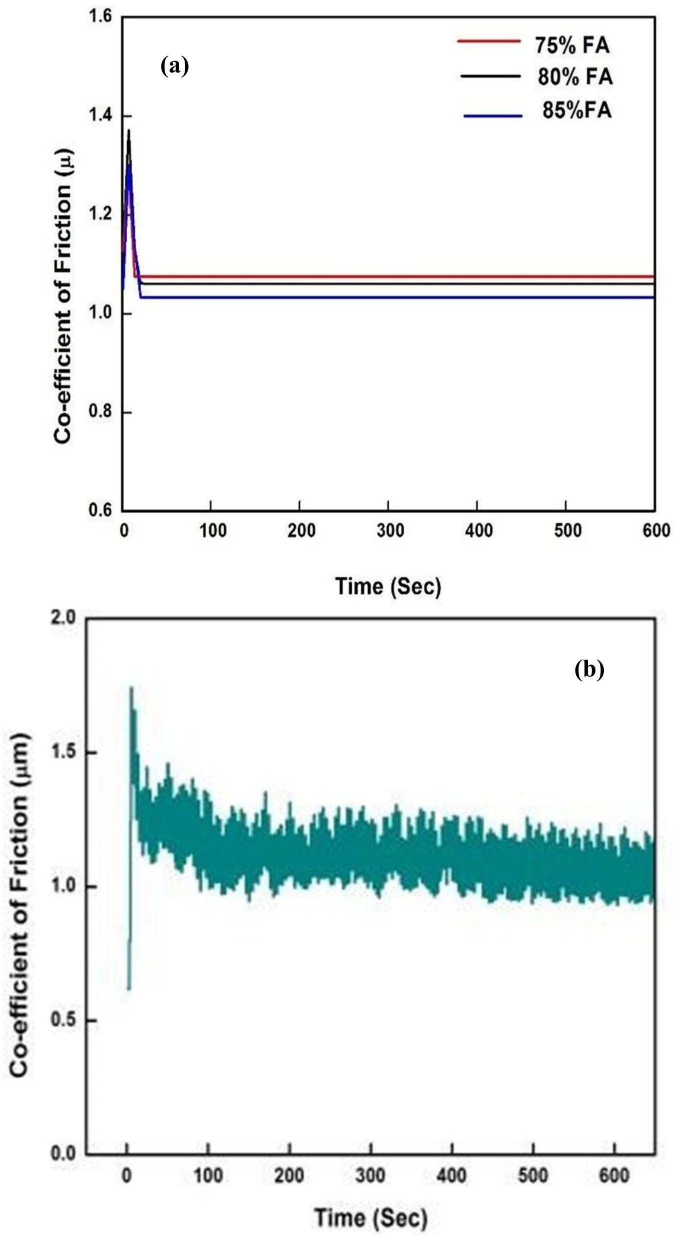

Fig.4.10 (a, b) Variation in co-efficient of friction w.r.t FA composition

IJARSCT

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Microstructural study of Fly ash polymer Composite

1) SEM Analysis: Microstructure of the composites with 75, 80 and 85 wt. % FA plus resin powder mix was studiedby the SEM at different magnifications. Particle size of FA powder was also determined. It has been found that the particle size of FA lies in the range of 9.63- 47.6 µm.

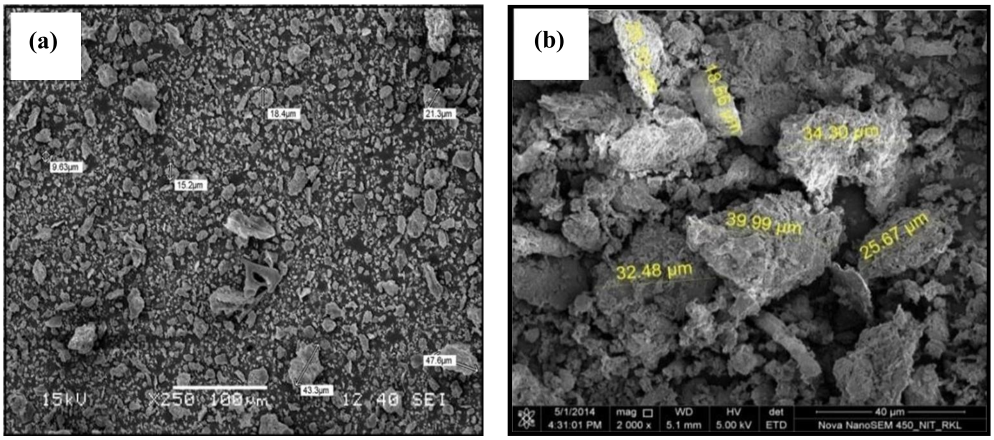
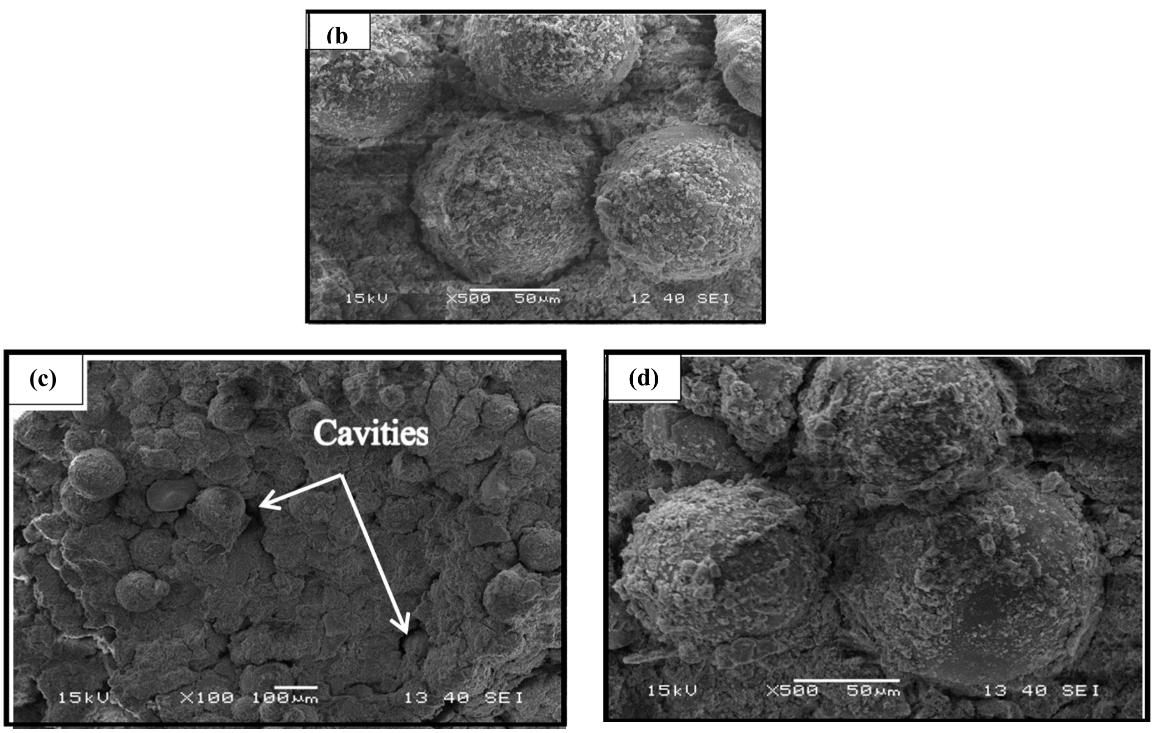


Figure.4.11 (a, b) Particle size distribution of FA powder at different Magnification

From the SEM micrographs it has been observed that FA particles are mostly spherical, messy and irregular in shape. FA particles are formed Coagulated junks. In case of 75 wt. % FA composition as shown in Figure 4.12 (a) the cracks at the inter particle boundary seems, pore type of interface periphery. With decrease in polymer addition i.e. increase in Fly ash amount it is seen that the interface bonding becomes better and less amount of cracks at the interfaces. With further decrease in resin addition, although there is good compaction but elongated cracks/cavities are found along the boundaries.



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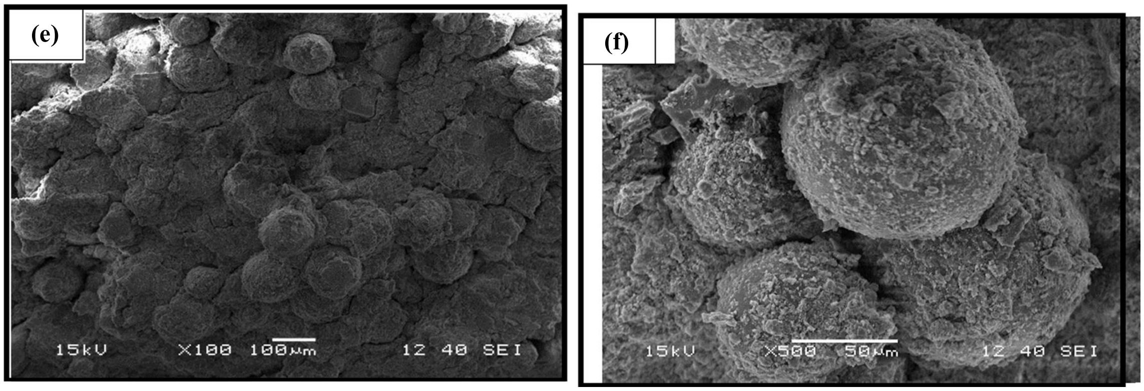

Fig.4.12 Morphology of Fly ash compacts with different composition and at differentmagnifications

Figure 4.13 shows the FESEM micrographs of wear track along sliding direction at different magnifications. These images show that wears mechanism is basically delamination, ploughing ofthe surface, formation of micro cracks and rubbing of tribolayer. Figure 4.13 (a) shows that Microcracks have been initiated in direction perpendicular to sliding distance which leads to wear of thesurfaces.Figure 4.13 (c) shows the wear track of dry compacts (80% FA composition) at very lowmagnification.

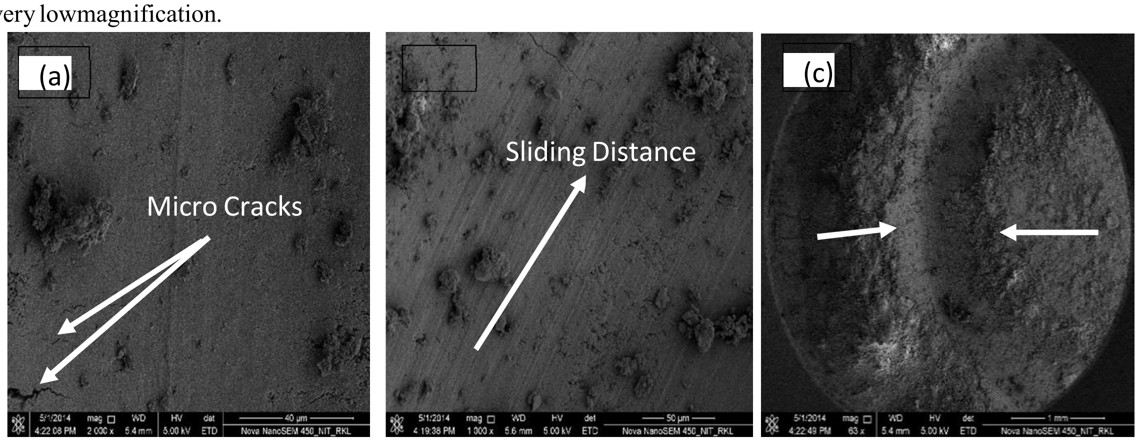

Fig. 4.13 FESEM image of wear track at different magnification

V. CONCLUSION

On the basis of present study following conclusion can be drawn:

- Water treated compacts shows positive effects on the hardness values. Out of all dry compacts, FA with 85 wt. % possesses a higher hardness value of 44.08 HV. Much improvement in the hardness value is achieved when the composites are treated in water at 110^0- 180^0C and this value rose to 47.37 HV. This increment in hardness value is due to the presence of CSH and CASH in the presence of moisture as obtained from XRD analysis.
- With an increase in polymer addition (resin powder), the compressive strength of dry compacts decreases to a lower value of 6.5 MPa. Composition of 75 wt. % FA shows lowervalue. No significant reduction in Compressive strength is achieved in the case of wet compact
- Wear study of different composites can easily be correlated with the hardness value. In both the dry and wet state, FA with 85 wt. % composition shows better resistance to wearthan other two compositions. Wear resistance increases with increase in FA content. The co-efficient of friction decreases with increase in FA percentage and follows a linear trendthroughout the time of testing.
- Thermal conductivity of FA increases with increase in temperature, whereas in case of resin powder FA mixes, the conductivity of composite decreases with increase in temperature. A much lower conductivity value is obtained and hence can be used as a substitute material with respect to clay.

- Water absorption increases with increase in FA content. Maximum of 19% water is absorbed in case of 85 wt. % FA.
- Density of dry compacts decreases with increase in FA content. While in case of wet compacts, it increases with increase in FA content.
- SEM analysis revealed the morphology of FA particles that are mostly spherical in shape.With decrease in polymer addition i.e. increase in FA content the interface bonding becomes better and less amount of cracks were found at the interfaces.
- XRD analysis revealed that FA particles mostly consist of Silica and alumina with less percentage of Fe_2O_3, Cao and others. The Fly ash –resin powder composite produced in the present study seem to be appropriate for use as construction material. Theproduction of this type of composite will certainly contribute to the use of fly ash for value added products. On the other hand, the reduction in clay usage for the production of conventional clay bricks will help to protect the environment.

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