DEVELOPMENT OF DUCTILE ULTRA LIGHT WEIGH CEMENTITIOUS COMPOSITES FOR STRUCTURAL APPLICATIONS

Submitted By

Marium Saleem

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00000172090

Supervisor

Dr. Muhammad Usman

NUST INSTITUTE OF CIVIL ENGINEERING (NICE),
SCHOOL OF CIVIL AND ENVIRONMENTAL ENGINEERING (SCEE),
NATIONAL UNIVERSITY OF SCIENCES AND TECHNOLOGY (NUST).
SECTOR H-12, ISLAMABAD, PAKISTAN

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Signature: ________________________

Supervisor: Dr Muhammad Usman

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Declaration

I certify that this research work titled "***Development of ductile ultra-light cementitious composites for Structural Applications***" is my own work. The work has not been presented elsewhere for assessment. The material that has been used from other sources it has been properly acknowledged / referred.

Marium Saleem
Fall 2016-MS Structural Engineering
00000172090

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Dedication

I dedicate this research to
Prof. Dr. Muhammad Usman, my mentor
And
To my parents

ABSTRACT

The objective of this study is assessment of the properties of fiber reinforced cement based composites and fiber reinforced cement based composites having fly ash cenospheres (FACs) as light weight filler material. The main focus on the improvement of ultra-light weight cementitious composites with exceptional mechanical properties at the age of 28 days. Fly ash Cenospheres (FACs) was used as a filler material. Polyethylene fibers (PE), Polypropylene fibers(PP) and Polyethylene Teraphthalate fibers (PET) were used to enhance the mechanical properties of the cementitious composites. The resultant composites depicted exceptional mechanical properties with elevated specific strength as 23.93-33.87 KPa/kgm^{-3}.The 28-days compressive and flexural strength were determined in the range of 36-48.1MPa and 4.15-8.06 MPa respectively. The high pozzolanic activity of FACs added towards the enhanced mechanical properties even at lesser density. Microstructural analyses of composites were done by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDS) spectroscopy. Results specified that FAC is a favorable material for generating lightweight and strong structural members for utilization in building structure which can endorse viable development. Though, too much FACs content in composite might cause elevated porosity because of chemical composition and hollow spherical shape of FAC particles which influences durability and mechanical behavior of cement-based composite.

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List of Abbreviations

SCC	Self-Compacting concrete
SRMs	Secondary raw materials
ACI	American Concrete Institute
ASTM	American Society of Testing Material
FACs	Fly ash cenosphere
LWF	Lightweight filler
PE	Polyethylene fibers
PP	Polypropylene fibers
PET	Polyethylene Teraphthalate fibers
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive X-ray
LWA	Lightweight aggregates
SCP	Self-compacting paste
PVA	Polyvenyl alcohol
OPC	Ordinary Portland Cement
SCMs	Supplementary cementitious materials
LG	Laser granulometry
XRF	X-Ray Fluorescence
BET	Brunauer-Emmet-Teller
LOI	Loss of ignition
PSD	Particle size distribution

CHAPTER 1: INTRODUCTION

1.1 General

In the quickly altering world of technology, development is carried out in all the areas of life. The world is changing from the conservative to innovative ideas. In this race of development, concrete technology is not in back. Everyday research untangles new vision of construction building, resolving the inadequacies of the past practices.

For centuries, the human race spent a good amount of effort into the old fatiguing exercise of manual construction. Mega constructions in the past took the lives of hundreds of people. For then, transportation and placement of building materials was very difficult.

After tiring effort finally, the era of slow and arduous construction was overcome by the discovery of a truly remarkable material, which we all know as self-compacting concrete (SCC). This material, through its astonishing properties, has managed to take the era of construction years forward and way easy.

The introduction of self-compacting concrete has made construction much easier and way faster. More architectural variations and improvements have been added to the structures with much better precision and finish. The problems of improved strength and durability were also better addressed. In other words, it redefined the field of building construction.

Now, with the mass construction made this much easier, the cement demand was significantly rising up and so was its carbon footprint. So that in order to counteract this problem, researchers came up with the idea of introducing secondary raw materials (SRMs) into the cement mix. They provide a relatively cheap and environment friendly replacement of cement. Further, they pass on useful properties to the cement mix as well.

The use of secondary raw materials (SRMs) enhances durability and strength by improving microstructure of concrete. It also improves strength by improving density via better packing and offering of extra nucleation sites. The use of these SRMS provides light weight concrete.

First put to test in 1980’s [1], self-compacting concrete (SCC) already has and is still evolving with the extensive research being carried out in the whole world. This particular study is also a humble addition to this set of knowledge.

1.2 Self-Compacting Cementitious Systems (SCCS)

Different standards define self-compacting concrete in different ways, but state the same axiom. Some of these are stated as under;

American Concrete Institute (ACI) defines self-compacting concrete like fresh concrete that can consolidate inside formwork and flow around reinforcement under self-weight without vibration [2]

American Society of Testing Material (ASTM) states it to be concrete that can consolidate under self-weight and flow around reinforcement with no extra effort and lacks more than the specified limit of segregation [3]

According to ENFARC SCC is concrete that is capable to flow and strengthen below its self-weight, entirely fill up formwork still in thick reinforcement, whist keeping homogeneity and lacking need for extra compaction [4].

However SCC is a get through in the practical application of architectural range, yet the need of costly chemical admixtures and high range of cement had restricted its use in the everyday construction. So the researchers gave the idea of replacing chemical admixtures with the mineral admixtures. This opened a new door way to the self-compacting technology, where the use of SRMs not only lowered the carbon footprint of the cement in the atmosphere but also reduced the SCC production cost. Their use also gives durability to the systems and put industrial waste to a constructive removal [5-7]. The SRMs, their origin, pozzolanic activity and advantages are mentioned in coming chapters of this research.

1.3 Secondary Raw Materials (SRM)

ACI defines supplementary cementitious materials (SCMs) or secondary raw materials (SRMs) as “Inorganic material that reacts pozzolanically or hydraulically for instance silica fumes, meta-kaolin, fly ash, or slag cement” [2]

ASTM defines SRMs in specification C1697-16 like “a pozzolan or slag cement that gives to the properties of concrete or mortar in the course of hydraulic or pozzolanic action, or both” [10]

Pozzolans are added during mixing phase of the cement formulation, so SRMs are also known as mineral admixtures. ENFARC elaborates SRMs as material added in minute amount during mixing process of concrete related in to the cementitious binder to change the characteristics of hardened or fresh concrete [4].

1.4 Fly ash Cenosphere

The cenosphere word is a combo of two Greek terms: kenos (hollow) & sphaira (sphere) which explain the main points of this matter. They are attained in coal combustion process from coal combustion power plants [8]. Since 1984, usage of FAC is common as a lightweight filler (LWF) material in fabrication of lightweight cementitious composites (LWC) by Montgomery and Diamond [9]. Cenospheres are hard, light, waterproof and insulative. Nowadays, Cenospheres are used as fillers in cement to manufacture concrete of low density. In recent times, some makers have started filling polymers and metals with cenospheres to make light-weight composite materials with greater strength as compared to other foam materials types. Such composite materials are known as syntactic foam.

Alongside with bottom ash and fly ash, a substantial amount of remaining waste comprises of thin-walled and spherical particles which are comparatively bigger in size (10–400 mm) as compared to fly ash [13] termed as cenospheres. FACs are the void, spherical, lightweight and inert alumino-silicate particles [19]. The shade of FACs is greyish white and the chemical composition and shape is approximately alike to fly ash [11]. Because of the chemical composition, it is taken as a worthy industrial waste by-product [12] and its valuable use helpful to sustainable development. Speedy urbanization and consumption of coal for electricity has severely amplified the waste generation per annum. Even though, fly ash has been previously used as a supplementary cementing material (SCM) used to replace cement in concrete. The quantity of fly ash utilized is minute, having a huge part last to disposed in landfills. Utilization of FACs as LWA reduces the waste to a larger extent and with the additional inducement of lesser unit weight; durability and mechanical characteristics of resulting composites may be enhanced.

1.5 Problem Statement

The objective of current Research was to produce Light Weight Cementitious Composites (LWCC) containing Fly ash cenosphere fillers, different fibers and present its potential fields of applications in concrete.

1.6 Research significance

Little published work on the use of Cenosphere fillers with different kind of fibers in self-compacting cementitious systems in literature. Besides this, FACs are waste material it is dumped in landfills. Considering the all benefits of FAC incorporated components, FACs is used with different types of fibers to achieve the ductile and ultra-light weight cementitious composites for structural applications.

Cenosphere incorporated cementitious composites are cost effective and due to its chemical composition considered as a worthy industrial waste and its beneficial utilization can help sustainable development. Rapid utilization and urbanization of coal for electricity has considerably enhanced the annual waste generation. Though fly ash has been previously used as a SCM as an alternate of cement in concrete, but small quantity is utilized leaving an immense portion after to be wasted in the landfills. So using FACS as Lightweight aggregates (LWA) decreases the waste up to a greater extent.

Besides this, FAC incorporated cementitious composites are excellent in making thermally insulated and mechanically strong composites having immense potential to be used in energy conservation buildings. FAC incorporated composite structures are economically viable and environmentally friendly.

1.7 Research Methodology

The research was started with the conveyance of relevant literature review. Having gained a deeper insight into the topic in question, some elementary tests were conducted on Cenosphere fillers to see how this filler behaves in moisture and changeable temperature conditions. To find out the type of organic compounds that are present in the cenosphere filler, characterization tests such as LG, XRD, SEM and XRF were conducted. These tests were performed to evaluate the response of cenosphere fillers in self-compacting paste (SCP) systems by using three different types of fibers. After these tests, the main laboratory tests were carried out. These included tests for flow, setting times, density in fresh formulations and the compressive strengths. In conclusion of detailed debate with logical reasoning in light of relevant literature review and supervisor and researcher’s own knowledge and understanding about the subject are presented to understand the behavior of cenosphere fillers in SCPs.

1.8 Research objectives

- Enhance the strength and toughness of FAC incorporated composites by using different types of fibers.
- Improve the ductility and strain hardening behavior.
- Achieving ultra-light weight concrete
- Improving Flexural strength and toughness of cementitious composites.
- Achieving economical concrete

1.9 Research scope

This research was conducted on self-compacting paste (SCPs) systems. The purpose of current research was to establish an approach into the analysis of Cenosphere fillers based cementitious systems. The results produced in this study can be used as basis for future researches on concrete.

1.10 Thesis organization

The first chapter gives an introduction to the thesis topic. The next chapter presents the literature review. The third chapter presents various standard methods and apparatuses employed for conduction of laboratory tests. The fourth chapter presents discussions and reasoning. The fifth chapter gives conclusions and recommendations.

CHAPTER 2: LITERATURE REVIEW

2.1 General

Literature review will first cover a brief overview of light weight concrete (LWC) and its development, the manufacturing mechanism of LWC, its advantages and effect on the environment. Afterward, secondary raw materials (SRMs) will be discussed in detail. This research will include introduction of pozzolan, the mechanism of their reactive behavior and their advantages. After that, studies already available on cenosphere fillers will be put in writing. The investigational techniques will be discussed for LWC systems. These will include SEM and EDX analysis.

2.2 Introduction to Lightweight concrete (LWC)

Lightweight concrete is a structural concrete having low density aggregate with a 28-days compressive strength of greater than 17.2 MPa and air-dried density of fewer than 1850 kg/m^3 as per ACI committee 213R-14 2003. [1]

Lightweight concrete (LWC) is a low density concrete, light in weight and adequately strong when used with steel reinforcement. Thus it is going to be more suitable and economical construction material than conventional concrete.

LWC has attained significant concentration of researchers in previous some decades, even though its utilization traced back to 3000 BC [14]. Light weight cementitious systems may be broadly classified into the following systems

- Light weight paste systems
- Light weight mortar systems
- Light weight concrete systems

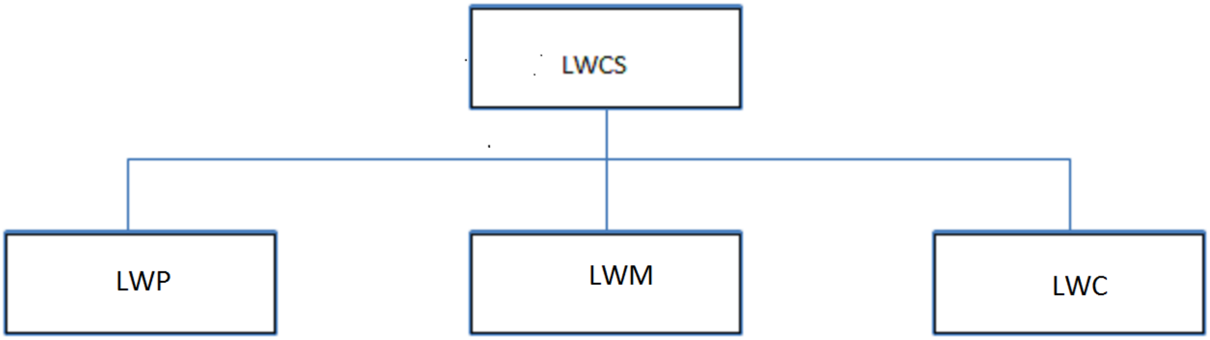

Figure 2.1: Schematic of LWCS

2.2.1 Background/ Development of LWC

The use of LWC is traced back to 3000 BC. The concept of light weight concrete was conceived by Romans. They used most common type of aggregate named Pumice in the second century . After that, the use of lightweight concrete has been extensively spread across other countries such as USA, United Kingdom and Sweden. LWC offers excellent durability and better thermal insulation.

Since then, extensive research has been carried out on this remarkable material to further enhance and customize its properties for custom use.

2.2.2 Advantages

As discussed above, the advantages of using LWC can be summed up in the bullets as under;

- Durability to chemical and frost attack due to low permeability
- Better thermal insulation
- Higher fire resistance
- The unit weight of LWC is from 1200 - 1800 kg/m^3.
- Reduced overall construction cost
- Excellent surface finish and durability

- Small cross sectional structural members and foundations can be adopted as dead load has been reduced.

2.3 Lightweight cementitious composites

LWC is valuable over normal weight concrete due to decrease in dead loads, easy to handle, and enhanced durability. It has been generally in use in floating marine structures and long-span bridges [15,16]. Furthermore, the improved fire resistance [17] ,durable to chemical and frost attack with lower permeability and enhanced thermal insulation properties [15] more supports its utilization in building structures such as facades and roof coverings, to enhance the thermal insulation properties and fire safety of infrastructures. As it is capable to attain the desired structural and mechanical properties, vigilant selection and proficient application of lightweight filler (LWF) materials is of imperative significance.

2.4 Lightweight Filler (LWF)

Fillers are materials whose function in concrete is based generally on its shape and size. It can interact with cement in numerous ways; to get better packing of particles and provide the fresh concrete other properties, and diminishes the quantity of cement in concrete with no reduction in strength.

Normally, a variety of fillers are utilized as lightweight aggregate (LWA) in cementitious composites to obtain lesser unit weight with enhanced functional and mechanical properties. It comprise natural materials like waste glass [18,20], pumice [21,22], Palm oil shells [23,24], volcanic ash [25,26], expanded polystyrene beads (EPS) [27-30], glass microspheres [31,32], expanded shale [33,34] and other materials [21,24,35-39]. These materials are appreciably working in LWC development, though a no. of problems connected with their utilization in cementitious composites has obstructed their application as a workable and structurally sound choice, because of minute mechanical strength, privileged water absorption and porous nature of these aggregates. Main issues are brittle behavior & lesser mechanical strength of resulting concrete, better porosity and greater voids in microstructure, larger CO_2 emissions related with manufacturing viewpoint and utilization of natural resources compromising sustainability. For resolving these issues, Fly ash cenosphere has been used as Lightweight filler with different kind of fibers.

The first research on FAC by Montgomery and Diamond [9] dates back to 1984. FACs are an appropriate filler material used in production of green light-weight binder paste due to their source attained from coal fired power plants as a waste residue [40], light-weight nature i-e bulk density ranges to 800 kg/m^{3}and fractional reactivity [41,42] and cost effective.

2.4.1 Role of filler/ supplementary cementing material (SCM)

Supplementary cementing material (SCM) and various fillers are often used by many researchers in self-compacting cementitious systems to attain better strength, economy, durability and environment friendliness.

2.4.2 Effect of w/p Ratio and Super Plasticizer Content

Abraham's law [44] discovers a counter relation among the w/p ratio and strength of the cementitious system. Therefore, one of the primary requirements of the SCC development is to use a lower w/p ratio. While using high w/p ratios results segregation and yield low strengths. Since lower w/p ratio cannot yield sufficient workability, but by adding high range water reducing admixtures (HRWRA) or super plasticizers (SPs), this purpose can be achieved.

Apart from imparting sufficient segregation resistance and deformability, the use of low w/p ratios and super plasticizers, give the advantages of low porosity, improved microstructure, strength and durability [45].

2.4.3 Environmental Effect

Sharif et al. found that cement production process utilizes extensive energy which results in consumption of natural resources and also a source of global warming [46-48]. This concept has been authorized by other researchers as well [49].

Literature constantly states that by using of SRMs largely decrease the CO2 emission and other harmful gases by falling the amount of cement utilized in the formulations.

By using industrial solid waste as mineral admixtures, sustainable concretes can be made, which could maintain the non-renewable energy resources and the environment. In addition, this practice can set wastes such as FAC i.e. fly ash cenospheres to a productive use.

2.5 Pozzolan

Pozzolans are aluminous and siliceous materials which have no or minute cementitious value but in finely divided form and water presence, pozzolans interact chemically with calcium hydroxide at normal temperature to make compounds having cementitious characteristics. Its reaction with calcium hydroxide and water is measured by its pozzolanic activity. Pozzolans are volcanic origin by nature.

Pozzolans are both natural and man-made, and utilized as Supplementary Cementitious Materials (SCMs). Volcanic ashes and Pumices are naturally occurring pozzolans. Artificial pozzolans are produced purposely e.g. meta-kaolin is attained by thermal activation of kaolin-clays, fly ash is attained from coal fired electricity process as a by-product from high temperature process and silica fume is achieved by silicon smelting. Mainly used pozzolans nowadays are industrial waste product silica fume, fly ash, and residues of burned organic matter abundant in silica include rice husk ash and highly reactive meta-kaolin. Pozzolan are largely used as secondary raw materials (SRMs) in many countries.

As stated by Johari et al., various materials have various effects on cementitious matrices properties and can be utilized as partial cement replacement materials or mineral additives as their various mineralogical and chemical compositions and particle characteristics, which is helpful in finding their reactivity, water requirement and packing ability when utilized as a part of binder for concrete. Generally, these materials usage in concrete can be associated with the improvement of the concrete pore structure [50].

2.5.1 Mechanism of Pozzolanic Behaviour

The pozzolanic activity depends upon two factors; reaction rate between calcium hydroxide and pozzolan in water presence or degree of reaction over time and quantity of calcium hydroxide accessible for reaction purpose with pozzolan. The quantity of calcium hydroxide is dependent on chemical properties of that pozzolan which is to be used, the essence of its active phase, the silica content in the active pozzolan and calcium hydroxide/pozzolan ratio in mixture. Pozzolanic reaction rate is reliant on the natural properties of pozzolan, in particular chemical composition, specific surface area, active phase content and temperature.

Hanif et al states that by addition of FACs, CH content decreases but CH content enhances with curing time. However, after passing 28 days, reduction in CH content is directly proportional to FACs addition.

The material used as an SRM depends upon its two properties, its fineness or the amorphous silica content. For every specific material, pozzolanic activity depends upon only one of these two properties. For some materials, the net pozzolanic activity depends upon both these parameters.

2.5.2 Physical Packing Effect

Properties of cementitious systems can be improved due to the filler effect of the SRM or pozzolan is known as Physical Filler Effect. Due to physical filler effect, SRMs show improved performance of cement formulations when its silica content is in crystalline state.

SRMs reduce the voids in the cement paste, facilitate placements and to obtain the denser microstructure and durability, SRMs used as an alternative in self-consolidated concrete (SCC).

Pozzolan usually have particle sizes lesser than that of cement, resulting in improved overall packing density. This will basically increases strength of the cement formulation.

In the presence of super plasticizer, the use of SRMs with continuously graded aggregates normally bring about lowering the voids due to packing effect and the cement requirement. But in the presence of poorly graded aggregates, physical packing effect is reduced.

In the process of production of cement, emission of co_2 and other gases occurs but due to SRMs reduced cement content achieves environmental benefits as to overall reduction in cement content and consequently reduces emission of $co_2$and other gases and saving in energy.

Other than denser packing, density also considers for reduced porosity i-e number of pores, their size and connectivity- secured lower water permeability [51]. Due to the reduced permeability normally lesser bleeding and evaporation occurs, as well as lesser possibility of corrosion, hence overall durability of the system improved [52].

2.5.3 Chemical Pozzolanic Effect

Silica is major constituent relating pozzolanic activity in all pozzolanic materials .The chemical activity of pozzolan has been elaborated substantially in literature. Cement reacts with water to form calcium hydroxide (CH) and calcium hydrate silicate (CSH) gel at first. This makes calcium hydroxide (CH). This reacts with SiO2 found from pozzolan to form additional CSH gel. This supplementary CSH is responsible for additional strength achieved due to the addition of pozzolan in the cement mix.

Hanif et al. [31] found that fly ash cenospheres (FAC) composition was about 73% silica. Wang et al.[32] and Xu et al.[33] also obtained alike values in percentages for silica composition as 60% and 61%, respectively in FAC. So under ordinary conditions FAC has elevated levels of silica, and if possible some factors of calcination are used as controlled calcination temperatures, burn time and rate of heating.

2.5.4 Advantages

Sharif et al. states that by addition of pozzolanic industrial waste materials enhances the strength and durability of concrete and also found helpful in minimizing global warming through productive ejection of waste material [53] .

By adding finer materials as an alternative of cement have manifest incredible development in packing density, lessening of thermal cracks, improvement of particle distribution and in improving mechanical properties [54].

Pozzolanic characteristics material may utilized in partial replacement of cement in concrete or mortars, and has been revealed to enhance the resulting products durability [55,56] .

Accordingly, the advantages of using SRMs in concrete systems can be summed up as follows;

- with lesser amounts of cement in both fresh and hardened state, Use of SRM can lead to getting of desired properties in concrete
- Use of SRM will decrease cement content which is an expensive component of concrete.
- Filler effect of SRM in concrete system give better mechanical properties like compressive and flexural strengths and enhanced microstructure.
- SRMs are finer than cement hence they provide better packing effect and better volume stability.
- Use of SRMs results in an environmental friendly concrete system

2.6 Fly ash cenospheres

Broad researches have been done on the usage of Fly ash cenospheres in cement formulations. Literature portrays utilization of Fly ash cenospheres in SCC systems for many purposes. Some researchers used it as a supplementary cementing material (SCM) to replace the cement content in concrete. By the FACs addition as filler material, density of FACC reduced directly and the resultant composites have the qualifications of structural light- weight concrete [57,58,59]

Huang et al. [60] use diverse industrial waste byproducts together with FACs , fly ash and iron ore tailings, and found that FACs are advantageous in lessening the entire density of resultant composite while sustaining adequate levels of mechanical properties which is the demand of current research. More, FACC integrated with SCMs like silica fume and fly ash have enhanced mechanical strength and packing characteristics. [61–63].

J.Y Wang et al. stated that elastic modulus of FACC is quite lesser than ordinary weight concrete because of its lesser density and compressive strength [62].

Figure 2.2: Fly ash cenosphere fillers

FACs based cementitious composites are pozzolanic due to the existence of amorphous silica and lime in FACs .The pozzolanic outcome causes better formation of CSH gel and lesser CH content creating FACC to have elevated mechanical strength at lesser density. [43]

Hanif et al concluded that by using privileged quantity of FACs in cementitious composites need more water content because of the surface area, particle size, and shape. The amount of water is directed by the flow ability of mortars to be casted as molds and forms [77].

Chia et al. [66] assessed shrinkage and creep of concrete including cenospheres and result was that concrete having cenospheres has the maximum creep largely due to the lesser values of elastic moduli and entire shrinkage was the least. Due to FAC inclusion in concrete, the lesser shrinkage indicates the less micro-cracking at early ages, pointing out the enhanced durability by restraining the access of ions and salts.

FACs based cementitious composites are partial reactive. The reactivity is restricted because of chemical composition, glassy surface and particle size of FACs particles. The pozzolanic reaction is comparatively deliberate and causes sufficient strength improvement merely at later ages [75,76].

Hanif et al. used cenospheres with 1% Nano silica and concluded that properties improvement mainly arise due to the pore filling effect and accelerated pozzolanic reaction of NS particles. Calcium hydroxide quantity left behind in the pastes is lower and enlarged formation of CSH added mostly to the enhanced properties [81].

Demirboga [69,70] stated that because of hollow nature of FAC thermal conductivity is affected, which formulate FACs an exceptionally realistic option for thermally insulated concrete.

Hanif et al. were carried out detailed study on porosity and it was concluded that porosity directly by increasing FACS in the cementitious composites. These pores are non-consistent and the permeability values are less which specifies that porosity is not a severe issue up to 50% weight fraction of FACs [40].

Barbare et al. [71] studied the permeability of FACC in which for FAC integrated concrete, all the fine aggregate was substituted with FACs and conveyance of water in the concrete from both plane surfaces through axial length, was calculated. The outcomes were established according to agreement of Washburn kinetics [72] at lesser time periods of 5 to 10 min, for FAC integrated and normal concrete. The moisture taken up by FACs was considerably high than sand representing the porous nature of FACs incorporated concrete, depth of penetration 20% more at time period of 6 min. Though, the efficient pore size for water penetration in sand concrete was more than its equivalent having FACs cementitious composites as fine aggregate, although the corresponding dia. for both was 1 nm. So, it proved that the pores are non-consistent and permeability of FACs concrete is restricted. Later, alike findings on diverse types of LWAs were found by Liu et al. [73] wherever it was supplementary confirmed that LWC has alike conveyance characteristics as normal concrete.

Hanif et al. used PVA fibers in FACs cementitious composites and concluded that FACs are incredibly efficient in making fiber reinforced cement based composites. Though, for improved ductility and further prominent strain hardening behavior, enhanced fiber by volume fraction may be helpful [77].

FACS particles have higher surface area due to low density. Particle size of FACs is inversely proportional to specific surface area likely finer particles have higher surface area of FACs [78,79].

Clarke et al. [57] performed testing on mechanical characteristics of FACC and concluded that normal concrete is less brittle in nature as compared to LWC. Consequently, to get better its ductility and performance, the utilization of irregular micro reinforcement is essential. Many researchers use FACs cementitious composites with fibers like Polyvenyl alcohol (PVA) fiber [80], Polyethylene (PE) fibers [61] and steel fibers [62].

Wang et al. [41] found out the alkali silica reactivity of FACC . The test results point out that the FAC particles were not probably harmful in cementitious systems with respect to alkali – silica reactivity matter. As identified in XRD patterns, slightly the amorphous silica in FAC lead to improved pozzolanic action of FACC influencing the mortar extension by dropping the pore solution pH and mortar permeability. This main outcome more motivated the usage of FACs as Light weight filler.

The natural brittle nature of FACC, akin to LWC, might be altered with addition of fiber into the mix. Between diverse variety of fibers, polyvinyl alcohol (PVA) fibers depicts the most excellent bond and better flexural enhancement of FACC because of its chemical bond with cement matrix.

2.6.1 Composition of Fly ash Cenosphere

Fly ash cenospheres are attained in coal combustion procedure in coal combustion power plant. A considerable quantity of residue waste with bottom ash and fly ash, comprises of spherical and thin-walled particles which are comparatively greater in size (10–400 mm) than fly ash [68], known as cenospheres. In thermal power plants, burning of coal makes fly ash comprising ceramic particles made mainly of silica and alumina. They are made at temperatures of 1500-1750 °C through complex physical and chemical transformation. The structure and chemical composition of fly ash changes appreciably, depending on composition of coal from which they are produced.

2.6.2 Problems accossiated with Fly ash cenospheres

Conversely, because of their chemical composition, glassy surface and particle size, the reactivity of the FACs in the cement composites is inadequate .The pozzolanic reaction is comparatively time-consuming, and led to sufficient strength improvement merely at later ages [75,76]. For counter these problems, three types of fibers are used in this study.

2.7 Super Plasticizers

A super plasticizer or high range water reducers are additives utilized in producing high strength concrete. It may decrease the water demand by 30 % or more without causing unnecessary retardation in setting times. Its uses are summarized as follows

- Establishes a concrete of enviable workability at a lower water-cement ratio. Concrete strength enhances when water cement ratio is less
- Reduces the curing of concrete.
- Increases the workability of the concrete and produce the self-consolidating concrete and high performance concrete. They appreciably get better performance of hardening fresh paste.

  High range water reducers (HRWR) admixtures used to enhance flow ability of concrete, can be classified as Type F or Type G high-range water reducing agents or they could fall in the Type 1 or Type 2 type of ASTM C 1017 depending on their properties. Their classification is done based on the following ingredients; sulfonated naphthalene-formaldehyde condensate, sulfonated melamine-formaldehyde condensate, and polyether poly-carboxylates and polycarboxylate ethers.

These admixtures are more proficient than conventional water-reducing admixtures. These ensure more effectual diffusion of fine materials such as cement and different SRMs [67].

2.8 Reinforcement of Cementitious Composites

The strength of normal strength concrete in tension is usually 1/8 to 1/12 of its strength in compression and it reduces more to 1/17 to 1/20 in case of high strength concrete [68]. A lot of efforts are performed by researchers and scientists to enhance the fracture properties of cementitious materials. Fibers have been utilized to reinforce the cementitious composites, which has enhanced the tensile strength and tensile strain by either controlling the opening of cracks or obstructing the smooth propagation of cracks. Initially steel fibers were used on macro scale; later different types of fibers, with different aspects ratios and concentration were investigated on macro, micro and Nano scales. Fibers with 10-60mm length and 0.1-1.0mm of least dimension are categorized as macro fibers. Whilst smaller than 10mm in length and 10-30µm diameter are categorized as micro fibers.

Steel and micro polyvinyl alcohol (PVA) fibers reinforced in self-compacting cementitious composites, revealed increased flexural strength, toughness and large maximum deflection. High tensile strength and aspect ratio of fibers were concluded important parameters for achieving deflection-hardening [65].

A hybrid mix of 1% by volume PET fibers and 1% by volume PVA fibers revealed sufficient tensile performance and exceptional impact resistance with co-friendly at less price [82].

Recycling of Polyethylene terephthalate wastes was done with the help of NaOH solution and used as fibers in strain hardening cementitious composites (SHCC) to extensively decrease the SHCCs material cost and environmental impact while disposing harmful PET wastes in building industry [83].

Fly ash cenosphere were reinforced with 1% PVA fibers which provided excellent bonding in the composites. However, greater fiber volume fraction can be helpful for improved strain hardening behavior and ductility [77].

J.Y.Wang et al. Studied on PE and steel fiber reinforced FACC and demonstrated that the initial cracking strength of PE reinforced FACC is 17% lesser than its corresponding have steel fibers due to lower elastic modulus of PE fibers having a value of 79 GPa comparing to steel fibers having 200 GPa [62].

Pichor [80] investigated the flexural strength and modulus of rupture for concrete including different volume fractions of cenospheres with polyvinyl alcohol (PVA) and polypropylene (PP) fibers and concluded that fibers enhance the flexural behavior and they too assist extra lessen the density of resultant concrete with high fiber volume fraction.

M. Usman [84] studied the flexural fatigue strength of light-weight lean laminated cementitious composites (LCCs) by including FACs and Galvanized Iron(GI) welded wire mesh and woven fiber glass (FG) mesh were utilized in the manufacture of LCCs as primary reinforcement and PVA fibers are used as irregular reinforcement and found that the LCCs manufactured by inclusion of FACs led to more enhanced flexural fatigue performance.

2.8.1 Polypropylene fibers reinforcement (PP)

Each year an immense quantity of fibrous textile waste is redundant into landfills throughout the world. Mostly there is carpets waste which decay rate is very slow and it is not easy to hold in landfills. For example, yearly disposal rate of carpet in USA is approximately 2–3 million tons, and 4–6 million tons throughout the world. The waste fibers of carpet which cause severe environmental problems could be transformed into beneficial products. Waste materials usage in a cement-based composite may be a hopeful direction for resources management and waste decrement. Waste carpet fibers or polypropylene fibers were utilized in cement-based composites concrete since the past few decades [85]. By polypropylene fibers addition, some characteristics such as : impact resistance, permeability and abrasion can be considerably enhanced [86]. Mostly, reports show no or slight enhancement of toughness, flexural, tensile, compressive strength , and elastic modulus properties. But, in a few cases polypropylene fibers addition reduces the ultimate strength of hardened concrete [87-90].

Mohammadian et al. stated that by enhancing volume fraction of polypropylene fiber enhancement of flexural strength is observed for fiber reinforced pozzolanic mixture but decrease in compressive strength of mixtures may bound its usage in several structural applications, however fibers matrix reinforced has several enviable properties i.e. toughness resistance, lesser density and improved ductility. In fact the capacity for absorbing energy increases [91].

M.H Hossein et al. stated that by addition of 0.5 % polypropylene fiber content, it reduces the compressive strength by 8% and for this same volume fraction, it increases the flexural strength by 18.5% [92].

Figure 2.3: Polypropylene fibers (PP)

Pichor et al. analyzed that concrete with different volume fractions of FACs having Polyvinyl alcohol fibers (PVA) and Polypropylene (PP) fibers improve the flexural behavior and decrease density of resulting concrete and making it light weight [80].

Bang Yeon Lee et al. stated that Cementitious composites using PP fibers show high tensile ductility as compared to PVA or PE fiber based strain hardening cementitious composites (SHCC)and he also concluded that PP fiber based cementitious composites are extensively more ductile as compared to normal strength concrete with at least 300 times tensile strain capacity [94].

2.8.2 Polyethylene fibers Reinforcement (PE)

Polyethylene is common plastic utilize nowadays. From 2017, annual production of polyethylene resins is more than 100 million tons, which is 34% of entire plastics market. In 1898, Polyethylene was prepared accidently by German chemist Hans von Pechmann.

High performance (HP) polyethylene fibers (PE) have been used as reinforcement over the past 15 years. The poor PE fiber / matrix adhesion is quite often responsible for the slightly inferior performance of these composites.

PE fiber is well known by high strength, high modulus, wear resistance, light fastness and corrosion-resistance. It is suitable for making high-strength ropes, such as cables and machinery ropes. Polyethylene fibers have a low self-weight and are capable to float in water. PE has good gas blockade characteristics and better chemical resistance against greases, acids and oils. It can be very transparent and colorless but thick sections are generally solid and off white. Like other polymer fibers, they can only take tension and that’s why they are used exclusively for tensile applications like ropes and weaves. PE have found use applications in cut and puncture resistance, twines and fish nets, ropes, etc.

Figure 2.4: Polyethylene fiber (PE)

S.F.U Ahmed et al. inspected the multiple cracking and strain hardening behavior of hybrid fiber composites including diverse volume fractions of PE and ST fibres having 12 mm length are accounted. PE fibres have an impact on the tensile strain capacity of hybrid fiber composites. With increase of PE fiber contents, the ultimate tensile strain capacity is found to enhance at peak load and the strain capacity is decreased beyond a particular PE fiber content. When PE fibers length is enhanced by 1.5 times, considerable enhancement in multiple cracking and strain hardening behavior and strain capacity of hybrid fiber composites are noticed. Hybrid fiber composites tensile strain hardening and multiple cracking behavior is adversely effected by sand addition [93].

M.V.Deepthi et al. utilized fly ash Cenospheres as reinforcement filler in High density polyethylene (HDPE) to make up lightweight composites and concluded that it will cause the extensive enhancement to mechanical characteristics and thermal stability of composites [104].

2.8.3 Polyethylene teraphthalate Reinforcement (PET)

In 1941, James Tennant Dickson and John Rex Whinfield patented PET. In 2016, annual production of PET was anticipated to be 56 million tons [95]. Technically, as recycling of most thermoplastics are common. Recycling of PET bottle is more realistic than numerous plastic applications due to more resin content and approximately limited usage of PET for commonly used carbonated soft drink and water bottling.

Polyethylene terephthalate is main familiar thermoplastic polymer resin from family of polyester and used as containers for foods & liquids, fibers for clothing, and thermoforming for manufacturing. PET plastic bottles are extensively used for soft drinks. PET is used in waterproofing barrier in undersea cables.

Polyethylene terephthalate can exist as a semi-crystalline polymer or an amorphous which is transparent. Semi-crystalline material may appear transparent having less than 500 nm particle size or white and opaque having about a few micrometers particle size dependent on its crystal structure and size of particle.

Figure 2.5: Polyethylene terephthalate (PET)

Cong Lu et al. stated that PET-SHCC illustrated lesser strain capacity in tension as no stronger bond between fibers/matrix ratio and fiber dispersion matters and he also recommended a hybrid mixture of 1% by volume PET fibers and 1% by volume PVA fibers in which the PET fibers my minimize material cost and PVA fibers give tensile strength [82].

Xiuyi Lin et al. stated that PET fibers depicted excellent mechanical properties as compared to high performance PVA fibers [83].

Jing Yu et al. stated that when 50% PVA fibers has been substituted by 50% PET fibers, crack control ability of composites improved and cost of the material is reduced by 40 percent [96].

2.9 Problems Associated with Fibers Reinforcement

Use of fibers has some serious issues which in spite of good mechanical, electrical and thermal properties have limited infield application of these fibers. It was noticed that by escalating number of fibers, the flow ability of mix in fresh state was reduced. .Systematic uniform dispersion of fibers was also required to get desired improvement in mechanical properties of cementitious composites, but these were noted to be uneven distribution of fibers within the cementitious composite due to entanglement problem in this material. Flexural strength of resulting composites is also effected by the contact point below loading in flexural strength. Uneven distribution of fibers affected the consistency and statistics of development get in different time periods by different researchers [77].

Length of fibers and increasing aspect ratio also diminish the flow. Xiuyi Lin et al.used PET fibers in his research and he concluded that fibers having smaller diameter and larger aspect ratio is useful in crack bridging but results in poor dispersion of fibers and poor workability and lower alkali resistance [83].

2.10 Investigational Techniques

Researchers have used various investigational techniques to approach different physical and chemical properties of self-compacting cementitious systems. A brief description of these is given below;

2.10.1 Workability

Workability of concrete, which is the characteristics of freshly mixed concrete, may find out the easiness of placement and homogeneity of mixing, consolidated, placed, and finished. It is determined through slump cone test using Hagerman's cone. The procedure is explained in depth in Chapter 3.

2.10.2 Scanning Electron Microscopy (SEM)

Use of the SEM in petrographic analysis of concrete microstructure and cementitious materials is becoming more and more familiar. SEM is a technique used for imaging the microstructure of concrete at very large magnifications and high resolutions less than 1 micrometer. This is a very helpful tool to collect quantitative information such as composition, phase abundance and distribution, its porosity and the connectivity of pores may be extracted from these data. Occurrence of diverse phases of cement and the products of hydration can also be detected with the assistance of scanning electron microscopy. By using SEM, the interfacial transition zone (ITZ) may be covered.

To comprehend the physical properties of the raw materials and the hydration products

Such information can help us. This will help to better understanding of the progress rate of hydration kinetics, mechanics of water absorption and pore development, and strength development. Hence, the compatibility of various materials can be assessed.

Significant information about progress of hydration, its products and their packing density can also be yield by SEM analysis. Hanif et al. studied these parameters and reported that a more permeable microstructure can be observed with the addition of FACS in the cementitious composites. Further, smaller particles shell may devour itself, because of pozzolanic activity of FACs, escalating the calcium silicate hydrate (CSH) gel volume and voids remaining after because of hollow nature of FACs [43,97].

2.10.3 X-Ray Diffraction (XRD)

X-ray powder diffraction (XRD) is a quick analytical technique mainly utilized for detection and detection of a crystalline material and may give information on unit cell magnitude. When applied to amorphous solids and liquids It also yields fundamental data. To assess the reactivity of the materials, the isolation of crystalline and amorphous materials is important in cement based formulations. Elements are strike at specified atomic distances in crystalline materials and so cannot take part in chemical reaction. But for cement hydration, the basic requirement is to be reactive. The amorphous substances fulfill this standard and hence are chosen for cement formulations.

To identify the various amorphous and crystalline structures in any material, XRD is a useful technique. Hanif et al. performed XRD of his powder Fly ash cenosphere samples and reported that alumina is the main mineral present in FACs. He further reported that Amorphous silica minerals in diverse forms like quartz and cristobalite have also been recognized in the patterns. The existence of amorphous silica and a minute quantity of lime can be useful in pozzolanic reactions in the cement-based system. Moreover, alumina and mullite are also observed [43,41,74, 98].

Hanif et al. performed of his hardened samples and reported that during the pozzolanic reactions CH is consumed, in the samples containing FACs the lesser quantity of free CH in the dried pastes specifies the pozzolanic reactivity of FACs. Though, the extent of pozzolanic reactivity is dependent on FACs particle size. Finer particles were likely to be greater reactive whereas the larger size particles might stay behind partial active or inert [43].

2.10.4 X-Ray Fluorescence (XRF)

XRF is a technique used to achieve chemical analysis of a material. It is important to carry out the chemical analysis by means of this can give us information about the chemicals present in the raw materials and their most likely products. Basically, specific raw materials should yield their characteristic products during hydration. The deviations from trends can be examined to find out the essential reasons.

This is also done to check suitability of the materials to be used in the research work. Satisfying standards makes the research work more worldwide applicable and easy pliable.

CHAPTER 3: EXPERIMENTAL PROGRAM

This chapter discusses the materials utilized and the procedures adopted to carry out the experiments.

3.1 Materials /Storage of Materials

The study was made on self-compacting mortar systems (SCMs) [5] with a target flow of 30+1cm [99] and cement: sand ratio is equal to 1:2. The replacement level of the SRM selected was different by weight of cement as mentioned widely in literature for optimized properties [40,42,43,77, 81].

In this study, all the materials utilized were kept at room temperature. Before carrying out the practical work, this was ensured by storing the materials in lab at least 24 hours.

3.1.1 Cement Type

Ordinary Portland Cement (OPC), grade 53, type I conforming to ASTM standard C150-04[100] was used. It is manufactured in Pakistan and is locally available in Islamabad. Physical properties and chemical composition of cement are summarized in Table 3.1 and 3.2.

Table 3.1: Chemical Composition of cement

Oxides	CaO	Al2O3	SiO2	Fe2O3	MgO	SO3	K2O
Content (wt. %age of cement)	63.33	4.88	20.38	3.26	2.41	2.70	0.58

Table 3.2: Mechanical and physical properties of cement

Color	Density (g/cm3)	Specific Surface Area (cm2/g)	IR (%)	LOI (%)	Compressive Strength (MPa)
Light grey	3.16	322	1.29	3.83	63.91

3.1.2 Fine Aggregate

Fine aggregate used in the present study was Lawrencepur sand. The size of sand was<2mm, For this purpose the sand was first sieved through ASTM sieve no. 10 (ASTM C807) using a mechanical shaker. The passing sand was get together and oven dried at 100°C for 24 hours. Then for one day, it was cooled at room temperature. To avoid contact with moisture, this sand was stored in dry air-tight container. To ensure better packing of the formulations, Well-graded sand was used to get better micro-structure and improved durability. Fineness Modulus of the sand is 2.09.

3.1.3 Super Plasticizer (SP)

Chemrite AG -300 is a whitish pale liquid type third generation high-performance super-plasticizer used in cement based mixes. For current research, chemrite AG -300 manufactured by Imporient chemicals (PVT) limited was used. It is based on carboxylic acid derivatives liquid, optimized for use as a plasticizer and water reducing agent and ensures development of high early strength. It helps in prevention of bleeding and segregation, when used in apposite proportions. The use of this type of super plasticizer improved shrinkage and creep characteristics. Its basic properties are listed below in Table 3.3.

Table 3.3: Properties of Super plasticizer (SP)

Appearance	Liquid
Color	Whitish pale
Density, [kg/lit] at 20ºC	1.06
pH value at 20ºC	7

3.1.4 Water

First of all, the Water Demand for each system has been worked out cautiously. Then, to make sure complete hydration and to allow minimum porosity due to excess water, the water to powder ratio for each formulation was kept equal to water demand for the system.

3.1.5 Fly ash cenospheres

Fly ash cenospheres found in china, obtained from Zhen Yang, Hebei China were used in this research. They are attained during coal combustion process in coal combustion power plants Physical properties of FACs are given in Table 3.4.

Table 3.4: Physical characteristics of FACs filler

Description	Surface Area (m2/g)	Average Particle Size (µm)	Color	Bulk Density (kg/m3)	iso-static crushing strength (MPa)
Fly ash cenospheres	6.02	180	Grey	720	70-140

Table 3.5: chemical characteristics of FACs filler

Description	CaO	Fe_2O_3	MnO	SO_4	K_2O	SiO_2	Na_2O	Al_2O_3	TiO_2
Fly ash cenospheres	1.06	1.96	0.05	0.42	3.94	73.10	2.42	16.70	0.35

3.1.6 Polypropylene fibers (PP)

Physical properties of Polypropylene fibers [108] are given in table 3.6.

Table 3.6 : Properties of PP fibers

Length (mm)	Diameter(µm)	Tensile strength (MPa)	Elastic modulus (GPa)	Young's modulus (MPa)
6mm ± 0.5 mm	>32	315	5	37

3.1.7 Polyethylene fibers (PE)

Physical properties of Polyethylene fibers [107] are given in table 3.6

Table 3.7: Physical properties of the PE fiber

Length (mm)	Diameter(µm)	Tensile strength (MPa)	Elastic modulus (GPa)	Density $(\frac{g}{cm^3})$
12	24	3000	120	0.97

3.1.8 Polyethylene teraphthalate fibers (PET)

Physical properties of Polyethylene teraphthalate fibers [82] are given in table 3.6.

Table 3.8: Physical properties of the PET fiber

Length (mm)	Diameter(µm)	Tensile strength (MPa)	Elastic modulus (GPa)	Density $(\frac{g}{cm^3})$
12	38	1095	10.7	1.37

3.1.9: Characterization of FAC

Scanning Electron Microscopy (SEM) was implemented to detect the particle size, surface texture, morphology and microstructure of FACs. Scanning Electron Microscope TESCAN VEGA3 was used for this purpose.

Figure 3.1: Scanning Electron Microscope

X-ray diffraction (XRD) analysis was executed to explore physical properties like composition and structure of carbonized materials. K-alpha-1 x-ray diffractometer (Bruker D8 Advance) with high-flux deflection radiations was used in the ‘2θ’ diffraction range of 10-80^0.

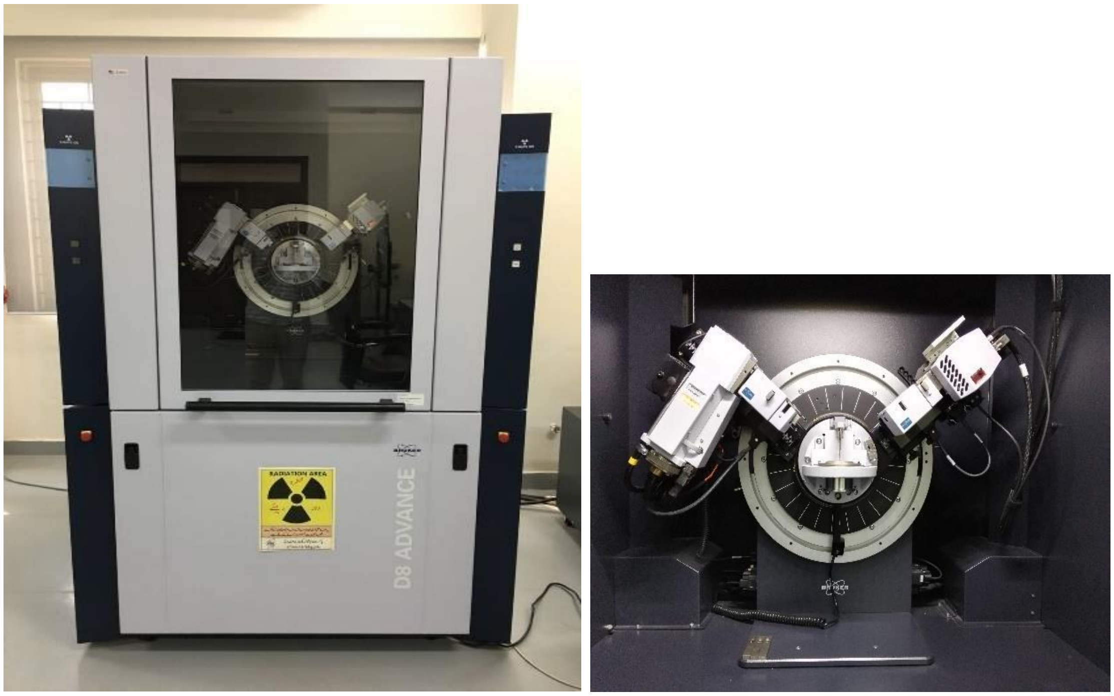

Figure 3.2: X-ray diffractometer.

Laser granulometry (LG) of FACs was performed using HORIBA Laser Scattering Particle Size Distribution Analyzer LA-920 (Fig.3.3)

Figure 3.3: Particle size distribution analyzer

X-Ray Fluorescence (XRF) is a powerful technique to study the elemental or chemical analysis of any material. It uses X-rays (or gamma rays) to chemically decompose and find out the ingredients of any substance. The process includes making palettes of the sample using a Hydraulic Press. Hydraulic press is a simple mechanism that exploits air pressure to densify the materials. The palettes made are then put into the Element Analyzer to find out the constituents. The typical output is a computer generated report, specifying the components along with their percentages.

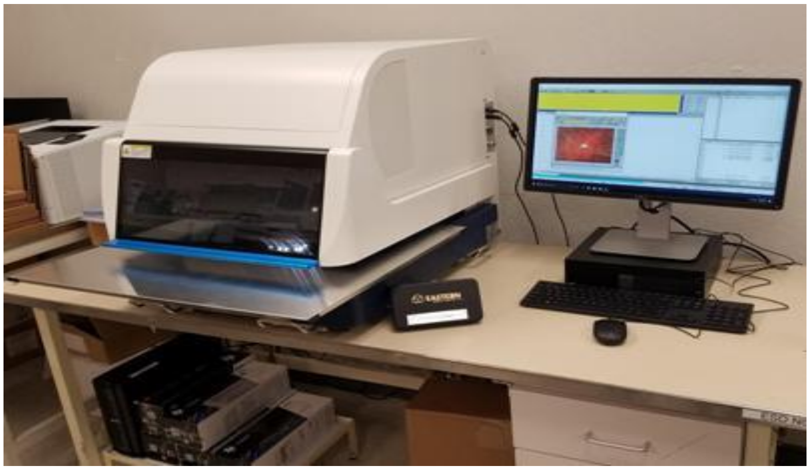

Figure 3.4: X-Ray Fluorescence (XRF)

3.2 Methodology

Experimental procedures followed for preparation and testing of Fly ash cenospheres reinforced cementitious composites are discussed in this section. Cementitious mortar (CM) was reinforced with different types of fibers for this study.

3.2.1 Mix Proportions

The designed experimental investigation consisted of six different SCM formulations. Three being the control mix with three different types of fibers i-e Polypropylene fibers (PP), Polyethylene fibers (PE) and Polyethylene teraphthalate fibers (PET). While the three others having fly ash cenospheres with three different types of fibers i-e Polypropylene fibers (PP), Polyethylene fibers (PE) and Polyethylene teraphthalate fibers (PET) . All the formulations used equal replacement percentage of fly ash cenospheres i.e. 40%. The detailed mix proportions of various SCM formulations used are given in table 3.9.

Cement to sand ratio was selected as 1:2 Water to cement ratio (w/c) was fixed as 0.4 while the quantity of water absorbed by FACs was compensated in the mix proportioning. SP dosage of 1.0% was sustained for the entire designed formulations to attain the sufficient flow-ability.

Table 3.9: Detailed Mix proportions

Formulation Number	Mortar Formulation	w/c	Cement (g)	Super Plasticizer	PP fibers	PE fibers	PET fibers
1	CM (F0)	0.4	1.0	1%	1%	-	-
2	FAC 0.4	0.4	0.60	1%	1%	-	-
3	CM (F0)	0.4	1.0	1%	-	1%	-
4	FAC 0.4	0.4	0.60	1%	-	1%	-
5	CM (F0)	0.4	1.0	1%	-	-	1%
6	FAC 0.4	0.4	0.60	1%	-	-	1%

3.6.3 Water Demand, Super Plasticizer Demand and Setting Times

In the experimental process, the first step included determination of the water demand (WD) and setting times of diverse mortar formulations, by means of Vicat Apparatus. Firstly, WD of mortar systems was determined as a sum of the water demand of paste in accordance with ASTM standards of C187, C127 and C128 respectively. Later, setting times were worked out in accordance with ASTM C807 Standards.

Chemrite AG-300 is a whitish pale liquid type third generation high-performance super-plasticizer used in cement based mixes to enhance the workability of system. Trial and Error approach was employed to determine the SP demand of the formulations to reach the target flow of 30+1 cm, using Hagerman’s Mini Slump Cone Apparatus.

All formulations had mixing water equal to water demand (WD) of the respective formulation. This was done to ensure complete hydration without compromising on strength by allowing porosity through excess water. The Super Plasticizer (SP) content used was as requisite to get the target flow of 30+1 cm for each formulation.

3.3 Sample Preparation

To minimize loss of moisture, moulds casted for strength tests were covered with polythene sheet. Then, they were de-moulded and weighed after 24 hours, and kept in the curing tank till the specified maturity was achieved. The samples were weighed again in SSD condition before strength testing was carried out.

3.4 Formulation type

Six SCP formulation were prepared at their respective super plasticizer demands for the target flow of 30 ± 1 cm measured by Hagerman's cone measuring 6 x 7 x 10 cm3 and constant water content i.e. 40 % by weight of total cement. The formulations tested were as follows:

1. CM-SP1-PE1-W0.40
2. FAC0.4-SP1-PE1-W0.40
3. CM-SP1-PP1-W0.40
4. FAC0.4-SP1-PP1-W0.40
5. CM-SP1-PET1-W0.40
6. FAC0.4-SP1-PET1-W0.40

A typical formulation of CM-SP1-PE1-W0.40 should be considered as having CEM 1 type cement, 1 % Super-plasticizer (SP) dose needed for target flow, 1 % PE type fiber and 40 % mixing water and FAC0.4-SP1-PE1-W0.40 as having fly ash 40% and 60% cement content 1 % Super-plasticizer (SP) dose required for target flow, 1 % PE type fiber and 40 % mixing water content at 25 ºC and 55 % relative humidity (RH) in the lab. All additional formulations may be implicated in view of that.

3.5 Preparation Scheme

The preparation of mixing of the mortar systems was done in Hobart Mixer (Fig.3.5) of 5 liters capacity. For perfect values of flow and SP quantity, the mixing regime was selected up from literature [102]. Mixing water was added in two sections, 50% by weight in two times, to boost up action of the super plasticizer (SP). First of all, the powders have been dry mixed for one minute with the addition of 50% water although constantly mixing for additional 30 sec. Then Super plasticizer and the remaining 50% water were added with the continuous mixing for another 30 sec. After that, the three different types of fibers were slowly dispersed in the mix one by one and the mixing procedure continued for 3 to 4 minutes until the even consistent mix achieved. Initially, the fibers were mixed at normal agitator speed and after that at high speed. Hence, the whole mixing procedure received a total mixing time of 6 to 8 minutes for each mix. Same procedure was followed for CM specimen's preparation of all batches. The detailed mixing regime is summarized in Table 3.10 as follows.

Figure 3.5: Hobart mixer

Table 3.10: Analyzed Mixing Regime

Mixing Time	Mixing speed	Constituents added
Slow Speed i.e. 145 rpm	00-60 sec (1.0 min)	Dry mixing containing Cement, Sand and SRM (quantities as per the mix design)
Slow Speed i.e. 145 rpm	60-90 sec (0.5 min)	Addition of 50% water
Fast Speed i.e. 285 rpm	90-120 sec (0.5 min)	Addition of 50% water and SP
Fast Speed i.e. 285 rpm	120-300 sec (3.0 min)	Addition of fibers

3.6 Investigational Techniques and Methodology

To determine the microstructure and mineralogical phases of FACs reinforced cementitious composites, SEM and EDX were performed using Scanning Electron Microscope TESCAN VEGA3 After compression test, small samples of less than 0.5cm x 0.5cm size were collected for these tests. By immersing these samples in acetone for a period of 24hrs, further hydration was stopped. To remove the traces of acetone, these were completely covered in isopropanol [103]. Samples were preheated for 24hrs at 100oC for SEM and EDX analysis.

Average density of controlled and FACs reinforced specimens were calculated to examine the effect of FACs addition on microstructure of cementitious composites.

3.7 Characterization Scheme for Enhanced Performance of Cementitious Composites

To evaluate improvement in performance of FACs reinforced cementitious composites; microstructure and fracture properties of cementitious composites were investigated. Followings tests were performed for this purpose.

3.7.1 Characterizing Cementitious Composites Behavior in Fracture

Total 18 prisms of 40mm x 40mm x 160mm size were tested in flexure. Three-point bending tests [110,111] were executed with Universal Testing Machine SHIMADZU of 20KN capacity at a very sensitive strain rate of 0.15mm/min and with a clear span of 120mm (Figure 3.6).

Figure 3.6: Universal testing machine

Compression Testing Machine CONTROLS MCC-08 was used for characterization of cementitious composites behavior in compression (Figure 3.6) at a loading rate of 1KN/sec in load-controlled mode. Broken halves of prisms which were left after three-point bending tests were used for compression test. The test was executed on total 54 samples, and it was performed according to ASTM C109 [101].

Figure 3.7: Compression testing machine

CHAPTER 4: RESULTS AND DISCUSSION

General

This chapter will present the results attained from the experimental program explained in the previous chapter. Characterization of FACs powder was performed by laser granulometry (LG), scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis, Brunauer-Emmet-Teller (BET) analysis, and X-ray Fluorescence spectroscopy (XRF).

4.1 Laser Granulometry

The particle size distribution (PSD) of FACs powder was found by Laser Granulometry. LG curve of FACs was plotted. It is concluded that a considerable fraction of particles lie in the range of 50 to 400 mm while approximately 5% particles are bigger than 400 mm.

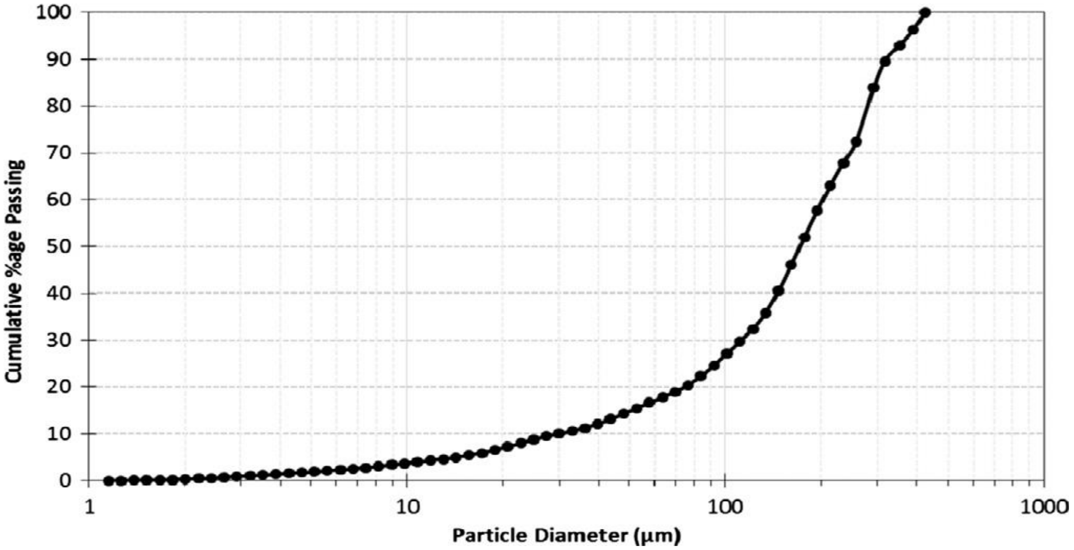

Figure 4.1: Particle Size Distribution of Powdered FACs

4.2 Scanning Electron Microscopy (SEM) of FACs

SEM was performed to identify the true particle size, morphology, surface texture and microstructure of FACs; along with EDX spectroscopy to obtain qualitative chemical composition.

Hollow spherical shape of FACs particles (Fig.4.2) with smooth glassy texture were observed. It was observed that these are hollow from inner side and well-rounded with a shell thickness of numerous microns. The size vary in diverse batches and has been found as 1–100 mm , 1–400, 1–300 mm and 1–600 mm . Due to the plentiful variety in particles size and shape, it could be suggested that these would give good filling in voids from capillary pores to gel pores. Hence retain prospective to develop the better microstructure and improve the fracture properties of cementitious composites.

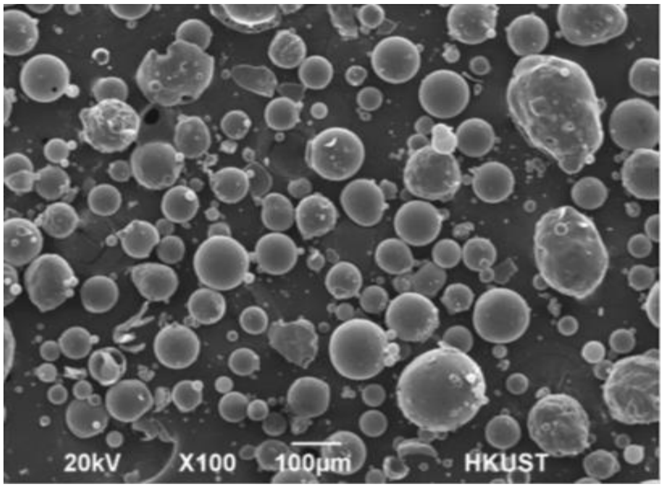

Figure 4.2: SEM micrograph of FACs

4.3 Crystallography of FACs by X-Ray Diffraction (XRD)

The phase-mineral composition of FAC developed by X-ray diffraction (XRD) can be seen in Fig. 4.3. Amorphous silica minerals in various forms like cristobalite and quartz have been recognized in patterns. More, mullite and alumina are too observed where alumina is a main mineral existing in FACs [81].

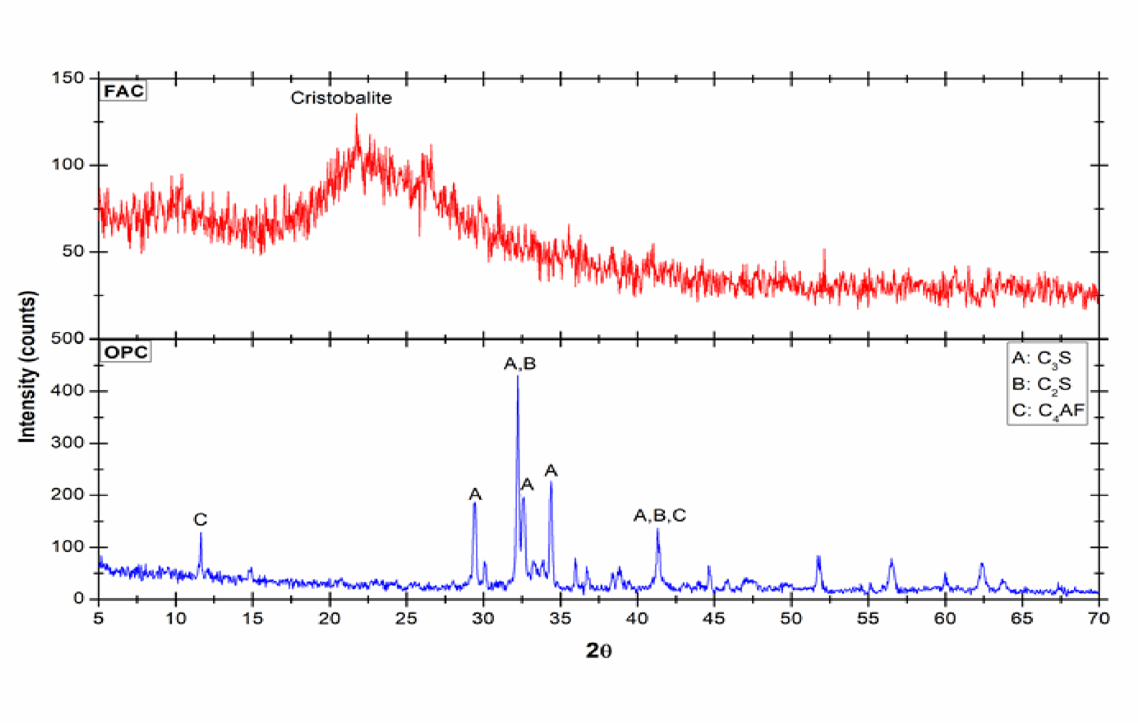

Figure 4.3: X-ray diffractograms of FACs

4.4 X-ray Fluorescence spectroscopy (XRF) of FACs

The chemical components of FSCs evaluated by X-ray Fluorescence spectrometer (XRF,JSX-320IZ) and results showed that approximately 90% portion included silicon dioxide and aluminium oxide while calcium oxides and other oxides are also present in small amount.

Table 4.1: X-ray Fluorescence spectroscopy (XRF)

Description	CaO	Fe_2O_3	MnO	SO_4	K_2O	SiO_2	Na_2O	Al_2O_3	TiO_2
Fly ash cenospheres	1.06	1.96	0.05	0.42	3.94	73.10	2.42	16.70	0.35

4.5 Flow Measurements

The flow of SCP systems integrated FACs was found by Hagerman's mini-slump cone of 6 x 7 x 10 cm3. The target flow was 30 ± 1 cm. The super-plasticizer requirement for all formulation was kept same. So, 1% SP added in this study for all formulations.

4.6 SEM and EDS of Hardened Samples

Scanning Electron Microscopy (SEM) along with Energy Dispersive Spectroscopy (EDS) analysis was executed again on the hardened samples to study the micro-structure. The imaging performed on the selected 28 day hardened samples.

Fig.4.4 evaluates the SEM images of the six pastes with and without incorporation of FACs. It can be evidently observed in the image that a few of the FACs particle were inert whereas several particles indicate partial reactivity due to the slender spherical shell was wrecked because of the pozzolanic reaction in few cases. More, it has as well been observed that enhancement of FACs fraction in composite causes improved porosity of composite. The pores were slightly non-uniform and irregular shaped and their irregularity was because of partial reactivity of FAC. Likewise, such as the particle size increases, the particle shape deviates from the perfect spherical shape. At higher size range very asymmetrical and irregular porous 'sponge-like" particles appear [106]. Besides, the specimen having higher weight fractions of FACs, agglomeration of FACs particles has also been observed in some areas.

EDX spectroscopy results are summarized in Table 4.2. Carbon content was found 14 by weigh and Oxygen content was found more than 50 % by weight in CM with PET fibers. Traces of Si, S, Ca, Ni, O and Al were also detected.

EDX spectroscopy results are summarized in Table 4.3. Oxygen content was found more than 50% by weight in CM with PET fibers. Traces of Si, Mg, O, S, Al, Ti and Ca were also detected.

EDX spectroscopy results are summarized in Table 4.4. Carbon content was found more than 90% by weight in CM with Polypropylene fibers. Traces of Si, O and Ca were also detected.

EDX spectroscopy results are summarized in Table 4.5. oxygen content was found more than 50% by weight in FACs with Polypropylene fibers. Traces of Si, K, Mg, Mn, C, S, Al, Fe, Ti and Ca were also detected.

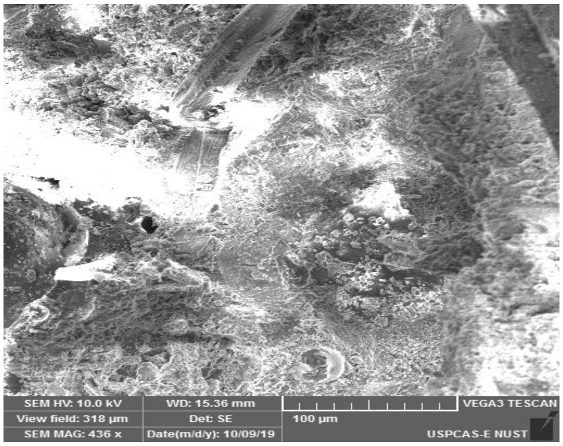

Fig.4.4 (a):CM Sample with PET fiber

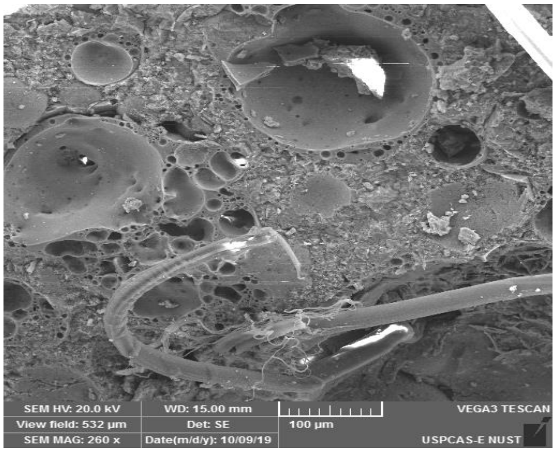

Fig.4.4(b):FACs incorporating Sample with PET fiber

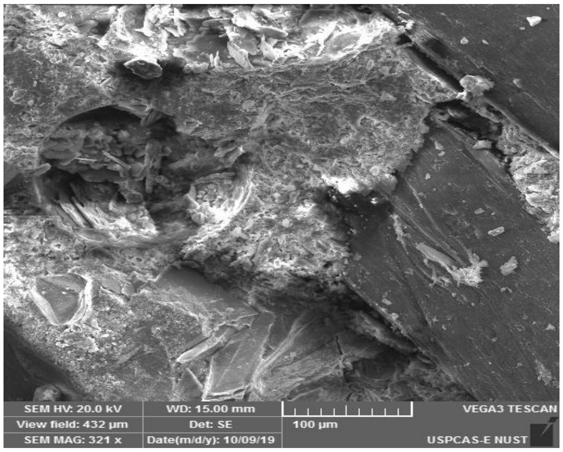

Fig.4.4 (c): CM Sample with PP fiber

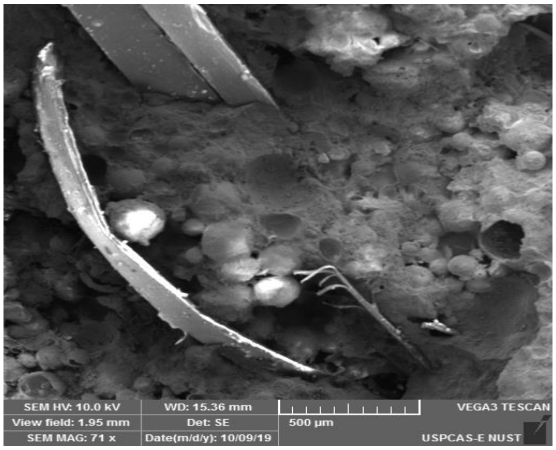

Fig.4.4 (d): FACs incorporating Sample with PP fiber

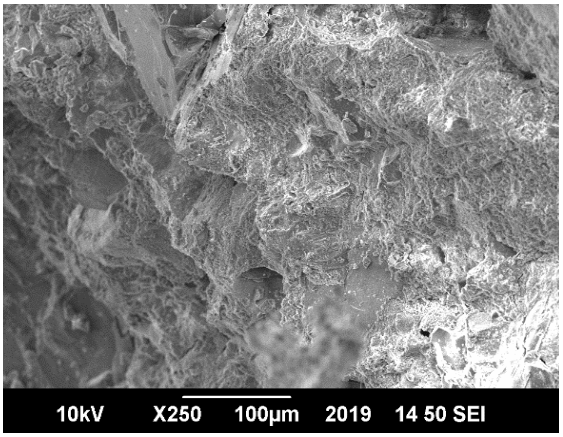

Fig.4.4 (e):CM Sample with PET fiber

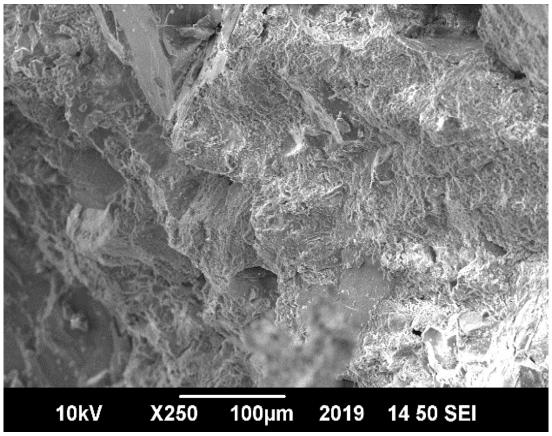

Fig. 4.4 (f):FACs incorporating Sample with PET fiber

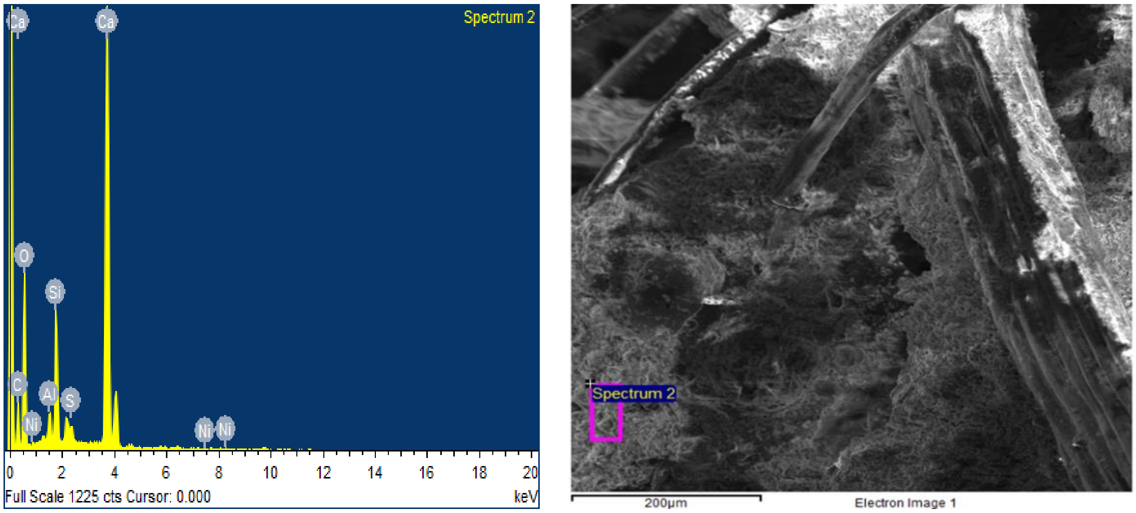

Figure 4.5: EDS spectra of CM with PET fibers

EDX spectroscopy results are summarized in Table 4.2. Carbon content was found 14 by weigh and Oxygen content was found more than 50 % by weight in CM with PET fibers. Traces of Si, S, Ca, Ni, O and Al were also detected (Figure 4.5).

Table 4.2: EDS spectra of CM specimen with PET fiber.

Element	**Weight%**	**Atomic%**
C K	14.92	22.15
O K	58.34	65.03
Al K	0.83	0.55
Si K	3.63	2.30
S K	0.57	0.32
Ca K	21.62	9.62
Ni K	0.09	0.03
Totals	100.00	

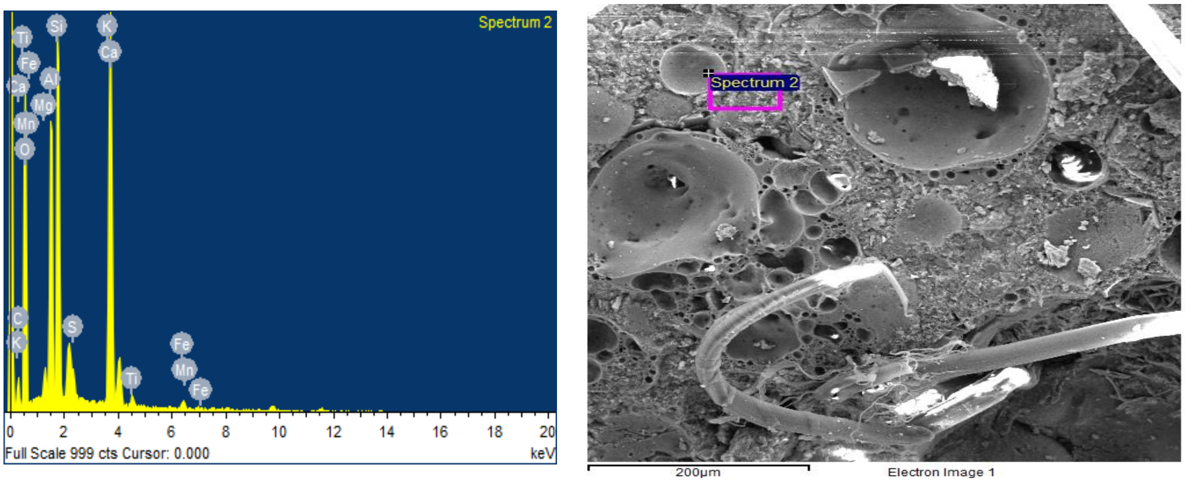

Figure 4.6: EDS spectra of FACs with PET fibers

EDX spectroscopy results are summarized in Table 4.3. Oxygen content was found more than 50% by weight in CM with PET fibers. Traces of Si, Mg, O, S, Al, Ti and Ca were also detected (Figure 4.6).

Table 4.3: EDS spectra of FACs specimen with PET fiber

Element	**Weight%**	**Atomic%**
C K	6.66	10.31
O K	61.79	71.80
Mg K	0.69	0.53
Al K	6.15	4.23
Si K	8.66	5.73
S K	0.76	0.44
Ca K	13.93	6.46
Ti K	0.39	0.15
Fe K	0.88	0.26
Totals	100.00	

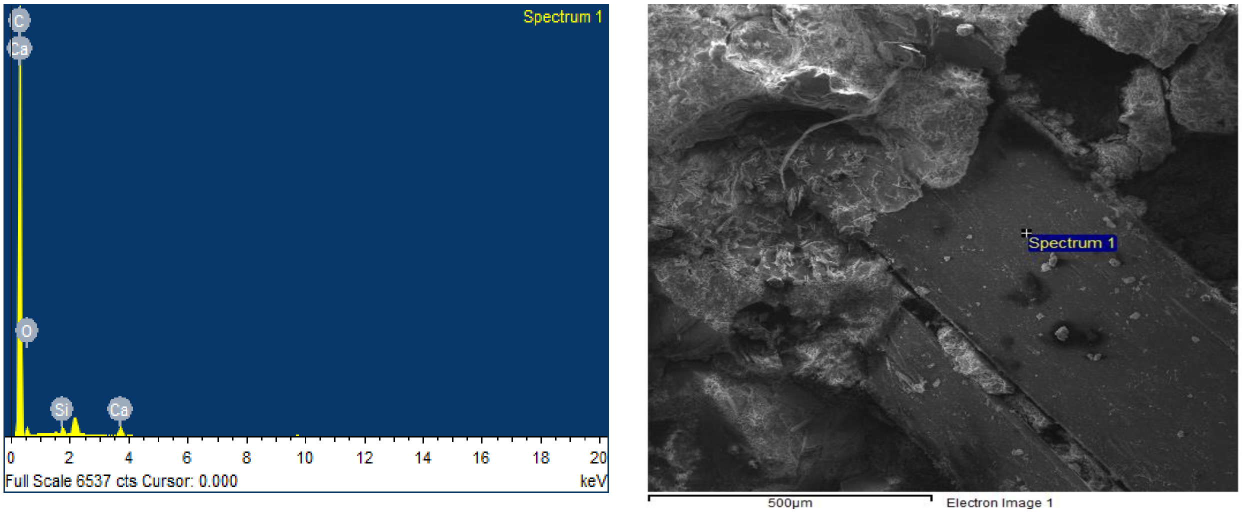

Figure 4.7: EDS spectra of CM with PP fibers

EDX spectroscopy results are summarized in Table 4.4. Carbon content was found more than 90% by weight in CM with Polypropylene fibers. Traces of Si, O and Ca were also detected (Figure 4.7).

Table 4.4: EDS spectra of CM specimen with PP fiber

Element	**Weight%**	**Atomic%**
C K	92.35	94.49
O K	6.80	5.22
Si K	0.21	0.09
Ca K	0.65	0.20
Totals	100.00	

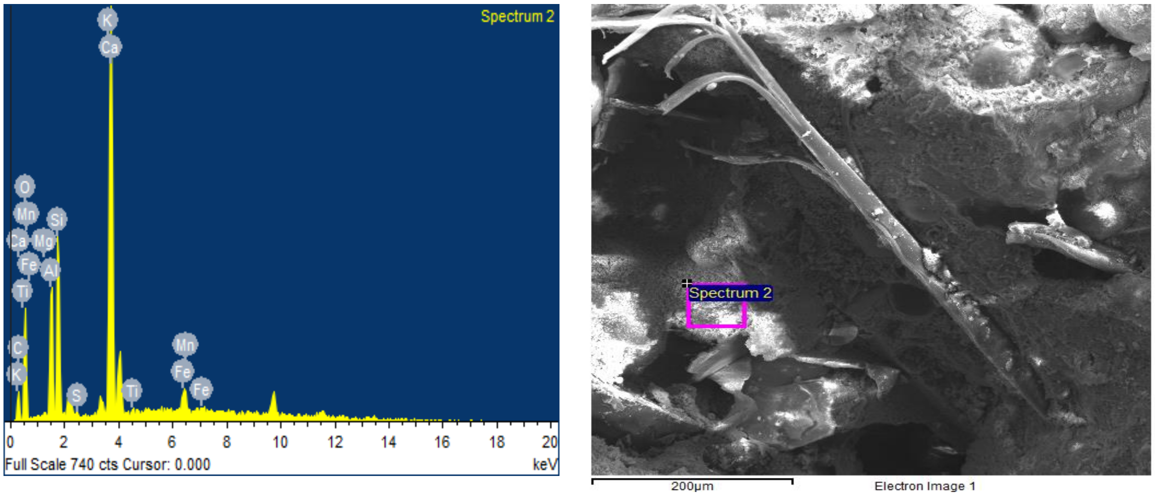

Figure 4.8: EDS spectra of FACs with PP fibers

EDX spectroscopy results are summarized in Table 4.5. oxygen content was found more than 50% by weight in FACs with Polypropylene fibers. Traces of Si, K, Mg, Mn, C, S, Al, Fe, Ti and Ca were also detected.

Table 4.5: EDS spectra of FACs specimen with PET fiber

Element	**Weight%**	**Atomic%**
C K	9.69	15.95
O K	50.33	62.20
Mg K	0.19	0.15
Al K	4.72	3.46
Si K	6.67	4.70
S K	0.04	0.02
K K	0.71	0.36
Ca K	24.12	11.89
Ti K	0.28	0.12
Mn K	0.17	0.06
Fe K	3.08	1.09
Totals	100.00	

Chapter 5: Fracture Properties

Fracture properties of FACs cementitious composites with various kinds of fibers reinforcement and CM based fiber reinforcement and mechanism involved in modification/intensification of these properties on fibers intrusion is discussed in this chapter.

5.1 Fracture Properties

Fracture properties of fibers reinforced CM specimens and FACs cementitious composites with fiber reinforcement were evaluated in terms of resistance in flexure and compression.

5.1.1 CM and FAC Specimens Performance in Flexure

Three-point bending tests were executed with strain controlled UTM at a very sensitive controlled-strain rate of 0.15mm/min. Flexural performance was found by means of flexural strength, rupture strain in flexure and toughness index.

5.1.1.1 Load-Displacement Curves

Load-displacement curves of CM specimens with and without addition of FACs were plotted (Figure 5.1). Effect of Fibers and FACs on flexural performance was observed from these plots.
In case of PE fibers, Pre-Cracking response of FACs based specimens increased as compared to CM specimens. Post cracking response of FACs specimens also increases as compared to CM specimens and Strain hardening behavior is also improved of FACs samples as compared to CM specimens (Fig. 5.1).

In case of PP fibers, there is no improvement in Pre-Cracking response of FACs based specimens as compared to CM specimens. There is also improvement in post-cracking response of FACs based specimens as compared to CM specimens and there is no difference between the strain hardening behavior of both specimens (Fig. 5.2).

In case of PET fibers, there is no improvement in pre-cracking response of FACs based specimens as compared to CM specimens. There is little improvement in post-cracking response of FACs based specimens as compared to CM specimens and there is little improvement between the strain hardening behavior of both specimen (Fig. 5.3).

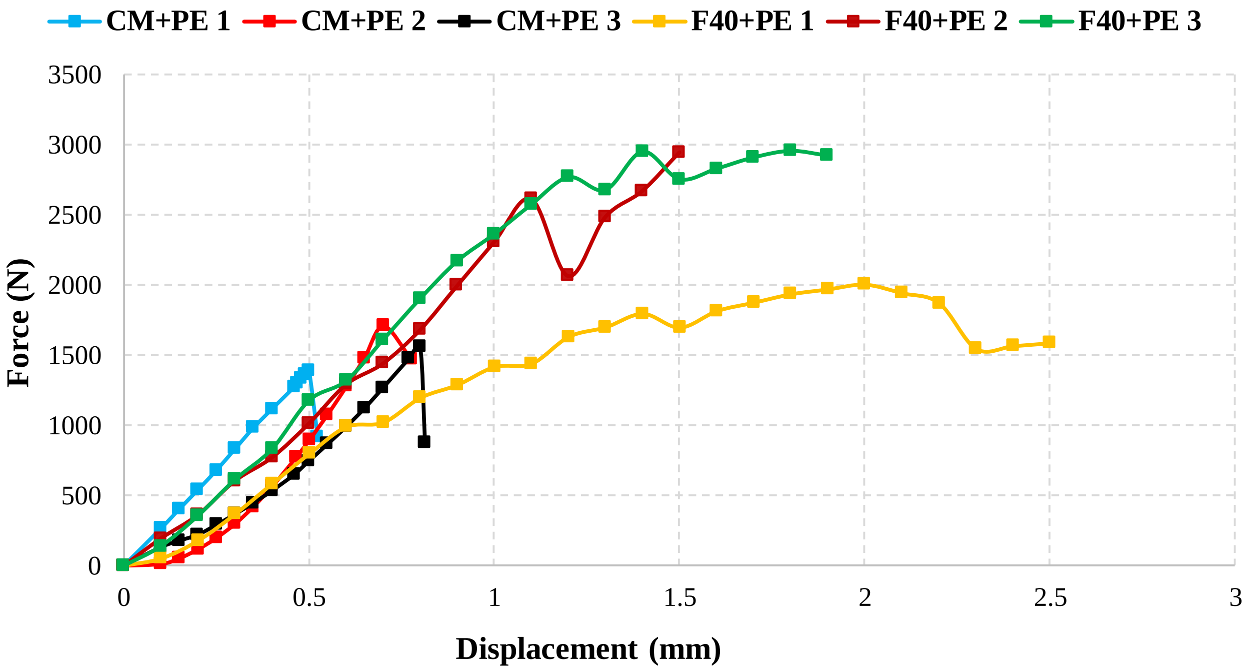

Figure 5.1: Load-displacement curves of CM and FACs specimens reinforced with PE fibers.

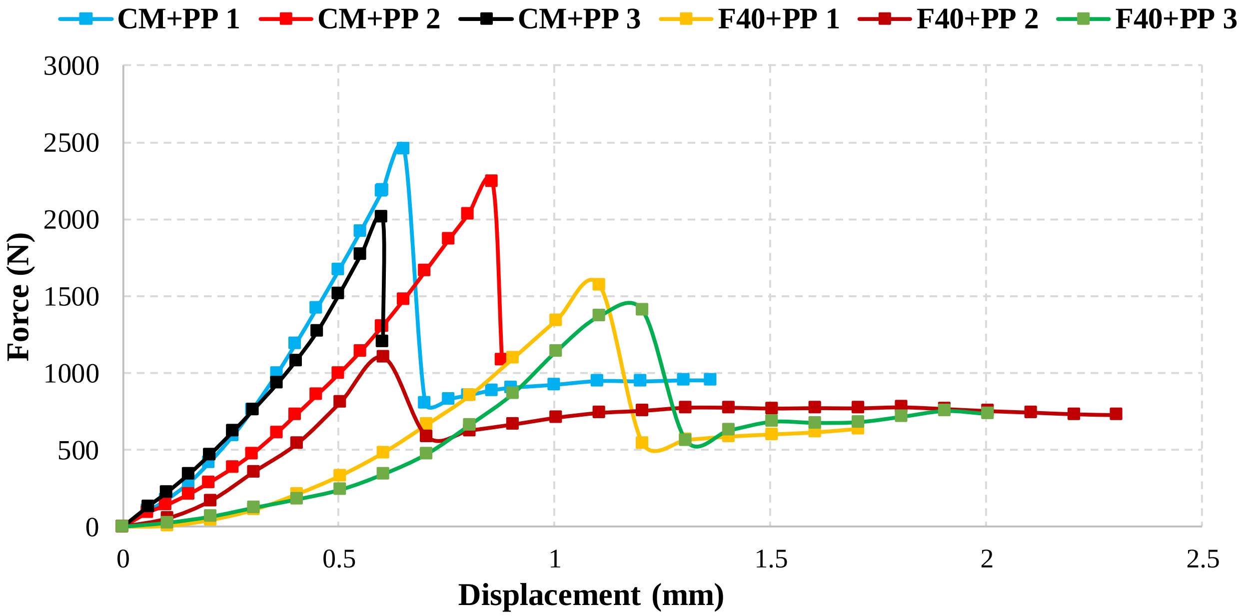

Figure 5.2: Load-displacement curves of CM and FACs specimens reinforced with PP fibers.

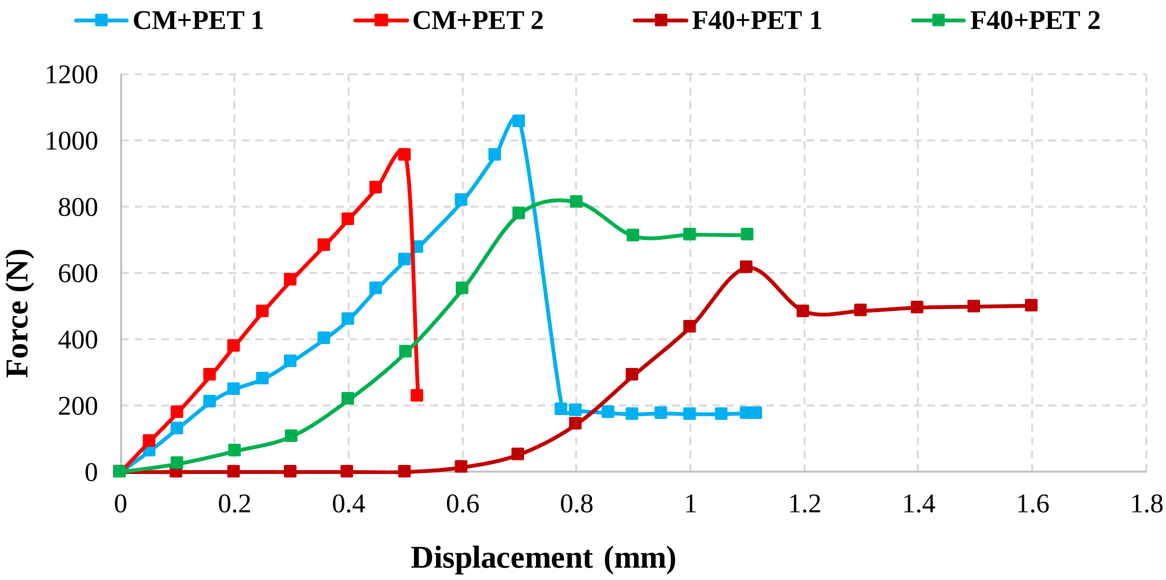

Figure 5.3: Load-displacement curves of CM and FACs specimens reinforced with PET fibers.

5.1.1.2 Stress-Strain curves

The flexural behavior of CM and FACs based specimens with PE fiber is depicted in Fig. (5.4).Although the low fiber content has led to the higher flexural strength, still the strain hardening is much pronounced in case of PE fibers for FACs based specimens but for the CM based specimens, it can be seen that there is a drop in the corresponding curve, due to the matrix fracture, after which the specimen still sustained some loading until the point of ultimate failure. Most of the specimen yielded less than 1.5% of strain at failure. The flexural strain capacity (flexural strain at peak flexural stress) was found increasing with the addition of FAC.

The flexural behavior of CM and FACs based specimens with PP fiber is depicted in Fig. (5.5). The strain hardening is not much definite in case of PP fibers for FACs based specimens but for the CM based specimens, it can be seen that there is a drop in the corresponding curve, due to the matrix fracture, after which the specimen still sustained some loading until the point of ultimate failure. Most of the specimen yielded less than 1.5% of strain at failure. The flexural strain capacity (flexural strain at peak flexural stress) was found little increasing with the addition of FAC.

With PET fiber, the flexural behavior of CM and FACs based specimens is showed in Fig. (5.6).In case of PET fibers for FACs based specimens, The strain hardening is little manifest but for the CM based specimens, it can be seen that there is a drop in the corresponding curve, due to the matrix fracture, after which the specimen still sustained some loading until the point of ultimate failure. Most of the specimen yielded less than 1.5% of strain at failure. The flexural strain capacity (flexural strain at peak flexural stress) was found little increasing with the addition of FAC.

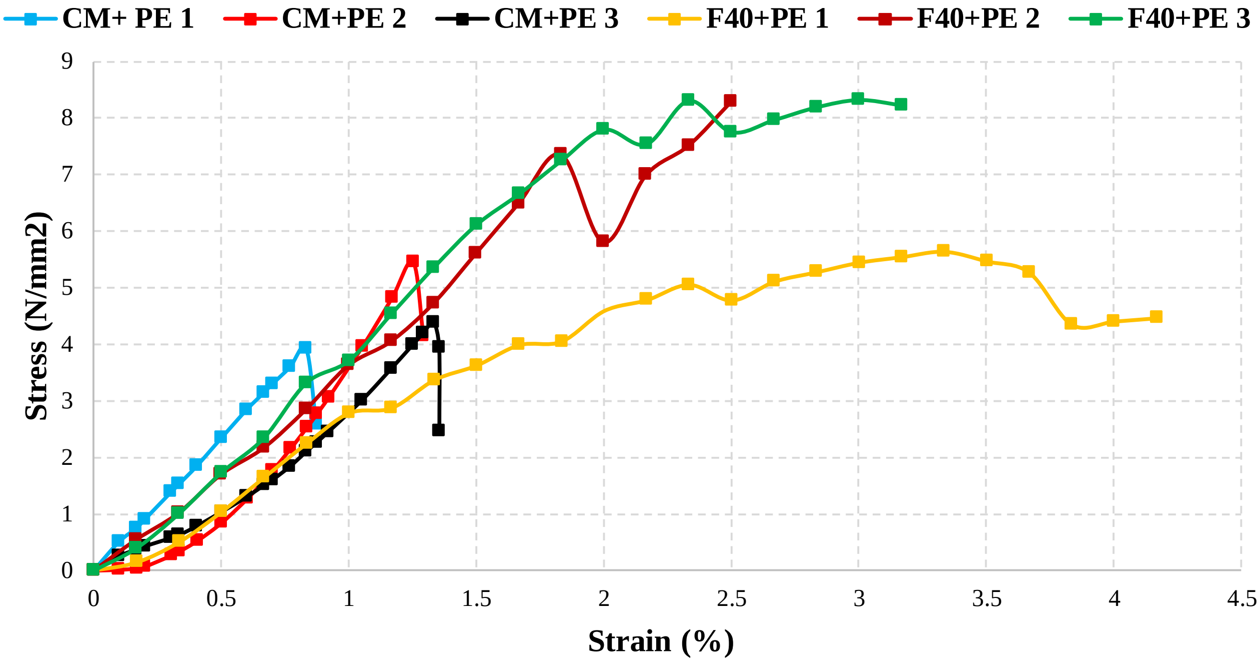

Figure 5.4: Graphical representation of stress strain behaviour of CM and FACs specimens with PE fiber.

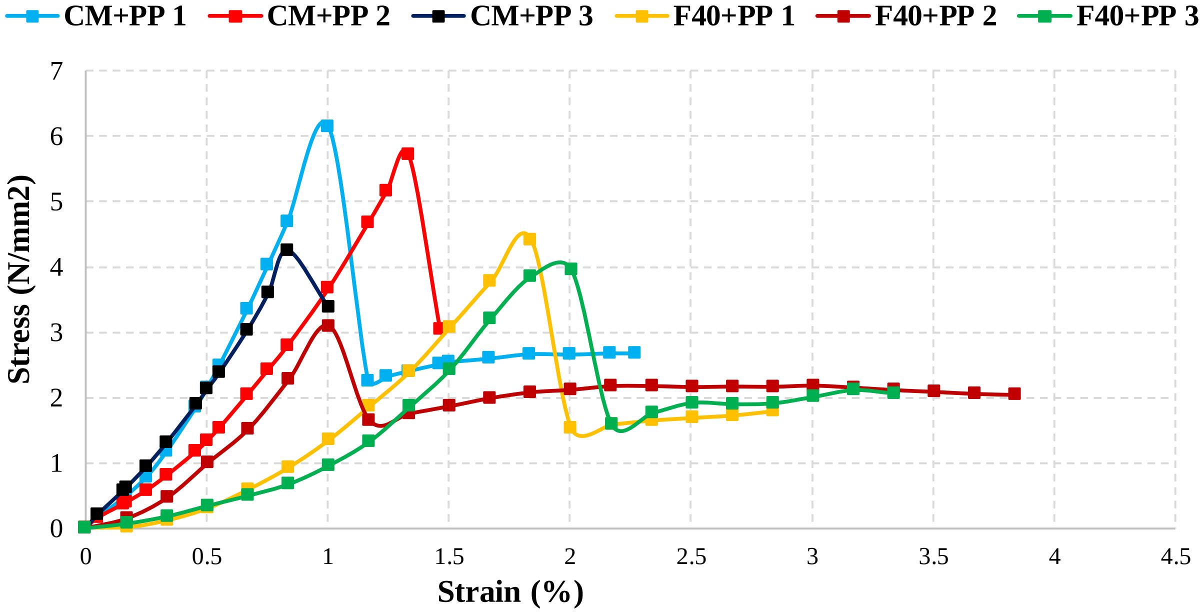

Figure 5.5: Graphical representation of stress strain behaviour of CM and FACs specimens with PP fiber

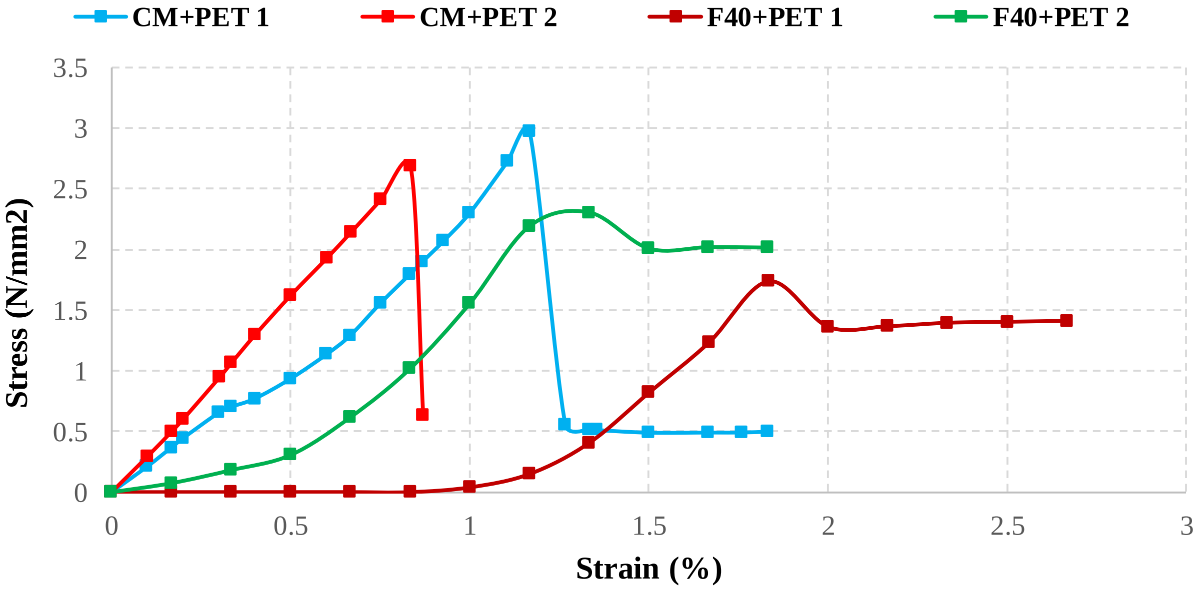

Figure 5.6: Graphical representation of stress strain behaviour of CM and FACs specimens with PET fiber

5.1.1.3 Flexural Strength

Flexural strength is also identified as bending strength or modulus of rupture (MOR); is the characteristic of a material and is define as material stress just before yielding in a flexural test. It can be found by using formula mentioned below in Eq. 5.1.

$$\textbf{Flexural Strength = MOR =} \frac{3FmaxS}{2\,w\,h^2} \qquad \textbf{Eq. 5.1}$$

Similarly, flexural strain can be found by formula mentioned in Eq. 5.2.

$$\textbf{Flexural Strain =} \frac{6\,H\,h}{S^2} \qquad \textbf{Eq. 5.2}$$

Where ‘S’ is the clear span of specimen. ‘Fmax’ is the maximum load acting on the specimen at the time of failure, ‘w’ and ‘h’ are width and height of specimen at mid-point, and ‘H’ is maximum deflection of sample at mid-point.

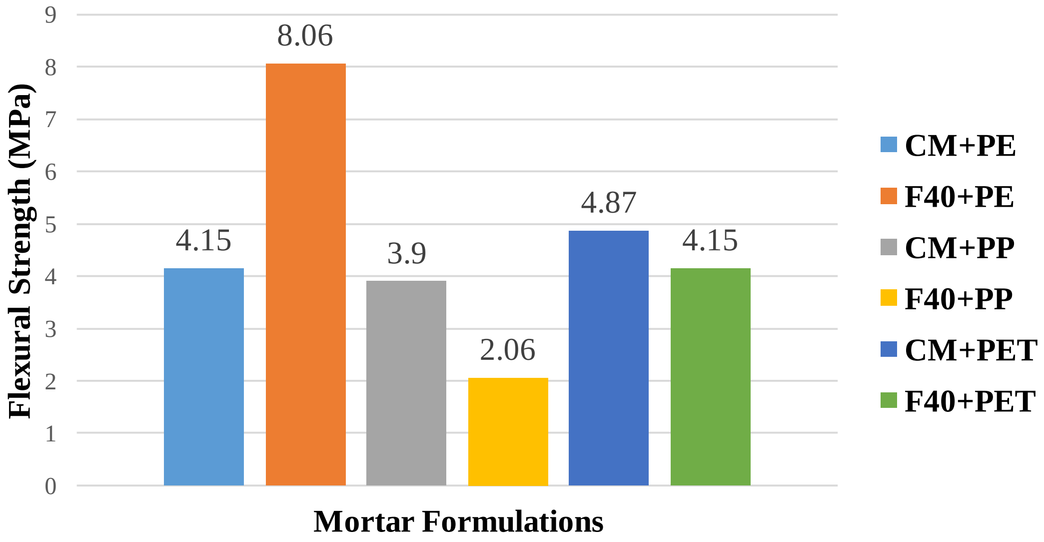

Figure 5.7: Flexural strength (or MOR) of CM and FACs with PE, PP and PET fibers

In case of PE fiber reinforcement, it was found that flexural strength (Figure 5.6) of FACs specimens was increased by 44.7% along with 68.6 % enhancement in rupture strain in flexural (Figure 5.7) as compared to CM specimens presenting increasing tendency with the addition of FAC.

But in case of PP fibers, flexural strength of FACs specimens was decreased by 47.17% along with 42.10% enhancement in rupture strain in flexural as compared to CM specimens presenting decreasing tendency of flexural strength with the addition of FACs.

In case of PET fibers, it was found that flexural strength of FACs specimens was decreased by 14.7% along with 50.26 % enhancement in rupture strain in flexural (Figure 5.7) as compared to CM specimens.

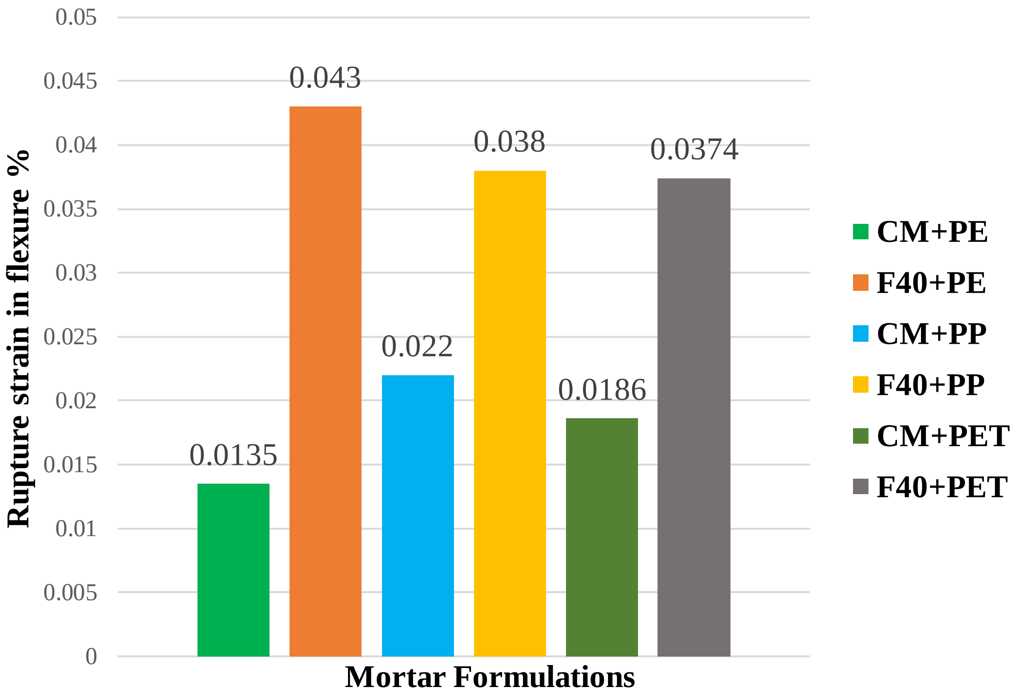

Figure 5.8: Rupture strain in flexure of CM and FACs with PE, PP and PET fibers.

Table 5.1: Flexural strength of CM and FACs specimens with PE, PP and PET fibers

FACs (%age wt. of cement)	Type of fiber	Flexural strength (MPa)
0.0	1% PE	4.15
0.40	1% PE	8.06
0.0	1% PP	3.9
0.40	1% PP	2.06
0.0	1% PET	4.87
0.40	1% PET	4.15

Table 5.2: Rupture strain in flexure of CM and FACs specimens with PE, PP and PET fiber

FACs (%age wt. of cement)	Type of fiber	Rupture strain in Flexure %
0.0	1% PE	0.0135
0.40	1% PE	0.043
0.0	1% PP	0.022
0.40	1% PP	0.038
0.0	1% PET	0.0186
0.40	1% PET	0.0374

5.1.1.4 Toughness

Toughness is a material property and related with its resistance to fracture. It is the ability of material to absorb energy before fracture [112].

5.1.1.4.1 Toughness Index

It is the most suitable approach for calculation of post-crack toughness of material. As per ACI Committee 544, it is the ratio of energy required to deflect the fiber reinforced specimen up-to fracture and the energy required to initiate the first crack in specimen. It can be evaluated from following formula (Eq. 5.3) provided the specimens are prepared and tested as per standards of ASTM C 1018 to create the load-displacement curves of the specimens [113-115].

$$\textbf{Toughness Index} = \frac{\boldsymbol{A1}}{\boldsymbol{A2}} \qquad \text{Eq. 5.3}$$

Where A_1 and A_2 are the areas under load-displacement curve up-to the fracture and first crack (also called the proportional limit of load-displacement curve) respectively.

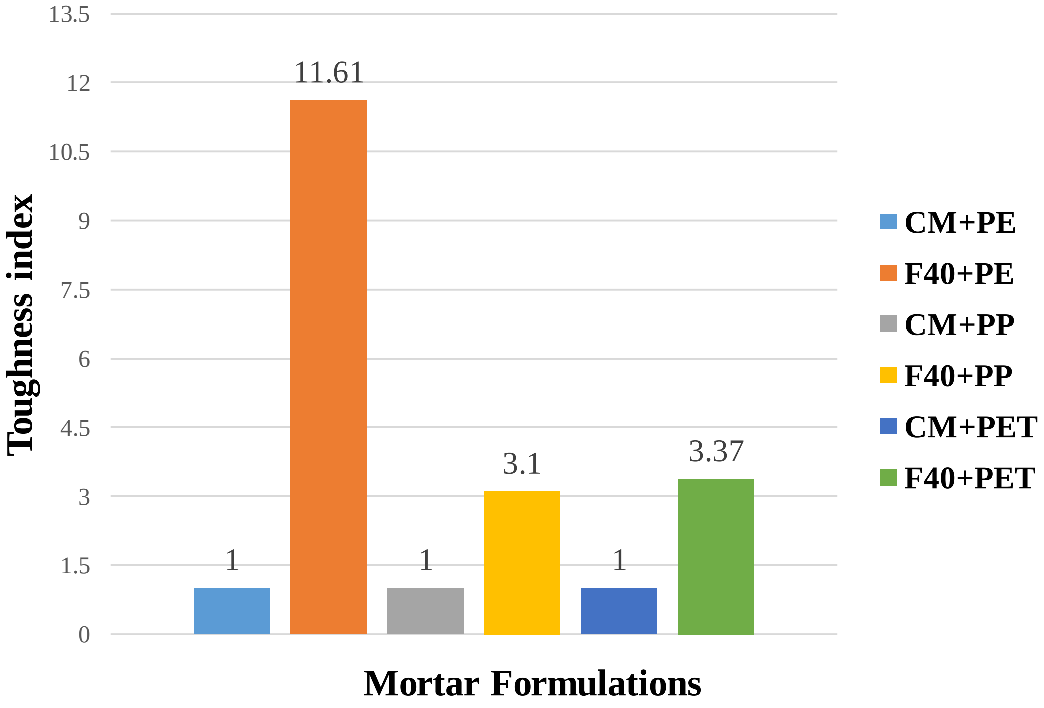

Figure 5.9: Toughness index of CM and FACs with PE, PP and PET fibers.

Table 5.3: Toughness index of CM and FACs specimens with PE, PP and PET fiber

FACs (%age wt. of cement)	Type of fiber	Toughness index
0.0	1% PE	1
0.40	1% PE	11.61
0.0	1% PP	1
0.40	1% PP	3.10
0.0	1% PET	1
0.40	1% PET	3.37

For PE (Figure 5.9), an addition of 40% FAC has enhanced 91% toughness index as compared to CM specimens Whereas for PP, 67.74% improvement in toughness index was observed on FAC inclusion and for PET fibers, 70.32% enhancement of toughness index was found on 40% addition of FAC as compared to CM specimens.

5.3 Density (unit weight)

The density of samples was measured at the age of 28 days under the saturated surface dry (SSD) condition. It is evidently seen that the 28-day SSD density of all the FACs based samples are less than 1600 kg/m3 and the lightest one is only 1420 kg/m3 which clearly indicates its classification as light weight concrete.

The higher the amount of FACs, the lower its density. So, the densities of FACs based specimens are significantly lower than cement based specimens due to the high air content accompanying with hollow nature of FACs particles and the air voids in the resulting composites. Whereas, the spherical shapes have been difficulty packed entirely, so the air voids may stay within.

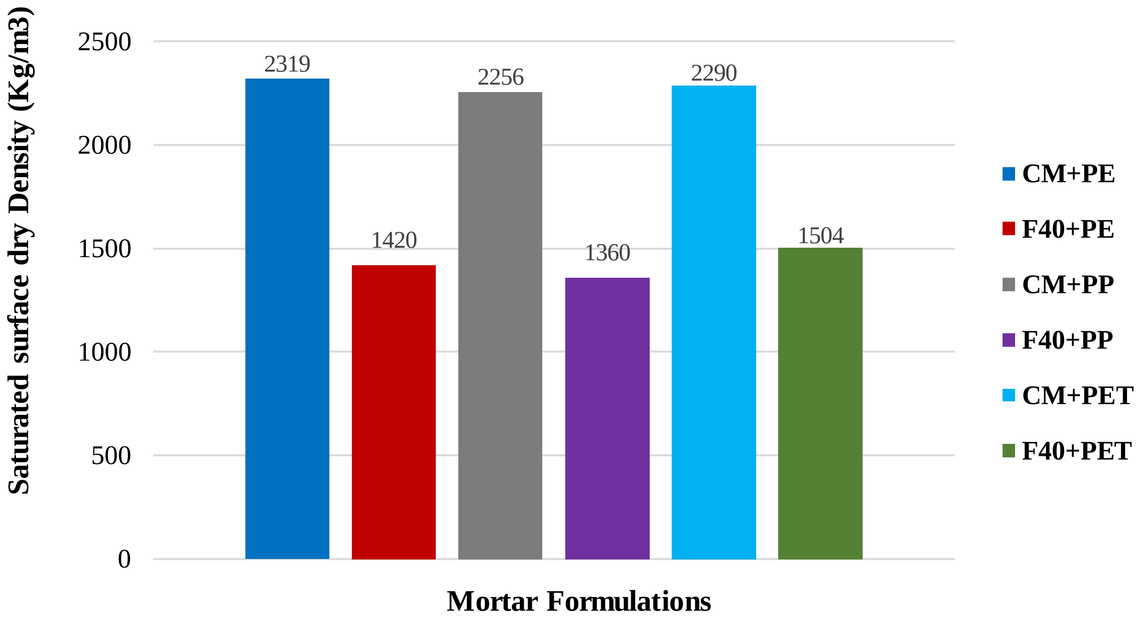

Figure 5.10: Densities (or unit weigh) of CM and FACs with PE, PP and PET fibers at 28-days

5.4 Compressive Strength Test

Compressive strength of CM specimens with and without addition of FACs was determined with compression testing machine. The results are average of 6 broken halves tested for a formulation. Following results were obtained mentioned in Figure 5.8. In case of Polyethylene (PE) fiber, the CM specimens increased the compressive strength by 46.13 % as compared to cementitious composites containing 40% FACs.

In case of Polypropylene (PP) fiber, the CM specimens increased the compressive strength by 49.55 % as compared to cementitious composites containing 40% FACs.

In case of Polyethylene terephthalate (PET) fiber, the CM specimens increased the compressive strength by 49.65% as compared to cementitious composites containing 40% FACs.

Results showed that CM formulation with Polyethylene (PE) fiber gives the maximum strength value i-e 89.3 MPa as compared to other formulations.

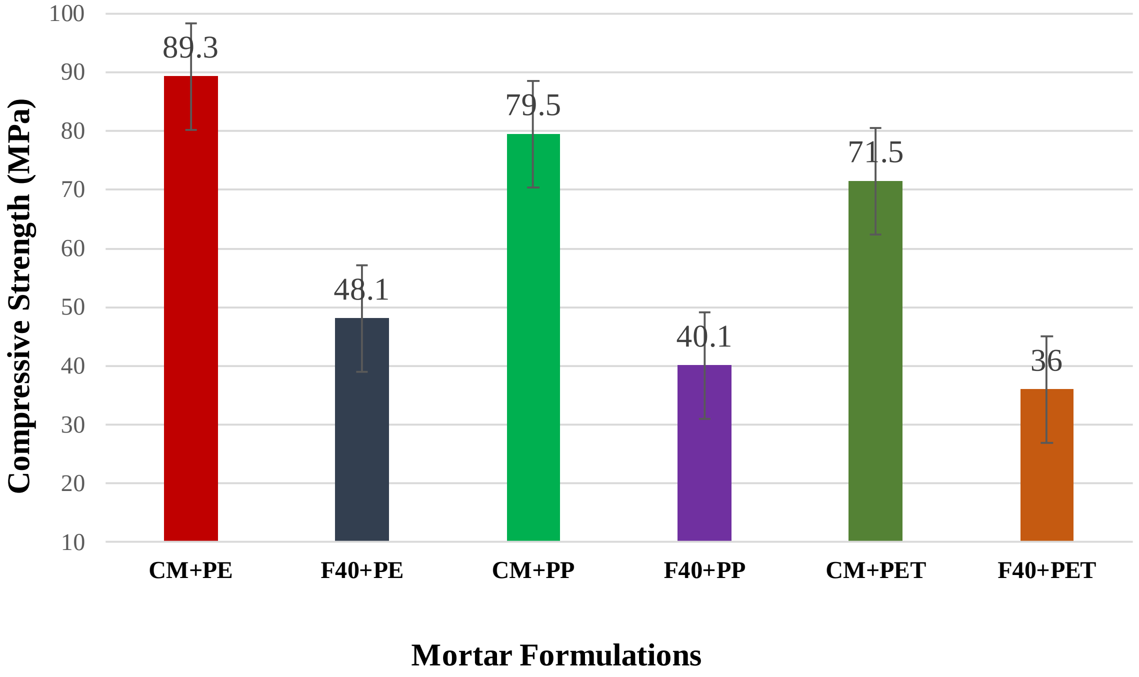

Figure 5.11: Compressive strength of CM and FACs with PE, PP and PET fibers at 28-days

5.5 Specific strength

The ratio of compressive strength to unit weigh is known as specific strength. To relate strength of the lightweight FACs composite with normal concrete of comparable density, we should determine the specific strength. It is shown in fig (5.9). that the composites even having lesser compressive strength values resulted in higher values of specific strength due to the ultra-lightweight nature of FACs. The lowest specific strength found was 23.93 which means that this particular composite (FAC-PET) is correspondent in strength to a normal weight concrete (of density 2400 kg/m3) have compressive strength of 57.43 MPa approximately which specifies satisfactory level of mechanical properties.

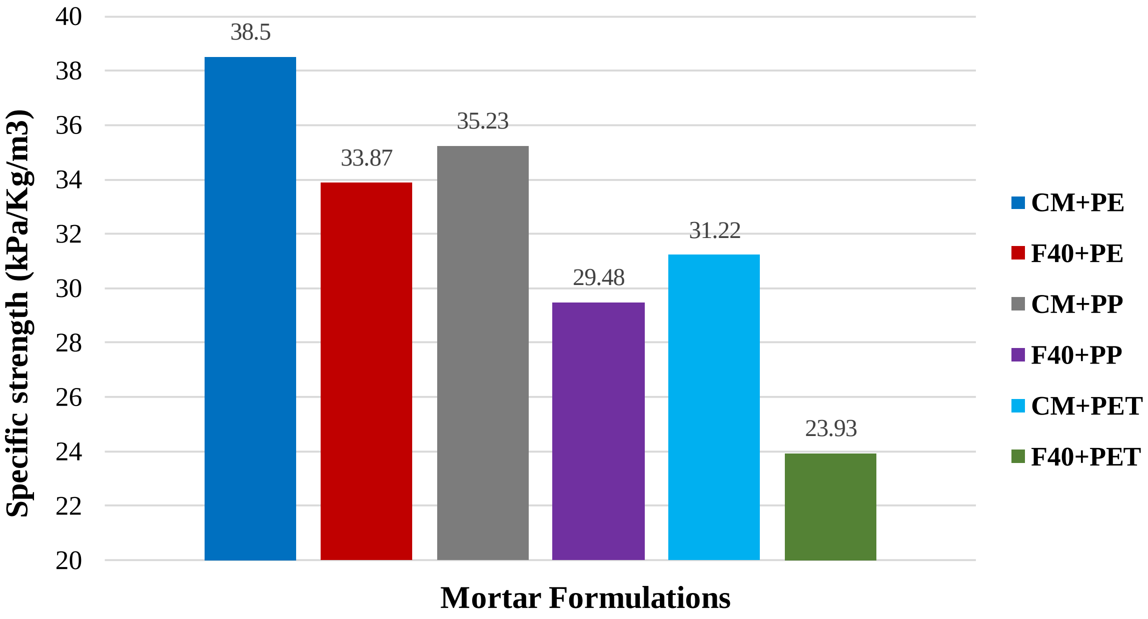

Figure 5.12: Specific strength values at 28-days

CHAPTER 6: Industrial applications

In common parlance, FACs behaves as a light weight filler material in the cementitious system. Rapid utilization and urbanization of coal for power manufacturing has extremely enhanced the annual waste generation. Though by using fly ash as a SCM as replacement cement in concrete, even quantity used is minute, a huge portion leaving after to be washed out in landfills. So using the FACS as Lightweight aggregates (LWA) lessens the waste to a larger extent. FACS based cementitious composites are exceptional aspirants for making mechanically tough and thermally insulated composites having huge capability to utilize in structures for energy conservation. Due to this reason Electricity consumption reduces. FACS are used in low cost pre-cast construction.

CONCLUSIONS AND RECOMMENDATIONS

1. The Flexural strength decreases in case of PP and PET fibers as compared to control mix, because of the air voids in the composites and lesser mechanical strength due to the thinner shell thickness of FACs particles. Flexural strength reduces with lessening density (with addition of FACs) but this trend is not linear in all types of fibers because of irregular distribution of fibers and change of type or nature of fiber.
2. The density and Compressive strength of FAC based composites drop directly as addition of FACs which is used as filler aggregate. Due to light weight nature of the FACs, density of resulting composites reduced. Compressive strength is direct function of density.
3. For 28 days, the hardened density of FACC has been described in the range of 1420-1504 kg/m^3 with the consistent compressive strength range of 36-48.1MPa and flexural strength range of 4.15- 8.06 leading to higher specific strength values.
4. FACs are very efficient in forming fiber reinforced cement based composites with different types of fibers and depicts exceptional bonding with PE fibers in composite. Though, for improved ductility and much prominent strain hardening performance, by increasing fiber volume fraction could be helpful.

5. Due to the existence of lime and amorphous silica, FACs holds some degree of pozzolanic activity which is cause for FAC composites to attain high strength to weight ratio. The pozzolanic activity is restricted to the age of 28 days.

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