MECHANICAL PROPERTIES AND SULPHATE RESISTANCE OF CONCRETE, INCORPORATING USED FOUNDRY SAND, SILICA FUME AND METAKAOLIN

A thesis submitted in partial fulfilment of the requirement for the award of degree of
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CERTIFICATE

This is to certify that the work presented in Thesis entitled “Mechanical properties and sulphate resistance of concrete incorporating foundry sand, silica fume and metakaolin” submitted by**, ISHWAR PARKASH RAWAT, Roll No. 801022010** in partial fulfilment of the requirements for the award of **Masters of Engineering in Civil (Structures) at Thapar University, Patiala**, is an authentic record of student’s own work carried out under our supervision and guidance. The embodied in this thesis has not been submitted anywhere for award of any other degree.

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ABSTRACT

Sand has been used for centuries as a moulding material in ferrous and non-ferrous metal casting industries because of its thermal conductivity. Foundries successfully recycle and reuse the sand many times in a foundry. When the sand can no longer be reused in the foundry, it is removed from the foundry and is termed as foundry sand. Use of foundry sand in various engineering applications can solve the problem of disposal of foundry sand and other purposes. This waste foundry sand has been used in this study by replacing the fine aggregates (ordinary sand) with 10% of waste foundry sand in all concrete mixes.

The primary aim of this study is to investigate the strength properties (compressive strength and splitting tensile strength) and sulphate resistance of concrete incorporating foundry sand mixed with Silica fume and Metakaolin. For this purpose cement is replaced by weight in three different proportions of 5%, 10% and 15% by Silica fume and Metakaolin separately. Specimens are tested for compressive strength, splitting tensile strength and sulphate resistance. Testing is done at age of 7, 28 and 56 days.

Concrete made in this work by replacing 10% foundry sand with fine aggregates and mixing Silica fume and Metakaolin passes all the plastic stage tests. Furthermore hardened stage tests (compressive strength and splitting tensile strength) results were also positive. There was increase in compressive strength and splitting tensile strength with incorporation of Silica fume and metakaolin. Also there was increase in sulphate resistance of SCC when cement is replaced by Silica fume and metakaolin.

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CHAPTER 1

INTRODUCTION

1.1 General

Concrete is one of the most common materials used in the construction industry. In the past few years, many research and modification has been done to produce concrete which has the desired characteristics. There is always a search for concrete with higher strength and durability. In this matter, blended cement concrete has been introduced to suit the current requirements. Cementitious materials known as pozzolans are used as concrete constituents, in addition to Portland cement. Originally the term pozzolan was associated with naturally formed volcanic ashes and calcined earths will react with lime at ambient temperatures in the presence of water. Recently, the term has been extended to cover all siliceous/aluminous materials which, in finely divided form and in the presence of water, will react with calcium hydroxide to form compounds that possess cementitious properties. The current area of research in the concrete is introducing silica fume and metakaolin in the concrete and 10% foundry sand in the ordinary sand.

1.2 Foundry sand

1.2.1 General

Waste Foundry sand is high quality silica sand with uniform physical characteristics. It is a by-product of ferrous and non-ferrous metal casting industries, where sand has been used for centuries as a moulding material because of its thermal conductivity. Foundries successfully recycle and reuse the sand many times in a foundry. When the sand can no longer be reused in the foundry, it is removed from the foundry and is termed as foundry sand.

Used foundry sand can be reused in various applications as an alternative to sending it to landfill, and reuse options are well established in England, Europe and North America. Reuse options include cement manufacture, asphalt, concrete, bricks and free-flow fill for certain construction applications. Some of these alternatives are starting to be adopted in India, but is still in early stage. Overseas examples show that it is not only better for the environment but is profitable for the foundry to use the sand alternatively. These foundries have significantly reduced the volume of waste sand going to landfill and actually offset the total cost of transporting the sand ‘in’ and ‘out’ (Siddique et al., 2011).

1.2.2 Uses and Availability

Foundry sand is basically fine aggregate. It can be used in many of the same ways as natural or manufactured sands. This includes many civil engineering applications such as embankments, flowable fill, hot mix asphalt (HMA) and portland cement concrete (PCC). Foundry sands have also been used extensively agriculturally as topsoil.

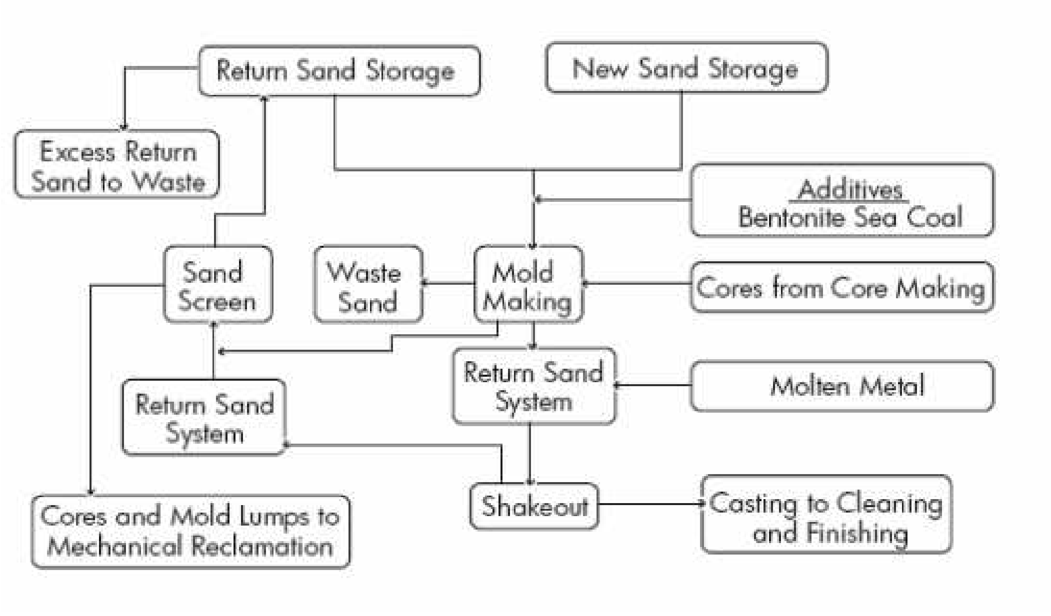

Fig. 1.1 Recycling of foundry sand

1.2.3 Physical characteristics of foundry sand

Foundry sand is typically sub angular to round in shape. After being used in the foundry process, a significant number of sand agglomerations form. When these are broken down, the shape of individual sand grains is apparent. Green sands are typically black, or gray, not green chemically bonded sand is typically a medium tan or off-white colour.

Fig. 1.2 Foundry sand (gyxzylc.en.made-in-china.com)

1.2.4 Types of Foundry Sand

Two general types of binder systems are used in metal casting depending upon which the foundry sands are classified as:

1. Clay bonded (Green sand)
2. Chemically bonded systems

Both types of sands are suitable for beneficial use but they have different physical and environmental characteristics:

- Green sand is composed of naturally occurring materials which are blended together; high quality silica sand (85-95%), bentonite clay (4-10%) as a binder, a carbonaceous additive (2-10%) to improve the casting surface finish and water (2-5%). Green sand is the most commonly used recycled foundry sand for beneficial reuse. It is black in color, due to carbon content, has a clay content that results in percentage of material that passes a 200 sieve and adheres together due to clay and water.

- Chemically bonded sands are used both in core making where high strengths are necessary to withstand the heat of molten metal, and in mold making. Most chemical binder systems consist of an organic binder that is activated by a catalyst although some systems use inorganic binders. Chemically bonded sands are generally light in color and texture than clay bonded sands.

1.2.5 Physical properties of foundry sand

Table 1.1 Typical physical properties of (Green) foundry sand

(American Foundryman's Society, 1991)

Property	Results	Test method
Specific gravity	2.39 – 2.55	ASTM D854
Bulk relative density, kg/m^3	2589 (160)	ASTMC 48 / AASTHO T84
Absorption, %	0.45	ASTM C128
Moisture content, %	0.1 – 10.1	ASTM D2216
Coefficient of permeability (cm/sec)	10^3-10^6	AASTHO T215 / ASTM D2434
Plastic limit/plastic index	Non-plastic	AASTHO T90 / ASTM D4318

1.2.6 Chemical Composition of foundry sand

Chemical Composition of the foundry sand relates directly to the metal molded at the foundry. This determines the binder that was used, as well as the combustible additives. Typically, there is some variation in the foundry sand chemical composition from foundry to foundry. Sands produced by a single foundry, however, will not likely show significant variation over time. Moreover, blended sands produced by consortia of foundries often produce consistent sands. The chemical composition of the foundry sand can impact its performance. Spent foundry sand consists primarily of silica sand, coated with a thin film of burnt carbon, residual binder (bentonite, sea coal, resins) and dust. Silica sand is hydrophilic and consequently attracts water to its surface. This property could lead to moisture-accelerated damage and associated stripping problems in an asphalt pavement. Antis tripping additives may be required to counteract such problems. Depending on the binder and type of metal cast, the pH of spent foundry sand can vary from approximately 4 to 8. It has been reported that some spent foundry sands can be corrosive to metals. Because of the presence of phenols in foundry sand, there is some concern that precipitation percolating through stockpiles could mobilize leach able fractions, resulting in phenol discharges into surface or ground water supplies. Foundry sand sources and stockpiles must be monitored to assess the need to establish controls for potential phenol discharges.

1.2.7 Foundry Sand Economics

The success of using foundry sand depends upon economics. The bottom line issues are cost, availability of the foundry sand and availability of similar natural aggregates in the region. If these issues can be successfully resolved, the competitiveness of using foundry sand will increase for the foundries and for the end users of the sand. This is true of any recycled material.

1.2.8 Foundry Sand Engineering Characteristics

Since foundry sand has nearly all the properties of natural or manufactured sands, it can normally be used as a sand replacement. It can be used directly as a fill material in embankments. It can be used as a sand replacement in hot mix asphalt, flowable fills, and Portland cement concrete. It can also be blended with either coarse or fine aggregates and used as a road base or sub base material.

1.3 Silica Fume

Silica fume is a highly reactive pozzolanic material primarily composed of silicon dioxide ($\mathrm{Si}O_2$) in noncrystalline form. It is a light to dark grey or bluish green-grey powder produced by an electric arc furnace during the manufacture of silicon or ferrosilicon alloy. It has a spherical like fly ash, but it is 100 times smaller, with an average diameter size of 0.1 μm. Silicon, ferrosilicon and other alloys are produced by reducing quartz with coal and iron or other ores, at very high temperatures (2000°C) in electric arc furnaces. Some silicon gas or fume is produced in the process, which reaches the top of the furnace with other combustion gases, where it becomes oxidized to silica in contact with the air and then condenses as < 0.1 um to 1 um spherical particles of amorphous silica (85-96 % SiO_2). However, the spherical particles are usually agglomerated so that the effective particle size is much coarser. Silica fume used in concrete derives from the manufacture of ferrosilicon alloys and is modified by densification, micropelletization or slurrification, to facilitate transportation and handling. Condensed silica fume has a surface area of about 2000 m^2/kg and a relative density generally in the range of 2.20 to 2.5.

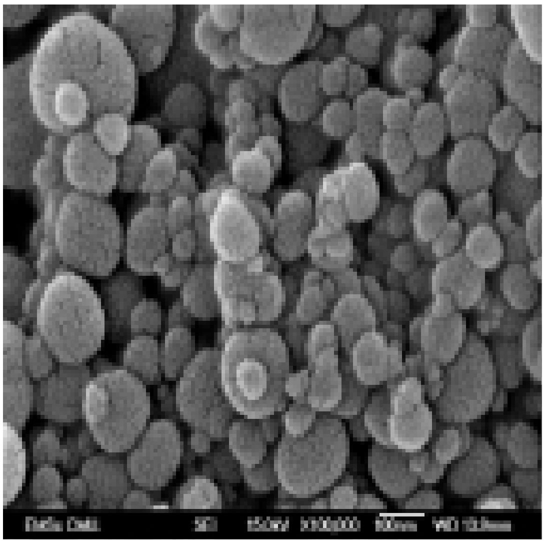

Fig. 1.3 Scanning electron micrograph of silica fume particles (Chaipanich, 1998)

Table 1.2 Physical properties of silica fume (khayat et al., 1987)

Colour	Varies from white or pale-grey to a dark grey
Specific gravity	It is generally equal to that amorphous silicon which is about 2.2. depending upon its chemical composition the specific gravity of silica fume particle can be as high as 2.4 – 2.55
Specific surface area	About 20000m^2/kg approx 10 times more than Portland cement
Particle size	Mostly fine spheres with a mean dia of .1 micron
Bulk loose density	230 – 300 kg/m^3

The pozzolanic reactions take place when silica fume is added to the concrete mixture, and the amorphous silica, which is the major component of the pozzolana, reacts with calcium hydroxide formed from the hydration of the calcium silicates with the resulting product being a calcium silicate hydrate (C-S-H). Usually after micro silica is being added to the concrete mix, the matrix of micro silica concrete becomes very dense (St. John, 1998). This denseness effect has been attributed to the extreme fineness of micro silica, due to the fact that 50,000 to 100,000 microspheres exist for every cement grain, allowing micro silica hydration products to infill the water spaces usually left within the cement hydrates. Silica fume is used in amounts between 5% and 10% by mass of the total cementitious material, in applications where high degree of impermeability and high compressive strength are needed in concrete. In some situations, the water demand of concrete containing silica fume increases with increasing amounts of silica fume, unless a water reducer or plasticizer is used. Some lean mixes may not experience an increase in water demand when only a small amount (less than 5%) of silica fume is present.

Table 1.3 Chemical composition of Silica Fume (khayat et al., 1987)

Constituents	Percent
SiO_2	90 – 96
Al_2O_3	0.5 – 0.8
MgO	0.5 – 1.5
Fe_2O_3	0.2 – 0.8
CaO	0.1 – 0.5
Na_2O	0.2 – 0.7
K_2O	0.4 – 1.0
C	0.5 – 1.4
S	0.1 – 0.4

1.4 Metakaolin

Metakaolin which is obtained by the calcinations of pure or refined kaolinitic clay at a temperature of between 6500C and 8500C, followed by grinding to achieve a fineness of 700-900 m^2/kg exhibits high pozzolanity. When used in concrete it will fill the void space between cement particles resulting in a more impermeable concrete. The metakaolin used in this study is Metacem of grade 85-C. The physical properties of metakaolin are given in Table 1.4.

Table 1.4 Physical properties of Metakaolin (www.metakaolin.com)

Properties	Results
Brightness	76%
Bulk density	308 gm/l
Oil absorption	56.9 gm/100 gm
Moisture	0.23%
Residue on 400 µm	0.6%
ρH	6.5

Unlike industrial by-products such as fly ash, silica fume, and blast-furnace slag, Metakaolin is refined carefully to lighten its colour, remove inert impurity, and control particle size. The particle size of Metakaolin is generally less than 2 µm, which is significantly smaller than that of cement particles, though not as fine as silica fume. Moreover, the use of Metakaolin in concrete in its present form is relatively a new concept. Recent works have shown that the inclusion of Metakaolin greatly influenced the mechanical and durability properties of concrete. It has also been demonstrated that concrete mixture incorporating high-reactivity Metakaolin gave comparable performance to silica fume mixtures in terms of strength, permeability, and chemical resistance. The utilization of this material is also environmentally friendly since it helps in reducing the CO_2 emission to the atmosphere by the minimization of the Portland cement (PC) consumption.

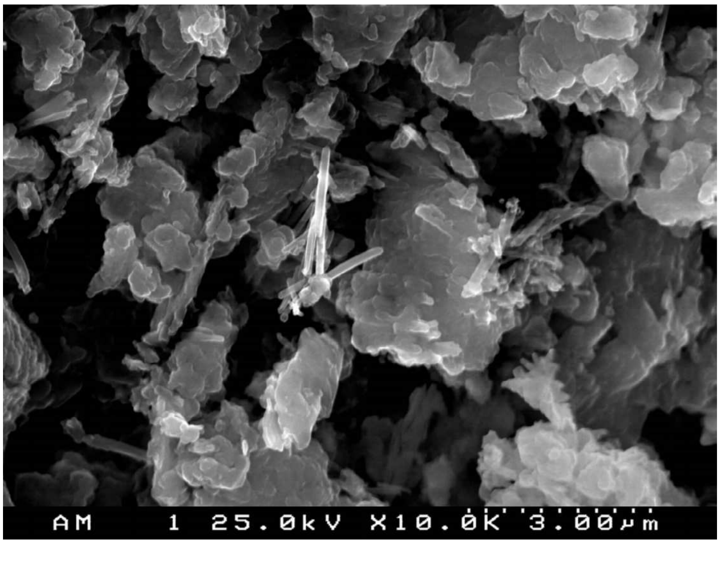

Fig. 1.4 Pozzolanic Reaction (CSH formation) in Metakaolin. (www.metakaolin-pro.eu)

1.5 Objective of this Study

This study is conducted to accomplish some predefined objectives:

- To study the performance of concrete containing 10% waste foundry sand replaced with fine aggregate (ordinary) sand.
- To study the performance of concrete incorporating 10% foundry sand, containing different percentages of metakaolin and silica fume to identify the optimum replacement percentage.
- To compare the performance of concrete mixed with metakaolin with silica fume concrete.

1.6 Significance of this Study

Concrete has been used in the construction industry for centuries. Many modifications and developments have been made to improve the performance of concrete, especially in terms of strength and durability.

The introduction of pozzolans as cement replacement materials in recent years seems to be successful. The use of pozzolan has proven to be an effective solution in enhancing the properties of concrete in terms of strength and durability. The current pozzolans in use are such as silica fume, fly ash and Metakaolin. Development and investigation of other sources of pozzolan such as kaolin will be able to provide more alternatives for the engineer to select the most suitable cement replacement for material for different environments.

Unlike other pozzolans, Metakaolin is not a by-product which means its engineering values are well-controlled. Therefore, using metakaolin should promise some advantages compared to other cement replacement materials. In this case, studies are needed to study the performance of concrete using metakaolin. The performance of metakaolin-concrete will be compared to the cost of production of metakaolin to determine whether metakaolin is worthy to be developed as a new cement replacement material.

In addition, the use of metakaolin is not common in the Indian construction sector. This study will be able to enhance the understanding on the suitability of metakaolin as cement replacement material.

CHAPTER 2

LITERATURE REVIEW

2.1 Concrete

Concrete is a mixture of cement, water, and aggregates, with or without admixtures. The cement and water will form a paste that hardens as a result of a chemical reaction between the cement and water. The paste acts as glue, binding the aggregates (sand and gravel or crushed stone) into a solid rock-like mass. The quality of the paste and the aggregates dictate the engineering properties of this construction material. During hydration and hardening, concrete will develop certain physical and chemical properties, among others, mechanical strength, low permeability and chemical and volume stability. Concrete has relatively high compressive strength, but significantly lower tensile strength, about 10% of the compressive strength.

Table 2.1: Typical Properties of Normal-Strength Portland cement Concrete.

(Gambhir, 1995)

Compressive strength	20–40 MPa
Flexural strength	3–5 MPa
Tensile strength	2–5 MPa
Modulus of elasticity	14,000–41,000 MPa
Permeability	$1x10^{-10}$ cm/sec
Coefficient of thermal expansion	10^{-5}/°C
Drying shrinkage	4–8 x 10^{-4}
Drying shrinkage of reinforced concrete	2–3 x 10^{-4}

2.2 Cement

Cement may be defined as an adhesive substance capable of uniting fragments or masses of solid mater to a compact whole. Portland cement was invented in 1824 by an English mason, Joseph Aspin, who named his product Portland cement because it produced a concrete that was of the same colour as natural stone on the Isle of Portland in the English Channel.

Raw materials for manufacturing cement consist of basically calcareous and siliceous (generally argillaceous) material. The mixture is heated to a high temperature within a rotating kiln to produce a complex group of chemicals, collectively called cement clinker (Neville, 1987). Cement is distinct from the ancient cement. It is termed hydraulic cement for its ability to set and harden under water. Briefly, the chemicals present in clinker are nominally the four major potential compounds and several minor compounds. The four major potential compounds are normally termed as Tricalcium silicate ($3CaO.SiO_2$), Dicalcium silicate ($2CaO.SiO_2$), Tricalcium aluminate ($3CaO.Al_2O_3$) and Tetracalcium aluminoferrite ($4CaO. Al_2O_3.Fe_2O_3$).

The American Society for Testing and Materials (ASTM) Standard C 150, Specification for Portland cement, provides for the following types of Portland cement:

- Type I General Portland cement
- Type II Moderate-sulphate-resistant cement
- Type III High-early-strength cement
- Type IV Low–heat-of-hydration cement
- Type V High-sulphate-resistant cement

2.3 Cement Replacement Material

With the extensively use of cement in concrete, there has been some environmental concerns in terms of damage caused by the extraction of raw material and CO_2 emission during cement manufacture. This has brought pressures to reduce the cement consumption in the industry. At the same time, there are getting more requirements for enhancement in concrete durability to sustain the changing environment which is apparently different from the old days.

With the development in concrete technology, cement replacement materials (CRM) have been introduced as substitutes for cement in concrete. Several types of materials are in common use, some of which are by-products from other industrial processes, and hence their use may have economic advantages. However, the main reason for their use is that they can give a variety of useful enhancements or modifications to the concrete properties. All the materials have two common features:

i) Their particle size range is similar to or smaller than that of Portland cement.

ii) They are pozzolan material.

2.4 Pozzolanic Behaviour

A common feature of nearly all CRM is that they exhibit pozzolanic behaviour. Pozzolanic material is the material which contains active silica (SiO_2) and is not cementitious in itself but will, in a finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form cementitious compounds. (Malhotra et at., 1983)

Types of Cement Replacement Materials

The cement replacement materials that are used in this study are:

2.5 Silica Fume

Silica fume (SF) is a by product of the smelting process in the silicon and ferrosilicon industry. The reduction of high-purity quartz to silicon at temperatures up to 2000℃ produces $\mathrm{Si}O_2$ vapours, which oxidizes and condense in the low temperature zone to tiny particles consisting of non-crystalline silica. By-products of the production of silicon metal and the ferrosilicon alloys having silicon contents of 75% or more contain 85–95% non-crystalline silica. The by-product of the production of ferrosilicon alloy having 50% silicon has much lower silica content and is less pozzolanic. Therefore, $\mathrm{Si}O_2$ content of the silica fume is related to the type of alloy being produced.

Silica fume is also known as micro silica, condensed silica fume, volatilized silica or silica dust. The American concrete institute (ACI) defines silica fume as a ‘‘very fine non crystalline silica produced in electric arc furnaces as a by product of production of elemental silicon or alloys containing silicon’’. It is usually a grey coloured powder, somewhat similar to Portland cement or some fly ashes. It can exhibit both pozzolanic and cementitious properties.

Silica fume has been recognized as a pozzolanic admixture that is effective in enhancing the mechanical properties to a great extent. By using silica fume along with super plasticizers, it is relatively easier to obtain compressive strengths of order of 100–150 MPa in laboratory. Addition of silica fume to concrete improves the durability of concrete through reduction in the permeability, refined pore structure, leading to a reduction in the diffusion of harmful ions, reduces calcium hydroxide content which results in a higher resistance to sulphate attack. Improvement in durability will also improve the ability of silica fume concrete in protecting the embedded steel from corrosion.

Figure 2.1 shows the schematic diagram of silica fume production. The silica fume is collected in very large filters in the bag house and then made available for use in concrete.

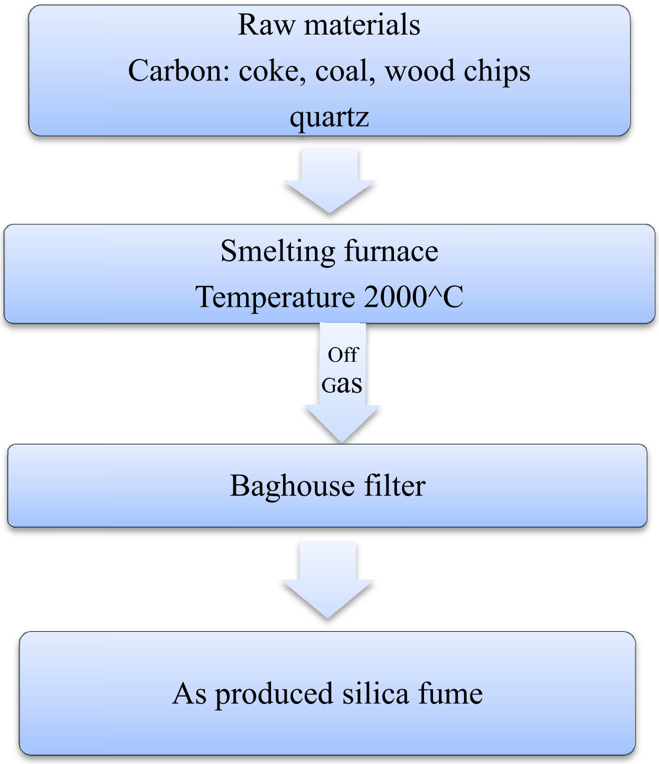

Fig. 2.1 Schematic diagram of silica fume production

Fig. 2.2 Silica fume (mainsource.en.alibaba.com)

2.5.1 Properties of Silica Fume

Physical Properties

Silica fume particles are extremely small, with more than 95% of the particles finer than 1 lm. Its typical physical properties are given in Table 2.2. Silica fume colour is either premium white or grey as shown in Fig. 2.2.

Chemical Composition

Silica fume is composed primarily of pure silica in non-crystalline form. X-ray diffraction analysis of different silica fumes reveals that material is essentially vitreous silica, mainly of cristobalite form. Silica fume has a very high content of amorphous silicon dioxide and consists of very fine spherical particles. Silica fume generally contains more than 90% SiO2. Small amounts of iron, magnesium, and alkali oxides are also found.

Table 2.2 Chemical composition of silica fume (Khayat and Aitcin, 1987)

Constituents	Percent
SiO_2	90 – 96
Al_2O_3	0.5 – 0.8
MgO	0.5 – 1.5
Fe_2O_3	0.2 – 0.8
CaO	0.1 – 0.5
Na_2O	0.2 – 0.7
K_2O	0.4 – 1.0
C	0.5 – 1.4
S	0.1 – 0.4

2.5.2 Reaction Mechanism

Because of its extreme fineness and very high amorphous silicon dioxide content, silica fume is a very reactive pozzolanic material. As the Portland cement in concrete begins to react chemically, it releases calcium hydroxide. The silica fume reacts with this calcium hydroxide to form additional binder material called calcium silicate hydrate which is very similar to the calcium silicate hydrate formed from Portland cement. It is an additional binder that gives silica-fume concrete its improved properties. Mechanism of silica fume in concrete can be studied basically under three roles:

(i) Pore-size Refinement and Matrix Densification:

The presence of silica fume in the Portland cement concrete mixes causes considerable reduction in the volume of large pores at all ages. It basically acts as filler due to its fineness and because of which it fits into spaces between grains in the same way that sand fills the spaces between particles of coarse aggregates and cement grains fill the spaces between fine aggregates grains.

(ii) Reaction with Free-Lime (From Hydration of Cement)

CH crystals in Portland cement pastes are a source of weakness because cracks can easily propagate through or within these crystals without any significant resistance affecting the strength, durability and other properties of concrete. Silica fume which is siliceous and aluminous material reacts with CH resulting reduction in CH content in addition to forming strength contributing cementitious products which in other words can be termed as ‘‘Pozzolanic Reaction’’.

(iii) Cement Paste–Aggregate Interfacial Refinement

In concrete the characteristics of the transition zone between the aggregate particles and cement paste plays a significant role in the cement-aggregate bond. Silica fume addition influences the thickness of transition phase in mortars and the degree of the orientation of the CH crystals in it. The thickness compared with mortar containing only ordinary Portland cement decreases and reduction in degree of orientation of CH crystals in transition phase with the addition of silica fume. Hence mechanical properties and durability is improved because of the enhancement in interfacial or bond strength. Mechanism behind is not only connected to chemical formation of C–S–H (i.e. pozzolanic reaction) at interface, but also to the microstructure modification (i.e. CH) orientation, porosity and transition zone thickness) as well.

2.5.3 Advantages of Using Silica Fume

- High early compressive strength
- High tensile, flexural strength and modulus of elasticity
- Very low permeability to chloride and water intrusion
- Enhanced durability
- Superior resistance to chemical attack from chlorides, acids, nitrates and sulphates and life-cycle cost efficiencies.
- Higher bond strength
- High electrical resistivity and low permeability

2.5.4 Effect of Silica fume on fresh properties of concrete

Fresh concrete containing silica fume is more cohesive and less prone to segregation than concrete without silica fume. Concrete containing silica fume shows substantial reduced bleeding. Additionally silica fume reduces bleeding by physically blocking the pores in the fresh concrete. Use of silica fume does not significantly change the unit weight of concrete.

a) Consistency

Rao (2003) determined the influence of silica fume on the consistency of cement pastes and mortars. Specific gravity and specific surface of the silica fume were 2.05 and 16,000 m²/kg, respectively. Silica fume was varied from 0 to 30% at a constant increment of 2.5/5% by weight of cement. Since the SF is finer than the cement, the specific surface increased with increase in SF content. The standard consistency of pure cement paste was found out to be 31.50%; while at 30% SF, it was 44.25%. It was observed that the consistency of cement increased with the increase in SF content. As much as 40% of additional water requirement was observed for cement pastes containing 20–30% SF. Fig. 2.3 shows the variation of consistency of cement at different silica fume contents.

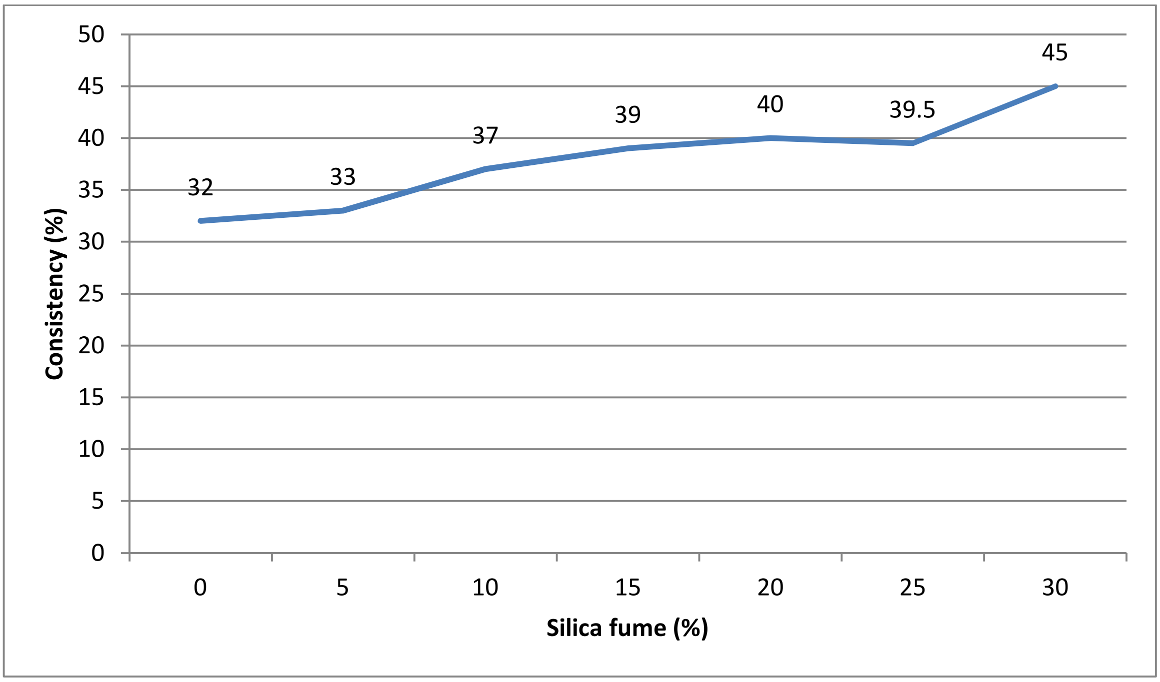

Fig. 2.3 Variation of consistency of cement pastes containing different percentages of silica fume (Rao, 2003)

Qing et al. (2007) examined the influence of nano-SiO_2 (NS) addition on consistency of cement paste incorporating NS or silica fume. The influence of NS or silica fume addition on consistency and setting time of fresh pastes is given in Table 2.3. It was found that with a increase in the NS content, fresh pastes for sample A-series grew thicker gradually and their penetration depths (consistency value) decreased gently as compared with that of control sample (CO). While with increasing the silica fume content, the pastes for sample B-series grew thinner and their depths increased. They concluded that silica fume makes cement paste thinner as compared with NS.

Table 2.3 Mix proportions, consistency of pastes made of cement and NS and silica fume (Quing et al., 2007)

Sample	Mix proportion in mass					Consistency (mm)
	Cement	Nano silica	Silica fume	Water	SM	
CO	100	0	0	22	2.5	34
A1	99	1	0	22	2.5	34
A2	98	2	0	22	2.5	33
A3	97	3	0	22	2.5	33
A5	95	5	0	22	2.5	32
B2	98	0	2	22	2.5	35
B3	97	0	3	22	2.5	35
B5	95	0	5	22	2.5	36

b. Setting Times

Alshamsi et al. (1993) reported that addition of micro-silica lengthened the setting time of pastes. This was expected since micro-silica replaces part of the OPC, reducing the early stiffening potential. While the addition of micro-silica (10%) had little effect on setting times, higher percentages produced significant influences. There was 6–20% increase in setting times when OPC was replaced with 20% micro-silica.

Lohtia and Joshi (1996) concluded that the addition of silica fume to concrete in the absence of water-reducer or super plasticizer causes delay in setting time, compared to non-silica fume concrete of equal strength, especially when the silica fume content was high. The additions of 5 - 10% silica fume to either super plasticized or non-super plasticized concrete with W/C (SF) ratio of 0.40 did not exhibit any significant increase in setting time. However, when 15% silica fume was added with super plasticizer, both the initial and final setting times were delayed by approximately 1 and 2 h, respectively. The observed delay was attributed to the relatively high dose of super plasticizers needed for the high amount of silica fume added to concrete.

c. Workability

Sellevold et al. (1983) reported that there is net decrease in water requirements in concretes containing high concentration of silica fume and water-reducer or super plasticizers. The addition of water-reducer or a super plasticizer causes the dispersion of cement and silica fume particles and reduces the concentration of contact points between the different grains; resulting in less water requirement to achieve a given consistency.

Khayat and Aitcin (1993) reported that addition of 10% silica fume in a lean concrete (100 kg/m^3) of cement reduced the water demand. However, it exhibited poor durability against freeze–thaw attack. In normal structure concrete, even with 5% silica fume addition, the water demand is increased to maintain constant slump. For producing very high strength and durable concrete, silica fume up to 10% is added as an admixture and use of super-plasticizer to maintain specified slump is found necessary. When no plasticizers are used an additional 1 l/m^3 of water should be used for every 1 kg/m^3 of silica fume addition to maintain constant level of fluidity.

Wong and Razak (2005) studied the cementing efficiency factor (k) of silica fume. Specific gravity of silica fume was 2.22. Three water-to-cementitious material ratios (w/cm) of 0.27, 0.30 and 0.33 were used in concrete mixtures. At each w/c ratio, cement was replaced with 0, 5, 10, and 15% silica fume. Slump and Vebe time results are shown in Table 2.4. It could be seen from this table that mixtures achieved slump values ranging from 30 to 260 mm, while Vebe time was in the range of 1–15 s. The large variation of workability across mixtures was due to the constant super plasticizer dosage used for mixtures with the same w/c ratio.

Table 2.4 Workability characteristics (Wong et al., 2005)

Mixture	w/c	Slump (mm)	Vebe (s)	w/c	Slump (mm)	Vebe (s)	w/c	Slump (mm)	Vebe (s)
C	0.27	165	8	0.30	225	3	0.33	240	1
SF 5		100	8		215	3		180	3
SF 10		50	12		117	5		100	6
SF 15		35	15		30	16		35	16

2.5.5 Effect of Silica Fume on the Hardened Properties of Concrete

a) Compressive strength

Bentur et al. (1989) reported that the strength of silica fume concrete is greater than that of silica fume paste which they attributed to the change in the role of the aggregate in concrete. In cement concrete, the aggregate functions as inert filler but due to the presence of weak interfacial zone, composite concrete is weaker then cement paste. But, in silica fume concrete, the presence of silica fume eliminates this weak link by strengthening the cement paste aggregate bond and forming a less porous and more homogenous microstructure in the interfacial region. Thus, silica fume concrete is stronger than silica fume cement paste, taking into account that the strength of aggregate exceeds the strength of cement paste.

Sobolev (2004) studied the compressive strength of high performance concretes. The compressive results of HPC mixture are shown in Table 2.5. It was observed that:

(i) Increase in super plasticizer dosage from 8 to 18% led to a reduction of w/c from 0.31 to 0.26 and improved the concrete compressive strength from 86 to 97 MPa.

(ii) Maximum compressive strength of 91 MPa was obtained at 15% silica fume.

(iii) Lower strength value of 90 MPa occurred at 10 and 20% silica fume.

(iii) Reduction of w/c 0.32–0.19 increased the compressive strength of cement concrete and resulted in super high strength concrete having strength up to 135 MPa.

Table 2.5 Details of HPC mixtures (Sobolev, 2004)

Proportions (kg/m^3)	SF (5%)	SF (10%)	SF (15%)	SF (20%)
Cement	426	449	449	478
Silica fume	22	50	83	120
Age	Compressive strength (MPa)			
1 day	16.8	24.1	34.4	45.1
3 days	28.6	42.2	63.0	84.9
7 days	50.1	67.2	84.8	102.5
28 days	60.0	80.0	100.0	120.0

Köksal et al. (2008) studied the compressive strength of steel fibre reinforced concrete with silica fume. Cold drawn steel fibres with hooked ends were used. Aspect ratios (l/d) of fibres were 65 and 80 and volume fractions (Vf) of steel fibres were 0.5 and 1%. Silica fume content was 0, 5, 10, and 15% weight of cement. They observed that (i) a considerable increase in the compressive strength of the concretes without steel fibres by increasing the silica fume content. The increases were 12, 73.4 and 85.5% for 5, 10 and 15% silica fume, respectively. These result were clearly dependant on increasing bond strength of cement paste–aggregate interface by means of filling effect of silica fume; and (ii) compressive strengths of concretes produced by additions of both steel fibre and silica fume had higher than the ones containing silica fume only.

b) Tensile Strength

Hooton (1993) reported the splitting tensile strength of silica fume concretes up to the age of 182 days (Table 2.6). It can be seen that except at 28 days, the splitting tensile strength was not improved for silica fume concrete mixes. Also it was observed that with increasing replacement of silica fume split tensile strength decreased.

Table 2.6 Splitting tensile strength of concrete (Hooton, 1993)

Test age (days)	Concrete mixes			
	Control	10% SF	15% SF	20% SF
28	5.2	6.3	6.2	4.6
91	6.8	6.7	6.2	5.6
182	7.1	6.2	6.5	5.6

Tanyildizi and Coskun (2008) studied the effect of silica fume on tensile strength of lightweight concrete exposed to high temperature. In mixtures containing silica fume, 0, 10, 20 and 30% of Portland cement by weight was replaced with silica fume. They reported that the tensile strength started to drop with temperature starting from 2008C. The reduction in splitting tensile strength of lightweight concrete containing 10% silica fume was 3.11, 11.46 and 80.15% at the 200, 400 and 800°C respectively. The reduction in splitting tensile strength of lightweight concrete containing 20% silica fume was 4.69, 12.91 and 78.87% at the 200, 400 and 800°C respectively. And the reduction in splitting tensile strength of lightweight concrete containing 30% silica fume was 5.8, 40.62 and 75.08% at the 200, 400 and 800°C, respectively. They concluded that addition of admixture silica fume prevented the decrease in the tensile strength of concrete.

González and Martinez (2008) studied the properties of concrete using recycled aggregates from Spanish demolition debris (RC mixes) and the impact of the addition of silica fume on the properties of recycled concrete (RCS mixes). A comparison was made between both these materials and standard conventional concrete (CC mixes), which was also modified by adding silica fume (CCS mixes). For the test, they made 10 CC, 7CCS, 10RC and 6RCS mixes. The results of splitting tensile strength are shown in Table 2.7. They stated that neither the addition of recycled aggregates nor the addition of silica fume had any particular impact on the tensile strength of the concrete types. The reason behind this was that silica fume mainly affected mechanical properties like compressive strength of high strength concretes.

Table 2.7 Average tensile splitting tensile strength (MPa) for the various test ages

(Gonzalez et al., 2008)

Cube	Average splitting tensile strength (MPa)		
	7 days	28 days	115 days
CC	3.12	3.15	3.32
CCS	3.04	3.15	3.31
RC	3.17	3.00	3.37
RCS	3.24	3.36	3.35

c) Sulphate Resistance

According to ACI Committee 234, the effect of silica fume on sulphate resistance is due more to the reduction in permeability than to dilution of the C3A content because of the relatively low doses of silica fume used in practice.

Sellevold and Nilsen (1987) reported field studies of concretes with and with out 15% silica fume. After 20 years' exposure to ground water containing 4 g/L sulphate and 2.5–7.0 pH, the performance of the silica fume concrete was found equal to that of the concretes made with sulphate-resisting Portland cement, even though the water/cementitious materials ratio was higher for silica fume concrete (0.62) than for control (0.50).

Cohen and Bentur (1988) studied the effect of 15% silica fume replacement of Types I and V Portland cement on the resistance to sulphate attack in magnesium and sodium sulphate solutions. The water–cementitious materials ratio was 0.3. In the sodium sulphate solutions, the silica fume concrete specimens were resistant to sulphate attack. In the magnesium sulphate solutions, all the specimens expanded, with the Type I cement specimens (with or without silica fume) expanding more than Type V cement specimens (with or without silica fume). Since specimens were thin (6 mm), the authors attributed the effect of silica fume on sulphate resistance more to chemical effects than to reduced permeability.

Shannag et al. (2003) prepared high-performance concrete mixes containing various proportions of natural pozzolan and silica fume (up to 15% by weight of cement). They were stored in sodium and magnesium sulphate solutions, in Dead Sea and Red Sea waters. After 1 year immersion in sulphate solution and sea water, the concrete mix containing a combination of 15% silica fume, and 15% natural pozzolan (by weight of cement) showed a maximum protection against sulphate attack compared to those investigated in the study. This mix retained more than 65% of its strength after 1 year of storage in sulphates solutions and sea waters. The superior resistance of that mix against sulphate attack was attributed to the pore refinement process and further densification of the transition zone occurring due to the conversion of lime forming from the hydration of cement into additional binding material through lime-pozzolan reaction. The results also showed that magnesium sulphates had a more damaging effect than sodium sulphates; which was consistent with the data available in the literature.

Lee et al. (2005) studied the effectiveness of silica fume in controlling the damage arising from sulphate attack. The water/cementitious materials ratios (w/cm) of the mortar mixtures were 0.35, 0.45 and 0.55. Under this sulphate environment, the incorporation of 10% silica fume in OPC matrix showed no evidence of spalling and cracking up to about 1 year of exposure, and strength loss increased as the w/cm ratio increased; and the total strength loss as well as that between different w/cm ratio levels was greater in mortar specimens without silica fume compared to those with silica fume.

Irassar et al. (1996) investigated the sulphate resistance of concrete made with silica fume. Concrete specimens were half-buried in sulphate soil for 5 years. Mineral admixtures were used as a partial replacement for ordinary Portland cement (C3A = 8.5%), and the progress of sulphate attack was evaluated by several methods (visual rating, loss in mass, dynamic modulus, strength, X-ray analysis). Results showed that silica fume improved the sulphate resistance when the concrete was buried in the soil. However, concretes with high content of silica fume exhibited a greater surface scaling over soil level due to the sulphate salt crystallization.

2.6 Metakaolin

Metakaolin (MK) is a pozzolanic material. It is obtained by calcinations of kaolinitic clay at a temperature between 500°C and 800°C. The raw material input in the manufacture of metakaolin ($Al_2Si_2O_7$) is kaolin clay. Kaolin is a fine, white, clay mineral that has been traditionally used in the manufacture of porcelain. Kaolinite is the mineralogical term that is applicable to kaolin clays.

Kaolinite is defined as a common mineral, hydrated aluminium disilicate, the most common constituent of kaolin. The Meta prefix in the term is used to denote change. In the case of metakaolin, the change that is taking place is dehydroxylization, brought on by the application of heat over a defined period of time. The behaviour of clay minerals on heating depends on their structure, crystal size and degree of crystallinity. At just above 100, clay minerals lose most of their adsorbed water. The temperature at which kaolinite loses water by de-hydroxilization is in the range of 500 – 800ºC. This thermal activation of a mineral is also referred to as calcining. Beyond the temperature of dehydroxylization, kaolinite retains two dimensional order in the crystal structure and the product is termed as metakaolin. The key in producing metakaolin for use as a supplementary cementing material, or pozzolans is to achieve as near as possible complete dehydroxylization without over-heating.

Successful processing of kaolinite results in a disordered, amorphous state, which is highly pozzolanic. Thermal exposure beyond a defined point will result in sintering and the formation of mullite, which is dead burnt and not reactive. In other words, kaolinite, to be optimally altered to a metakaolin state, requires that it is thoroughly roasted but never burnt.

Metakaolin is one of the innovative clay products developed in recent years. It is produced by controlled thermal treatment of kaolin. Metakaolin can be used as a concrete constituent, replacing part of the cement content since it has pozzolanic properties. The use of metakaolin as a partial cement replacement material in mortar and concrete has been studied widely in recent years. Despite of the recent studies, there are still many unknowns with the use of metakaolin. Study is needed to determine the contribution of metakaolin to the performance of hardened concrete. If it is proven that the concrete is durable and strong, this will lead to the use of metakaolin to replace part of the cement.

2.6.1 Uses of Metakaolin

- High performance, high strength and lightweight concrete
- Precast concrete for architectural, civil, industrial, and structural purposes
- Fiber cement and ferrocement products
- Glass fiber reinforced concrete
- Mortars, stuccos, repair material, pool plasters
- Improved finishability, colour & appearance

2.6.2 Advantages of Using Metakaolin

- Increased compressive and flexural strengths
- Reduced permeability
- Increased resistance to chemical attack
- Increased durability
- Reduced effects of alkali-silica reactivity (ASR)
- Reduced shrinkage due to particle packing, making concrete denser
- Enhanced workability and finishing of concrete
- Reduced potential for efflorescence

2.6.3 Properties of Metakaolin

Physical Properties

Metakaolin is 99.9% finer than 16 µm, and has a mean particle size of 3 µm (as measured by MicroTrac laser diffraction granulometer method (www.metakaolin.com). Some physical properties of metakaolin are given in Table 2.8.

Table 2.8 Physical properties of metakaolin (www.metakaolin.com)

Property	Value
Specific gravity	2.60
Bulk density (gm/cm^3)	0.3 – 0.4
Physical form	Powder
Colour	Off – white
GE Brightness	79 - 82

Chemical Composition

Major constituents of metakaolin are SiO_2 and Al_2O_3. Typical chemical composition as reported by (Ambroise et al., 1994) is given in Table 2.9. Metakaolin must meet the requirements of ASTM C 618, Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete," Class N, with the following modifications.

Table 2.9 Typical chemical composition of Metakaolin (Ambroise et al., 1994)

Item	% By weight
SiO_2 (%)	51.52
Al_2O_3 (%)	40.18
Fe_2O_3 (%)	1.23
Cao (%)	2.0
MgO (%)	0.12
Na_2O (%)	0.08
K_2O (%)	0.53
Loss on ignition (%)	0.91

2.6.4 High Reactivity Metakaolin (HRM):

High Reactivity Metakaolin (HRM) is a refined form of an ASTM C 618, Class N (natural) pozzolan that is produced by calcining purified kaolinite clay at a specific temperature range. The particle size of HRM is significantly smaller than cement particles, yet not as fine as silica fume. It has the highest content of siliceous and aluminous material among the purified kaolin. This is because at least 90% of the product consists of silica and alumina.

The HRM is manufactured under controlled conditions to provide consistent product in terms of particle size distribution, surface area, and colour and chemical composition. Unlike industrial by products, such as silica fume, fly ash, and blast-furnace slag, HRM is water-processed to lighten its colour, remove inert impurities, and control particle size distribution. The carefully controlled refining process results in an almost 100% reactive white powder that is consistent in appearance and performance from lot to lot. The HRM by virtue of its highest purity is white in colour, which is critical to architectural applications. HRM shows promise as a mineral admixture for high-performance concrete. Because of its white colour, HRM does not darken concrete as silica fume does. This makes it ideal for colour matching and other architectural applications as it produces concrete similar in colour to conventional exposed concrete.

HRM is thermally activated such that the heat treatment results in maximum pozzolanic activity for the product. Typically added to concrete at rates of 5% to 10% by weight of cement, HRM improves concrete performance by combining chemically with free lime (by-product Portland-cement hydration) to form additional cementitious materials. It is proven that the compressive and flexural strength development of HRM mixes is significantly greater than that of a non pozzolanic control mixture and similar to that of silica-fume mixtures. Some of the benefits of the HRM concrete are:

i. Requires 25% to 35% less super plasticizer than silica-fume concrete to achieve a comparable slump (at water- cementitious materials ratios above 0.35).

ii. Has very low chloride permeability, similar to that of silica fume concrete.

iii. Exhibits less drying shrinkage than conventional Portland-cement concrete, and drying shrinkage similar to that of silica-fume concrete.

2.6.5 Effect of Metakaolin on Fresh Properties of Concrete

Wild et al. (1996) studied the workability of metakaolin concretes. Replacement levels of ordinary Portland cement (OPC) by metakaolin (MK) were 0, 5, 10, 15, 20, 25, and 30% and water-binder ratio (w/b) was 0.45. OPC concrete mixture proportion was 1:2.3:3.4.Workability results (slump, compacting factor and vebe time) are given in Table 2.10.

Table 2.10 Workability of metakaolin concretes (Wild et al., 1996)

Metakaolin (%)	Superplasticizer (%)	Slump (mm)	Compacting factor	Vebe time (sec)
0	0	5	0.81	26
5	0.6	10	0.84	15
10	1.2	15	0.88	10
15	1.8	25	0.89	9
20	2.4	75	0.89	7
25	3.0	75	0.89	4
30	3.6	90	0.90	5

Brooks and Johari (2001) reported the slump and setting times of concretes containing 0, 5, 10, and 15% metakaolin (MK). Control concrete mixture proportion was 1:1.5:2.5 with water-binder ratio of 0.28. Slump and setting times results are given in Table 2.11. It can be seen that slump decreased and setting times increased with the increase in MK content.

Table 2.11 Workability, setting times of MK concretes (Brooks and Johari, 2001)

Concrete mixes	Slump (mm)	Initial setting time (hours)	Final setting time (hours)
OPC	100	5	7.7
MK5	30	6.42	8.82
MK10	20	6.98	9.42
MK15	5	6.45	9.31

Li and Ding (2003) investigated the fluidity of Portland cement containing metakaolin (MK) and ultra-fine slag (S). Four series of pastes were made. First series contained 100% cement, 0% MK, and 0% slag; second had 90% cement and 10% MK, 0% slag; third had 70% cement, 10% MK, and 20% slag, and the fourth series had 60% cement, 10% MK, and 30% slag. Description of Physical properties of cement paste is given in Table 2.12.

(i) After incorporation of MK into PC, the fluidity of MK blended cement became poorer than that of PC at the same dosage of super plasticizer and the same water/binder ratio.

(ii) Incorporation of 10% MK and 20% or 30% ultra-fine slag jointly into PC, improved the fluidity of blended cements, also the 28-day compressive strength.

(iii) Both MK and slag can react with CH released by cement clinker hydration to produce secondary C-S-H gel inside the cement paste. The secondary formed C-S-H gel improved the microstructure of cement paste matrix.

(iv) XRD analysis indicated that more $Ca(OH)_2$ was consumed after adding both mineral admixtures.

Table 2.12 Physical properties of cement paste (Li and Ding, 2003)

Mixture type	Composition			Water required for standard consistency (%)	Setting times (hours: mins)	
	Cement	MK	Slag		Initial	Final
PO	100	0	0	26.3	1:36	3:27
M1	90	10	0	29.1	1:11	2:12
S2	70	10	20	28.4	1:55	4:34
S3	60	10	30	28.0	2:19	6:15

Bai et al. (2003) used neural networks to predict workability of concrete incorporating metakaolin and fly ash. The predictions reflected the effect of graduated variations in pozzolanic replacement in Portland cement up to 15% MK and 40% FA. The results showed that the models are reliable and accurate and illustrate how neural networks can be used to beneficially predict the workability parameters of slump, compacting factor and Vebe time across a wide range of PC-FA-MK compositions. On the basis of the models developed, effects of MK and FA on workability were analyzed and iso - slump maps were plotted for w/b 0.25, 0.4 and 0.5. Through this, it is possible for the designer to produce mixtures with various blend compositions for a given range of slumps.

Li and Ding (2003) investigated the consistency and setting times of Portland cement containing metakaolin (MK) and ultra-fine slag (S). Four series of pastes were made. First series contained 100% cement, 0% MK, ad 0% slag; second had 90% cement and 10% MK, 0% slag; third had 70% cement, 10% MK, and 20% slag, and the fourth series had 60% cement, 10% MK, and 30% slag. Test results of water consistency and setting times are presented in Table 2.12. They concluded that:

(i) Standard consistency of cement paste was in the range of 26.3–29.1%. Mixture PO had the lowest water requirement, but MK blended cement M1 had the highest Water requirement. After the slag was mixed with MK blended cement, the water requirement of cement mixture S2 and S3 was reduced, but it was still higher than that of PC.

(ii) Initial and final setting times of the cement incorporating 10% MK were shorter than those of PC, while the initial and final times of S2 and S3 cements were longer than those of PC.

Badogiannis et al. (2005) reported the results of water demand and setting times of cements containing five metakaolins. The metakaolinite contents in metakaolins MK1, MK2, MK3, and MK4 (derived from poor Greek kaolins) were 36, 37, 71, and 49%, respectively, where as it was 95% in a commercial metakaolin (MKC) of high purity. Cement was replaced with 0, 10, and 20% Metakaolin. They concluded that:

i) Blended cements demanded significantly more water than the relatively pure cement. With a metakaolin content of 10%, the water demand varied from 29.0% to 32.5%, while the PC had a water demand of 27.5%. With a metakaolin content of 20%, the water demand varied from 31.5% to 41.5%. Metakaolins MK1 and MK2 showed the best behaviour and increase in water demand was less. The increase in water demand could be attributed to the high fineness of metakaolin as well as to their narrow particle size distribution.

ii) The initial and final setting time of metakaolin cements was affected by the metakaolin content. Cements with 10% metakaolin, generally, exhibited similar setting times to that of PC, while for 20% metakaolin content there was a delay in the setting. MK4 showed the greatest effect on the setting delay of the cements.

2.6.6 Effect of Metakaolin on the Properties of Hardened Concrete

a) Compressive Strength

Poon et al. (2001) showed that the cement pastes containing 5% – 20% MK had higher compressive strengths than the control at all ages from 3 to 90 days, with the paste containing 10% MK performing the best. The cement pastes containing SF or FA had lower compressive strength than the control at early ages. The SF replacements resulted in higher compressive strengths than the control at 28 and 90 days, while the FA replacement resulted in a higher compressive strength than the control only at 90 days. The above results indicated that at early ages, the MK contributed better to the compressive strength development of high-performance cement pastes than the SF.

Roy (2001) studied the effect of silica fume (SF), metakaolin (MK), and low-calcium fly ash on compressive strength of concrete. They reported that compressive strength increased on addition of MK. SF also increased compressive strength but lesser than MK. Similarly, FA also increased compressive strength but to a lesser magnitude. No significant change in compressive strength was found as a function of replacement level for SF and MK series. However, the compressive strength decreased as the FA replacement level increased. This clearly showed that low-calcium fly ash is less reactive than SF and MK and needs an external activation.

Wild et al. (1996) investigated the effect of varying percentages of metakaolin on the compressive strength of concrete mixtures. Replacement levels of ordinary Portland cement (OPC) by metakaolin (MK) were 0, 5, 10, 15, 20, 25, and 30% and water-binder ratio (w/b) was 0.45. OPC concrete mixture proportion was 1:2.3:3.4. Compressive strength tests were conducted up to the age of 90 days, and results are presented in Table 2.13. They concluded that inclusion of MK as partial replacement of cement enhanced the compressive strength of concrete at all ages, but the optimum replacement level of OPC by MK to give maximum long term strength enhancement was about 20%.

Table 2.13 Compressive strengths and densities of metakaolin concretes (Wild et al., 1996)

MK (%)	Density (kg/m^3)	Compressive strength (MPa)				
		1 day	7 days	14 days	28 days	90 days
0	2490	19.07	50.23	57.10	62.60	72.43
5	2440	21.50	53.80	58.97	63.50	71.63
10	2460	22.43	62.30	69.23	71.00	80.07
15	2470	20.23	64.80	74.67	76.00	83.70
20	2480	19.33	66.47	75.73	82.47	85.13
25	2470	15.73	62.50	69.77	73.93	82.23
30	2480	14.53	60.53	72.33	76.73	81.80

Curcio et al. (1998) examined the role of metakaolin as pozzolanic microfiller for high-performance mortars. They characterized super-plasticized mortars containing metakaolin (MK) as 15% replacement of cement and with a water/binder ratio of 0.33. Four commercially available MK samples and one silica fume sample were studied for comparison. They concluded that:

(i) Specimens containing three of the four metakaolin samples had a higher rate of compressive strength development as compared to that of the control at ages below 28 days, a consequence of the higher hydration rate. Silica fume and the fourth and coarser metakaolin had a less pronounced effect.

(ii) At 90 and 180 days metakaolin and silica fume specimens gave similar strengths. The difference in the compressive strength between the specimens with

> microfillers and the control decreased after 28 days, because of a smaller slowdown of the hydration rate in the control.

DTA calcium hydroxide peak heights on mortars and the pozzolanic activity test on mixtures of 80% cement and 20% microfiller showed that three of the four metakaolins had a remarkable pozzolanic activity comparable to that of silica fume, even if slightly lower at longer ages. The fourth and coarser metakaolin sample was less reactive. The amount of water that was lost after 24 h at 90◦C was different for all types of specimens and was dependent on the differences of the hydration products formed and on the permeability of the materials. It can be related to the fineness of the microfiller in the specimens with metakaolin.

Brooks and Johari (2001) determined the compressive strength of concretes containing 0, 5, 10, and 15% metakaolin (MK). Mixture proportion of OPC concrete was 1:1.5:2.5 with a water-to-cement ratio of 0.28. 28-day Compressive strength results of MK concretes are given in Table 2.14. It is evident from the table that compressive strength increased with the increase in the metakaolin content.

Table 2.14 Compressive strengths of MK concrete mixes (Brooks and Johari, 2001)

Concrete mixes	Compressive strength (MPa)
OPC	87.0
MK5	91.5
MK10	104.0
MK15	103.5

Bai et al. (2002) reported the influence of the composition of Portland cement– pulverized fuel ash–metakaolin binders on strength development of PC–PFA–MK concrete cured both in air and water. Concrete mixtures were made with four different total cement replacement levels (10, 20, 30 and 40%) for PC–PFA–MK concrete with various MK/PFA proportions and cured in water and air up to 18 months.

In water-cured concrete made with PC–PFA–MK binder, the MK enhanced early (28 days) strength, and PFA retarded early strength. Air-cured concrete showed a loss in strength relative to equivalent concrete that was water cured and the strength difference increased with curing period. The difference was enhanced in concrete made with PC–PFA binder at high replacement levels, which showed a much reduced strength gain with time when air cured, whereas for PC–PFA–MK concrete, this difference was reduced as the MK content increases.

Jin and Li (2003) investigated the effect of mineral admixtures such as silica fume, slag, fly ash, and metakaolin on the mechanical behaviour of young concrete under either uniaxial compression or tension. Tests were conducted at ages of 1/2, 1, 2, 3, 7, and 28 days. They reported that:

i) Stress-strain (deformation) curves for young concretes were different from mature concretes in their capability in resisting the deformation. More ductile behaviour was observed for a young concrete. However, the complete stress-deformation curves for young concrete under uniaxial tension showed less ductility as compared to those under uniaxial compression.

ii) Incorporation of metakaolin, silica fume, and slag into concrete can prompt both the strength and modulus of elasticity development of a young concrete.

iii) Metakaolin showed the best enhancement on the mechanical properties of young concrete. The reactivity of slag modified concrete was speeded up after one day.

b) Tensile Strength

Qian and Li (2001) investigated the tensile strength of concrete incorporating 0, 5, 10, and 15% metakaolin as partial replacement of cement. Metakaolin had specific surface area 12000 m^2/kg, and its average particle diameter was 2.23 µm. 300×100×20 mm samples were tested under direct tension. Tests were conducted at the age of 28 days. Tensile strength test results are presented in Table 2.15. The results showed that tensile strength of concrete increased systematically with increasing metakaolin replacement level. The average tensile strength increases were 7% (5% metakaolin), 16% (10% metakaolin), and 28% (15% metakaolin), and the average ultimate strain increases were 3% (5%metakaolin), 19% (10% metakaolin), and 27% (15% metakaolin). The descending area of over-peak stress was less steep when metakaolin replacement was 5% and 10% whereas with 15% metakaolin it was similar to that for concrete without metakaolin. The tensile elasticity modulus for these specimens is in the range from 26 to 27 GPa.

Table 2.15 Tensile strength of concrete with different metakaolin replacements

(Qian and Li, 2001)

Age (days)	Tensile strength (MPa)			
	MK (0%)	MK (5%)	MK (10%)	MK (15%)
28	3.35	3.58	3.88	4.29

c) Sulphate Resistance

Sulphate attack is one of the most aggressive environmental deteriorations that affect the long-term durability of concrete structures. The sulphate attack on concrete leads to expansion, cracking, and deterioration of many civil engineering structures exposed to sulphate environment such as piers, bridges, foundations, concrete pipes, etc. The sulphate ions in solution, which come from the soil, ground water, and seawater, are found in combination with other ions such as sodium, potassium, magnesium and calcium ions. The sulphate attack is generally attributed to the reaction of sulphate ions with calcium hydroxide and calcium aluminate hydrate to form gypsum and ettringite. The gypsum and ettringite formed as a result of sulphate attack is significantly more voluminous (1.2–2.2 times) than the initial reactants. The formation of gypsum and ettringite leads to expansion, cracking, deterioration, and disruption of concrete structures. In addition to the formation of ettringite and gypsum and its subsequent expansion, the deterioration due to sulphate attack is partially caused by the degradation of calcium silicate hydrate (C–S–H) gel through leaching of the calcium compounds. This process leads to loss of C–S–H gel stiffness and overall deterioration of the cement paste matrix.

Khatib and Wild (1998) evaluated the effect of metakaolin on the sulphate resistance of mortar. Two types of cements were used; high C3A and intermediate C3A content. Cement was replaced with 0, 5, 10, 15, 20, and 25% of metakaolin (MK). Specimens of size 150 × 150 × 150 mm were moist-cured in air for 14 days, and their length was measured before immersing in 5% Na2SO4 solution. Test results demonstrated that:

(i) Expansion decreased systematically with the increase in MK content for both types of cement.

(ii) For mortars containing high C3A cement, the control mortar and mortars containing 5% and 10% MK showed rapid expansion and deterioration between 40 and 70 days exposure to sulphate solution. Mortars containing 15% and 20% MK showed small but quite sharp expansions also between 40 and 70 days, but subsequently stabilized. By 520 days, mortar containing 20% and 25% MK showed no significant changes in length.

(iii) With intermediate C3A content in cement, trend was similar to the mortars made with high C3A content. The control mortar (0% MK) showed significant expansion followed by 5% MK mortar. Mortar containing 10% MK exhibited little expansion and other mortar specimens containing 15, 20 and 25% were found to be extremely stable, showing little overall change and no sign of deterioration after 520 days of exposure in sodium sulphate solution.

Lee et al. (2005) reported an experimental study on the magnesium sulphate resistance of mortar and paste specimens incorporating varying percentages of metakaolin (MK). The resistance of mortar specimens was evaluated by expansion measurements. Four different mortar mixtures were prepared. Cement was partially replaced with 0, 5, 10 and 15% of MK by mass. The water–cementitious material ratio (w/cm) was fixed at 0.45 by mass. The cementitious material/fine aggregate was invariant at 2.0 in all mortar mixtures. Prisms (25 × 25 × 285 mm) were cast for expansion measurements. All mortar and paste samples were immersed in magnesium sulphate solution (MgSO_4) for 360 days. Sulphate solution concentrations used were 0.42% (3380 ppm of $SO_4{}^{2-}$), 1.27% (10,140 ppm of $SO_4{}^{2-}$) and 4.24% (33,800 ppm of $SO_4{}^{2-}$respectively. Test results of expansion of mortars at 360 days are shown in fig. 2.4.

They concluded that:

(i) At low solution concentration (0.42%) of magnesium sulphate (MgSO4), only the MK5 mortar specimen showed an expansion value of over 0.1%.

(ii) At magnesium sulphate solution concentration of 1.27%, the mortar specimens with 5% to 15% replacement levels of MK exhibited poor resistance to magnesium sulphate attack. After 360 days of exposure, expansion values were 0.343, 0.339 and 0.469% for MK5, MK10 and MK15 mortar specimens, respectively, whereas the MK0 mortar specimens exhibited about 0.185% expansion after the same period of exposure.

(iii) At high solution concentration of magnesium sulphate (4.24% MgSO4), the expansion values for the MK0 mortar was about 0.244% after 360 days of exposure, whereas mortar specimens with partial replacement levels of MK showed expansions of 0.360%, 0.410% and 0.556% for MK5, MK10 and MK15 mortar mixtures, respectively.

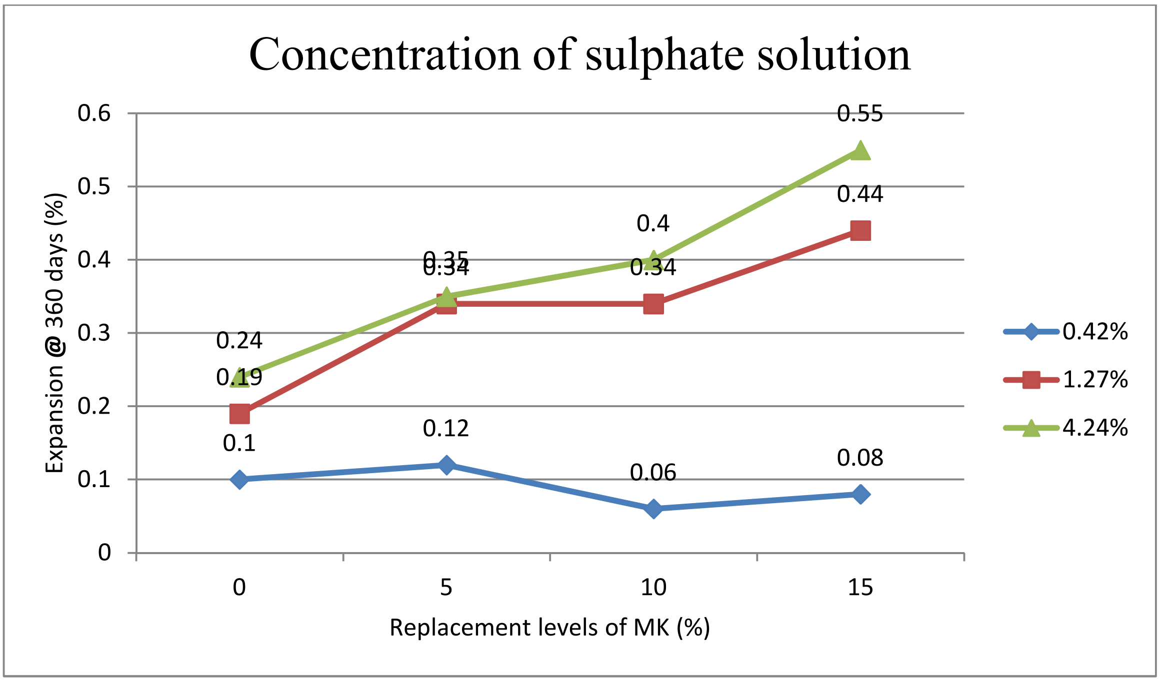

Fig. 2.4 Effect of solution concentrations and MK replacement levels on the expansion of mortar specimens exposed to magnesium sulphate solution (Lee et al., 2005)

CHAPTER 3

EXPERIMENTAL PROGRAMME

3.1 Aim of the Experimental Programme

The aim of the experimental programme is to compare the properties of concrete incorporating foundry sand mixed with and without silica fume and Metakaolin, used as supplementary cementing materials. The various tests performed on concrete samples are discussed in this chapter, followed by a brief description about mixture proportion design and curing procedure adopted. Following concrete properties have been discussed: compressive strength, splitting tensile strength and sulphate resistance of concrete when exposed to a sulphate solution.

3.2 Materials Used

3.2.1 Cement

In this work J.K. cement of 43 grade was used for casting cubes and cylinders for all concrete mixes. The cement was of uniform colour i.e. grey with a light greenish shade and was free from any hard lumps. The various tests conducted on cement are initial and final setting time, specific gravity, fineness and compressive strength. Testing of cement was done as per IS: 8112-1989. The various tests results conducted on the cement and composition limits of Portland cement are reported in Table 3.1, Table 3.2 and Table 3.3 respectively.

Table 3.1 Physical properties of cement

Characteristics	Values obtained	Standard value
Initial setting time (minutes)	42 min.	Not be less than 30 minutes
Final setting time (minutes)	310 min.	Not be greater than 600 minutes
Fineness (%)	4.9 %	< 10
Specific gravity	3.09	--

Table 3.2 Compressive strength of cement

Days	Specimen	Compressive Strength (N/mm^2)	Average Compressive Strength (N/mm^2)
7	1	35.2	35.3
	2	36.1	
	3	34.7	
28	1	42.8	44.1
	2	44.1	
	3	45.3	

Table 3.3 Composition of Portland cement (IS: 8112-1989)

Ingredient	% Content
Lime	60 – 67
Silica	17 – 25
Alumina	3 – 8
Iron oxide	0.5 – 6
Magnesia	0.1 – 4
Alkalies	0.4 – 1.3
Sulphur	1.3

3.2.2 Coarse aggregate

Locally available coarse aggregates having the maximum size of 10 mm and 20mm were used in the present work. Testing of coarse aggregates was done as per IS: 383-1970. The 10mm aggregates used were first sieved through 10mm sieve and then through 4.75 mm sieve and 20mm aggregates were firstly sieved through 20mm sieve. They were then washed to remove dust and dirt and were dried to surface dry condition. The results of various tests conducted on coarse aggregate are given in Table 3.4, Table 3.5 & Table 3.6.

Table 3.4 Physical properties of coarse aggregate

Characteristics	Value
Type	Crushed
Maximum size	20 mm
Specific gravity (10 mm)	2.704
Specific gravity (20 mm)	2.825
Total water absorption (10 mm)	1.6432 %
Total water absorption (20 mm)	3.645 %
Moisture content (10 mm)	0.806 %
Moisture content (20 mm)	0.7049 %
Fineness modulus (10 mm)	6.46
Fineness modulus (20 mm)	7.68

Table 3.5 Sieve Analysis of 10mm Aggregates

Sieve no.	Mass retained (kg)	% retained	5 passing	Cumulative 5age retained
80 mm	-	0.00	100	0.00
40 mm	-	0.00	100	0.00
20 mm	-	0.00	100	0.00
12.5 mm	0.555	18.5	81.5	18.5
10 mm	0.8905	29.68	51.82	48.18
4.75 mm	0.9565	31.88	19.94	80.06
Pan	0.5970	19.90	0.04	99.96
			ΣC =	146.74

Fineness Modulus of Coarse aggregate (10 mm) = ΣC+500/100 = 146.74+500/ 100 = 6.46.

Table 3.6 Sieve analysis of 20 mm aggregates

Sieve no.	Mass retained (kg)	% retained	% passing	Cumulative %age retained
80 mm	-	0.00	100	0.00
40 mm	-	0.00	100	0.00
20 mm	0	0.00	100	0.00
12.5 mm	2.1865	72.883	27.117	72.883
10 mm	0.6745	22.483	4.634	95.366
4.47 mm	0.1390	4.633	0.01	99.999
Pan	0	0.00	-	-
			ΣC =	268.244

Fineness Modulus of Coarse aggregate (20mm)= ΣC+500/100 = 268.244+500/ 100 = 7.68.

3.2.3 Fine Aggregate

The sand used for the experimental programme was locally procured and conformed to grading zone III as per IS: 383-1970. The sand was first sieved through 4.75 mm sieve to remove any particles greater than 4.75 mm and then was washed to remove the dust. Physical properties and sieve analysis results of fine aggregates are listed in Table 3.7 and Table 3.8 respectively.

Table 3.7 Physical properties of fine aggregates

Characteristics	Value
Type	Uncrushed (natural)
Specific gravity	2.68
Total water absorption	1.02 %
Moisture content	0.16 %
Net water absorption	0.86 %
Fineness modulus	2.507
Grading zone	Zone III

Table 3.8 Sieve analysis of fine aggregate

	Retained, (gms)	Retained, %	Passing, %	%age Retained
4.75 mm	95.0	9.5	90.5	9.5
2.36 mm	42.5	4.25	86.25	13.75
1.18 mm	110.5	11.05	75.2	24.8
600 m	128.5	12.85	62.35	37.65
300 m	308.0	30.8	31.55	68.45
150 m	281.0	28.1	3.45	96.55
Pan	34.5	3.45	-	
			ΣF =	250.7

Total weight taken = 1000gms

Fineness Modulus of fine aggregate = ΣF/100 = 250.7/100 = 2.507

3.2.4 Foundry Sand

Investigations were made on foundry sand procured from Insaf Foundries, Mandi Gobindgarh, Punjab. The physical and chemical properties of the foundry sand used in this investigation are listed in Table 3.9 and Table 3.10 respectively. Table 3.11 shows the sieve analysis for 10% replacement level of sand with foundry sand.

Table 3.9 Physical Properties of Foundry Sand

Property	Results	Test method
Specific gravity	2.47	ASTM D854
Bulk relative density, kg/m^3	2589	ASTMC48/ AASTHO T84
Absorption, %	0.45	ASTM C128
Moisture content, %	0.1 – 10.1	ASTM D2216
Clay lumps and friable particles	1 – 44	ASTM C142/ AASTHO T112
Coefficient of permeability (cm/sec)	$10^{-3} – 10^{-6}$	AASTHO T215/ ASTM D2434
Plastic limit/ plastic index	Non plastic	AASTHO T90/ ASTM D4318

Table 3.10 Foundry sand sample chemical composition

Constituent	Value (%)
SiO_2	87.91
Al_2O_3	4.70
Fe_2O_3	0.94
CaO	0.14
MgO	0.30
SO_3	0.09
Na_2O	0.19
K_2O	0.25
TiO_2	0.15
P_2O_5	0.00
Mn_2O_3	0.02
SrO	0.03
LOI	5.15
TOTAL	99.87

Table 3.11 Sieve analysis of Fine Aggregates at 10% replacement level

Sieve no.	Mass retained (gms)	% Retained	% Passing	Cumulative %age retained
4.75 mm	37.0	3.7	96.3	3.7
2.36 mm	10.5	1.05	95.25	4.75
1.18 mm	43.0	4.3	90.95	9.05
600 m	67.5	6.75	84.2	15.8
300 m	23.90	2.390	60.3	39.7
150 m	54.90	5.490	5.4	94.6
Pan	5.15	0.515	0.25	99.75
				ΣF =267.35

Fineness Modulus of fine aggregates with 10% foundry sand = ΣF/100 =267.35/100 = 2.6.

3.2.5 Silica fume

Densified silica fume obtained from Mehtab Pvt. Limited, Chandigarh, is used in this work. It was grey in colour. It is shown below in Fig. 3.1.

Fig. 3.1 Silica Fume mixed with cement

3.2.6 Metakaolin

The metakaolin used in this study is Metacem of grade 85-C, obtained from Iliyas chemicals, New Delhi. It was pinkish white in colour and is shown below in Fig. 3.2.

Fig. 3.2 Metakaolin

3.2.7 Water

Potable tap water was used for the concrete preparation and for the curing of specimens.

3.2.8 Admixture

Admixture used in this work is Conplast SP430, which complies with IS-9103:1979 and BS:5075 Part 3 and ASTM-C-494 type ‘F’ as a high range water reducing admixture. Conplast SP 430 is a ready to use admixture that is added to the concrete at the time of batching. Conplast SP430 is differentiated from conventional superplasticizers in that it is based on aqueous solution of lingo sulphonates, organic polymer with long lateral chains. This greatly improves cement dispersion. Conplast SP430 is supplied as brown liquid instantly dispersible in water and specially formulated to give high water reduction up to 25% without loss of workability. Specific gravity is 1.22 to 1.225 at 30º C.

3.2.9 Magnesium Sulphate

Powder form of magnesium sulphate was obtained from Scientific Junction, Patiala. It was white in colour. Its solution of strength 5% by adding it to water was made and used for sulphate resistance test.

Fig. 3.3 Magnesium Sulphate

3.3 Batching, Mixing and Casting of Specimens

Cubical moulds of size 150mm^3 were used to prepare the concrete specimens for the determination of compressive strength of concrete. Care was taken during casting and vibrator was used for proper compaction. Cylindrical moulds of size 150 mm × 300 mm were used to prepare the concrete specimens for the determination of split tensile strength of concrete. All the specimens were prepared in accordance with Indian Standard Specifications

IS: 516-1959. All the moulds were cleaned and oiled properly. These were securely tightened to correct dimensions before casting. Care was taken that there is no gaps left from where there is any possibility of leakage of slurry.

A careful procedure was adopted in the batching, mixing and casting operations. The coarse aggregates and fine aggregates were weighed first with an accuracy of 0.5 grams. The concrete mixture was prepared by hand mixing on a non-absorbing platform. PPC having 43 grades was used in casting. Fine aggregates are replaced with 10% foundry sand and thoroughly mixed. After that coarse aggregates are added to it. Super plasticizer as per requirement was added to required quantity of water separately in different containers. Then water was added carefully so that no water was lost during mixing. For each mix 27 samples were prepared which consists of 9 cubes ($150 \times 150 \times 150$mm) for 7, 28 and 56 days compressive strength, 9 cubes ($150 \times 150 \times 150$mm) for 7, 28 and 56 days sulphate resistance test and 9 cylinders (300 × 150mm) for split tensile strength at 7, 28 and 56 days. Vibrations were stopped as soon as the cement slurry appeared on the top surface of the mould. The specimens were allowed to remain in the steel mould for the first 24 hours at ambient condition. After that these were de-moulded with care so that no edges were broken and were placed in the curing tank at the ambient temperature for curing. The ambient temperature for curing was 27 ± 20°C. (IS: 10262-1982)

3.4 Experimental Procedure

3.4.1 Testing the compressive strength and splitting tensile strength of concrete

Firstly, measured quantity of 20mm and 10mm coarse aggregates are mixed together. Ordinary sand is replaced with 10% waste foundry sand on a separate platform which is kept constant in all the mixes, and then fine aggregates are mixed with coarse aggregates. Cement mixed with desired silica fume content is then added to the mixture. Super plasticizer SP - 430 is added to the water of desired quantity and this water is then finally added to the mix. This final concrete mixture was then put into properly oiled and fitted steel moulds (cubes of $150mm^3$) in layers and then placed on a vibrator for proper settlement of concrete. After the cubes were casted, they were left for 24 hours in normal room temperature before de moulding. And finally, the cubes were placed in the water curing tank and taken out after 7, 28 and 56 days respectively to get the compressive strength test readings for each mix.

3.4.2 Testing the Sulphate resistance of concrete

For this, 24 cubes each of size $150mm^3$ were prepared (3 cubes for each mix) for testing the decrease or increase in compressive strength due to external sulphate attack on the concrete specimen. The cubes were first water cured for 28 days after de-moulding. Then after 28 days of curing, the cubes were immersed in 5% $MgSO_4$ solution for 7, 28 and 56 days respectively. Finally the compressive strengths of these cubes were examined at 7, 28 and 56 days of immersion period.

3.5 Mixture proportioning

Table 3.12 Various Mix proportions

Mixture	C.M	C.M1	M1	M2	M3	M4	M5	M6
Cement kg/m^3	372	372	353	335	316	353	335	316
Silica fume (%)	0	0	5	10	15	-	-	-
Silica fume kg/m^3	0	0	19	37	56	-	-	-
Metakaolin (%)	0	0	-	-	-	5	10	15
Metakaolin (kg/m^3)	0	0	-	-	-	19	37	56
Sand (kg/m^3)	539	485	485	485	485	485	485	485
Foundry sand (%)	0	10	10	10	10	10	10	10
Foundry sand kg/m^3	0	54	54	54	54	54	54	54
C.A 10mm (Kg/m^3)	410	410	410	410	410	410	410	410
C.A 20mm (kg/mm^3)	819	819	819	819	819	819	819	819
Water (kg/m^3)	186	186	186	186	186	186	186	186
w/c	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SP (kg/m^3)	4.5	5.9	5.9	5.9	5.9	5.9	5.9	5.9

3.6 Tests Conducted on Hardened Concrete

3.6.1 Compressive Strength Test (IS: 516 – 1959)

This test is performed on cube specimens to determine compressive strength at various ages.

Apparatus

Testing Machine - The testing machine may be of any reliable type, of sufficient capacity for the tests and capable of applying the load at the rate specified. The permissible error shall be not greater than ± 2 percent of the maximum load. The testing machine shall be equipped with two steel bearing platens with hardened faces. One of the platens (preferably the one that normally will bear on the upper surface of the specimen) shall be fitted with a ball seating in the form of a portion of a sphere, the centre of which coincides with the central point of the face of the platen. The other compression platen shall be plain rigid bearing block. The bearing faces of both platens shall be at least as large as, and preferably larger than the nominal size of the specimen to which the load is applied.

Age at Test

Tests shall be made at recognized ages of the test specimens, the most usual being 7 and 28 days. Test at age of 56 days can also be performed. Ages of 13 weeks and one year are recommended if tests at greater ages are required. Where it may be necessary to obtain the early strengths, tests may be made at the ages of 24 hours ± ½ hour and 72 hours ± 2 hours. The ages shall be calculated from the time of the addition of water to the dry ingredients.

Number of Specimens

At least three specimens, preferably from different batches, were made for testing at each selected age.

3.6.2 Splitting Tensile Strength Test (ASTM C496)

This test is performed on cylinder specimen to evaluate its tensile strength at various different ages.

Apparatus

Compression Testing Machine

Bearing Strips - 2 each, 1/8 in. thick plywood strips, 1 in. wide (the length shall be slightly longer that of the specimens). The bearing strips are placed between the specimen and the upper and lower bearing blocks of the testing machine (or between the specimen and supplementary bearing bars if used).

Supplementary Bearing Bars - Steel bar 2 inches wide, 3 inches thick, and 12 inches long.

Procedure

1) Draw diametric lines on each end of the specimen so that they are in the same axial plane.
2) Centre one of the plywood strips along the centre of the lower bearing block.
3) Place the specimen on the plywood strip and align so that the lines marked on the ends are vertical and centered over the plywood strip.

4) Place the second plywood strip and the bearing bar so that they are lengthwise on the cylinder, centered on the previously marked lines on the ends.
5) Apply the load continuously at a constant rate of 100 to 200 psi/minute of splitting tensile stress until failure occurs (the load rate is 11000 to 22000 lb/minute for 6”diameter by 12” long specimens).
6) Record the maximum load at failure.
7) Calculate the splitting tensile strength as follows:

$$f'_{st} = \frac{2P}{\pi\ ld}$$

Where P is the maximum load at failure in pounds, and l and d are the length and diameter of the cylindrical specimen, respectively, in inches.

Report

1) Report the type of concrete, unit weight, and unconfined compressive strength.
2) Report the splitting tensile strength to the nearest 5 psi.

3.6.3. Sulphate Resistance Test

Tests performed for sulphate resistance in this work are compressive strength test after immersing the cube specimen in 5% magnesium sulphate solution for 7, 28 and 56 days. Before immersing them in sulphate solution, specimens are cured for 28 days in water under normal temperature. Compressive strength test has been already discussed above in article 3.6.1.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 General

In this chapter, Compressive strength, Tensile strength and Sulphate resistance of various concrete mixes incorporating 10% foundry sand, containing different percentages of metakaolin and silica fume is discussed. All the tests conducted were in accordance with the methods described in chapter three. Results were compared and checked for compressive strength, split tensile strength and sulphate resistance of concrete.

4.2 Effect of Silica fume on compressive strength, Splitting tensile strength and sulphate resistance of concrete

4.2.1 Compressive Strength Test (IS: 516 – 1959)

In order to study the effect on compressive strength, the cubes containing different proportion of silica fume were prepared and kept for curing for 7, 28 and 56 days. The test was conducted on ACTM of capacity 3000 KN. A compression machine, conforming to ASTM C39, was used for all compression strength testing at a load rate of 0.15 MPa/s. From the results listed in Table 4.1, it is obtained that 56 days strength of all the mixes is invariably higher than corresponding 7 days and 28 days strength. This is due to continuous hydration of cement with concrete.

Fig. 4.1 Photograph of Automatic Compression Testing Machine (ACTM)

Fig. 4.2 Compressive strength test in progress

Table 4.1 Compressive strength of concrete mixes, incorporating 10% foundry sand mixed with silica fume at 7, 28 and 56 days.

MIX	Compressive strength (N/mm^2)			Average Compressive strength (N/mm^2)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1 (10% foundry sand)	22.2	33.1	35.0	22.0	32.7	35.2
	22.5	32.5	35.1			
	21.3	32.1	34.8			
M1 (5% SF)	25.3	38.2	42.3	27.1	38.1	42.1
	27.1	38.5	42.3			
	28.9	37.6	41.8			
M2 (10% SF)	30.6	42.0	45.2	29.5	42.7	46.9
	28.3	41.9	46.7			
	29.6	44.2	48.8			
M3 (15% SF)	27.8	37.9	42.5	27.7	38.9	42.8
	26.9	39.2	42.0			
	28.5	39.6	43.9			

The water-cement was kept constant at 0.5. The test results indicated that, when 5 to 10 percent by weight replacement of silica fume for cement is done, compressive strength increases. When 15% replacement of cement is done by silica fume, strength starts decreasing. Compressive strength of silica fume concrete at 28 days compared to control mix was increased by 20 to 37 percent on increasing the silica fume content from 5 to 10%. Also there is 20% to 25% increase in 28 days strength with 15 % replacement when compared to control specimen but there is decrease of compressive strength when compared to 10% replacement level.

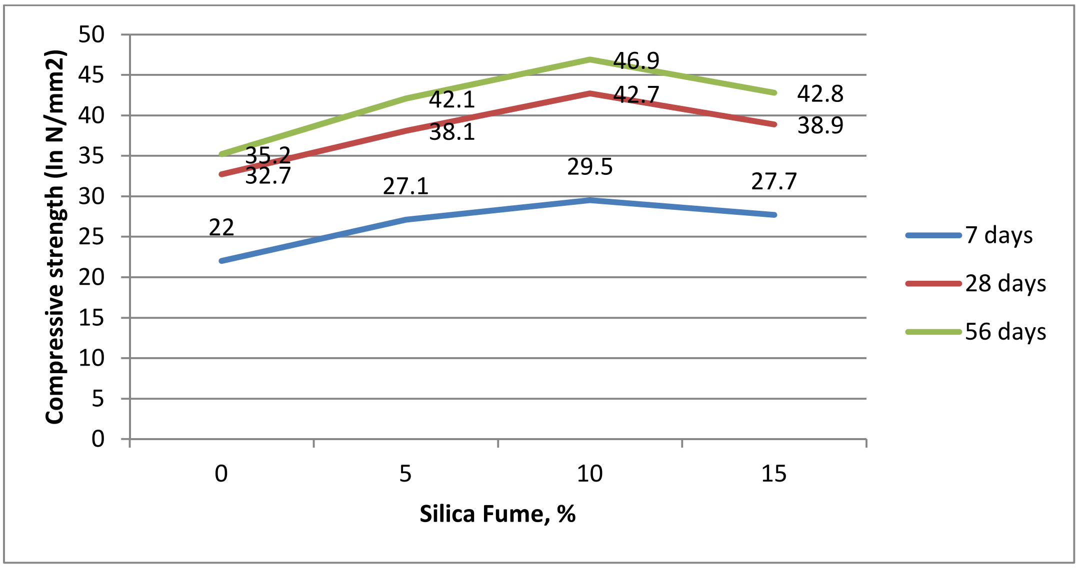

Fig. 4.3 Influence of silica fume on compressive strength of concrete at w/c =0.50

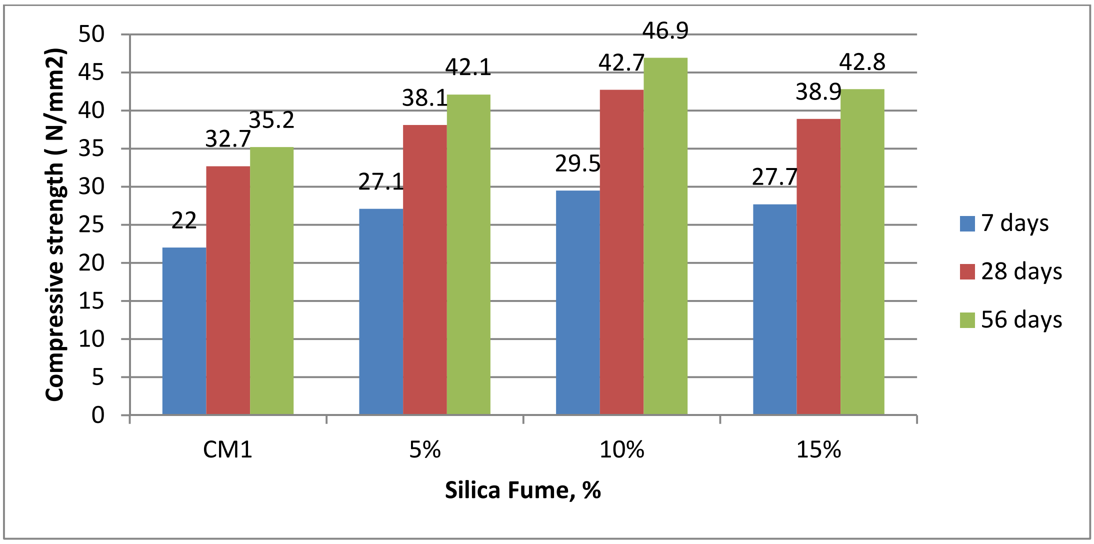

Fig. 4.4 Compressive strength of concrete mixes incorporating 10% foundry sand mixed with silica fume at various ages.

It can be clearly observed in fig. 4.3 and fig. 4.4 that initially when 5% of silica fume was added in cement, the compressive strength increased at 7, 28 and 56 days as compared to the control mix with no silica fume. The maximum increase in strength was observed after 7 days test. When the content of silica fume in cement was increased from 5 to 10%, similar increase in compressive strength was observed at 7, 28 and 56 days. But further increase in silica fume content from 10 to 15 % in cement resulted in decrease in the compressive strength at all ages. Highest strength of 46.9 N/mm^2 was observed for 10% silica fume concrete mix at 56 days.

4.2.2 Splitting Tensile strength Test

Splitting Tensile strength studies were carried out at the age of 7, 28 and 56 days. The results are shown in Table 4.2 and fig. 4.5.

Table 4.2 Splitting Tensile strength of concrete mixes incorporating 10% foundry sand mixed with silica fume at 7, 28 and 56 days.

MIX	Splitting Tensile strength (N/mm^2)			Average splitting strength (N/mm^2)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1	1.6	2.6	3.0	2.0	2.6	2.7
	2.3	2.7	2.9			
	2.1	2.52	2.2			
M1 (5% SF)	2.0	3.1	3.7	2.2	3.0	3.2
	1.9	3.1	3.3			
	2.7	2.8	2.6			
M2 (10% SF)	2.5	3.0	3.5	2.6	3.4	3.6
	2.6	3.3	3.3			
	2.7	3.9	4.0			
M3 (15% SF)	2.5	3.5	3.4	2.1	3.2	3.4
	2.1	3.3	3.6			
	1.7	1.8	3.2			

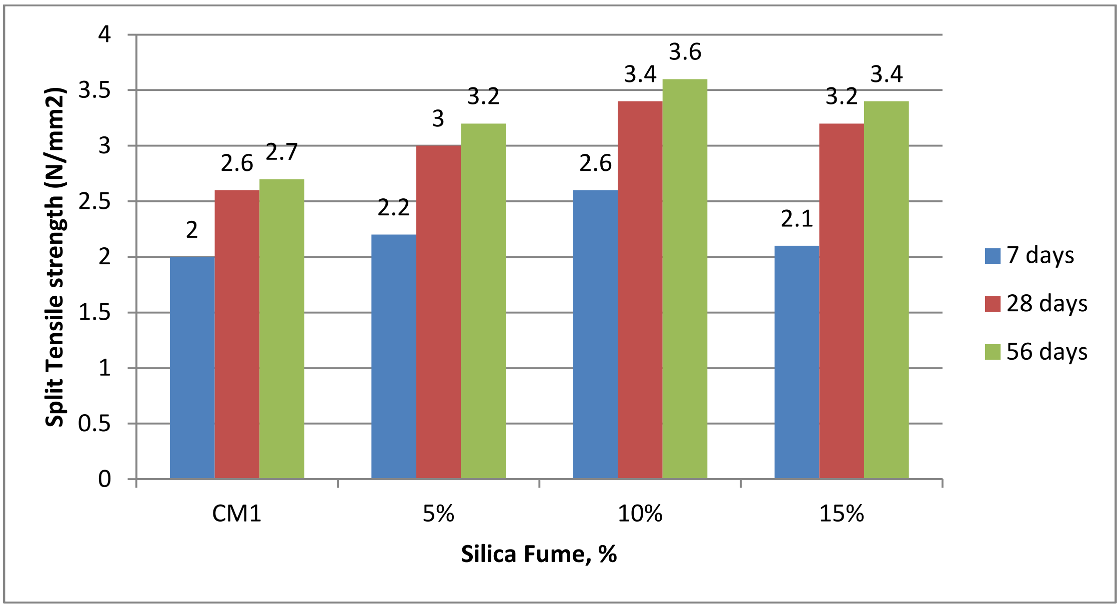

Fig. 4.5 Splitting tensile strength of 10% foundry sand incorporated concrete mixed with silica fume.

The percentage of silica fume replaced by cement is 5%, 10% and 15% and the water cement ratio is kept at 0.5. The test results indicated that 5 to 10% by mass replacement of silica fume for cement provided the highest strength. When we increased the replacement of silica fume by 15% the tensile strength decreased. Splitting tensile strength of silica fume concrete at 28 days compared to conventional concrete/ Control mix increased by 20 to 40 percent with silica fume addition of 5 to 10% respectively. Highest Splitting tensile strength value of 3.6 N/mm^2 was obtained in the 10% Silica fume concrete mix at 56 days testing. These results are represented graphically above in Fig. 4.5.

Fig. 4.6 Splitting Tensile Failure of cylinder

4.2.3 Sulphate Resistance

This test was conducted on 150 x 150 x 150mm cube specimens. The cubes were casted and cured in water for 28 days. Magnesium sulphate solution of strength 5% was used to evaluate sulphate resistance of concrete. Cubes were immersed in solution after 28 days of water curing, and were tested for compressive strength at 7, 28 and 56 days. Test results are given below in Table 4.3. There was decrease in compressive strength of specimens when compared with their normal compressive strengths. But when silica fume and metakaolin were used as partial replacements of cement, then this strength loss was minimized.

When this compressive strength is compared with compressive strength of specimens cured in water at same ages, it is found that there is a loss of compressive strength. After immersion in solution for 28 days, there is 1.5% loss of strength for control specimen. Control specimen simply cured in water has 56 day strength of 31.7 N/mm^2 while those immersed in solution have strength of 31.2 N/mm^2. When cement is replaced by silica fume, this loss tends to decrease. 10% replacement level gives the best result. There is loss of .7% strength. While there is loss of 1.0% and .8% respectively at replacement level of 5% and 15 %

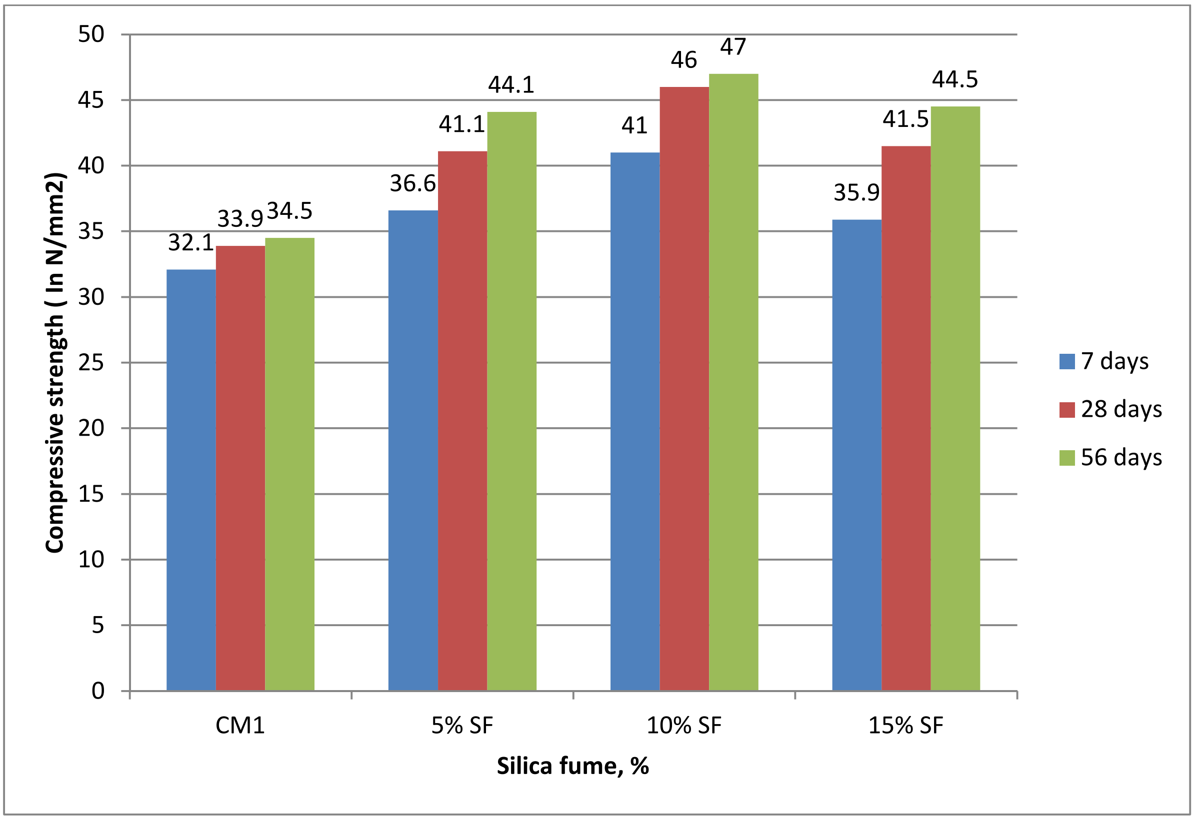

Fig. 4.7 Compressive strength of concrete, incorporating 10% foundry sand, mixed with silica fume at 7, 28 and 56 days after immersion in 5% $MgSO_4$ solution.

Table 4.3 Compressive strength of concrete, incorporating 10% foundry sand, mixed with Silica fume after immersion in $MgSO_4$ solution at various ages.

MIX	Compressive strength (N/mm^2) (After 28 days curing)			Average Compressive Strength (N/mm^2) (After 28 days curing)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1	29.8	35.0	31.0	32.1	33.9	34.5
	31.9	33.4	34.4			
	34.5	33.3	37.9			
5% SF	35.6	44.1	46.2	36.6	41.1	44.1
	36.7	39.6	43.1			
	37.5	39.7	43.0			
10% SF	40.5	45.1	49.7	41.0	46.0	47.0
	40.9	44.5	45.7			
	41.6	48.4	45.6			
15% SF	35.0	39.7	42.6	35.9	41.5	44.5
	36.2	42.3	42.9			
	36.5	42.5	48.0			

From the obtained readings, as shown in Table 4.3, the pattern can be clearly observed that there is an increase in the 7, 28 and 56 days compressive strengths when we increased the silica fume content from 5 to 10% but when we further increased this content to 15%, we see a decline in the compressive strength.

The compressive strength of controlled mix after 28 days of water curing came out to be 32.7 N/mm^2. After 28 days when we removed the cubes from water and were immersed in 5% $MgSO_4$ solution for next 7 days, the compressive strength reduced to 32.1 N/mm^2. Also, the compressive strength of control mix after 28 days of immersion period in sulphate solution came out to be 33.9 N/mm^2 which is less than the 56 days compressive strength of control mix immersed in water.

Thus, this confirms the deteriorating effect of sulphate solution on concrete specimen.

4.3 Effect of Metakaolin on compressive strength, splitting tensile strength and sulphate resistance of concrete

4.3.1 Compressive strength test

In order to study the effect on compressive strength when metakaolin is added in concrete, incorporating 10% foundry sand, as cement replacement, the cubes containing different proportion of silica fume were prepared and kept for curing for 7, 28 and 56 days. The test was conducted on ASTM of capacity 3000 KN.

The specimens were tested in a dry state following the moist curing. A compression machine, conforming to ASTM C39, was used for all compression strength testing at a load rate of 0.15 MPa/s. The strength of specimens was recorded as the load was gradually applied until failure (IS: 516 – 1959). Test results are given below in Table 4.4 and are represented in Fig. 4.8.

Table 4.4 Compressive strength of concrete mixes, incorporating 10% foundry sand, mixed with Metakaolin at 7, 28 and 56 days.

MIX	Compressive strength (N/mm^2)			Average Compressive strength (N/mm^2)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1 (10% FS)	24.2	35.5	34.5	22.0	32.7	35.2
	20.3	32.7	36.1			
	21.9	29.9	35.0			
5% MK	28.5	36.0	41.0	26.2	37.0	41.4
	26.2	36.5	41.0			
	23.9	38.5	42.2			
10% MK	30.8	42.0	50.3	30.6	44.1	47.8
	32.4	45.2	45.3			
	28.6	45.0	47.8			
15% MK	31.1	49.8	50.1	32.7	46.9	47.9
	33.5	45.5	46.8			
	33.5	45.4	46.8			

W/c ratio is kept constant throughout at 0.5 and percentage replacement of metakaolin is 5, 10 and 15%.

The test results indicated that, 5 to 15% by weight replacement of metakaolin for cement gives better strength at both short and long terms. Compressive strength of metakaolin concrete at 28 days compared to control mix was increased by 39 to 58 percent with metakaolin added 5 to 15% respectively. Increase in compressive strength in represented graphically below in figure 5.8. Compressive strength of 5% MK concrete is 26.2, 37.0 and 41.4 N/mm^2 at 7, 28 and 56 days respectively. While that of 15% MK concrete is 32.7, 46.9 and 47.9 at age of 7, 28 and 56 days respectively. There is roughly 25% increase in 28 day strength, when replacement level of metakaolin is increased from 5 to 15%.

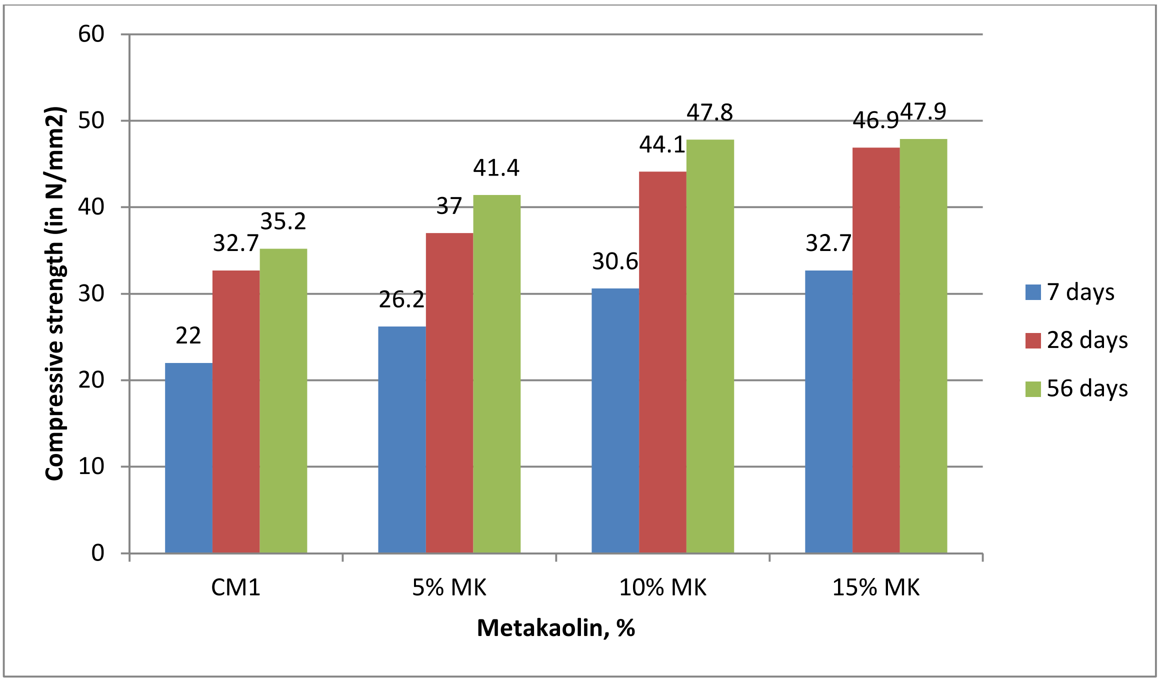

Fig. 4.8 Compressive strength of concrete mixes incorporating 10% foundry sand mixed with metakaolin at various ages.

The compressive strength of concrete, incorporating 10% foundry sand, mixed with metakaolin increases with an increase in the metakaolin content in cement from 5% to 10% to 15% at all ages.

4.3.2 Splitting Tensile strength

Splitting Tensile strength tests were carried out at the age of 7, 28 and 56 days. The results are shown in Table 4.5 and fig. 4.9.

Table 4.5 Splitting Tensile strength of concrete mixes, incorporating 10% foundry sand, mixed with Metakaolin at 7, 28 and 56 days.

MIX	Splitting Tensile strength (N/mm^2)			Average Split Tensile strength (N/mm^2)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1 (10% FS)	1.6	2.9	2.5	2.0	2.6	2.7
	2.1	3.2	2.9			
	2.3	1.7	2.7			
5% MK	2.74	2.8	3.7	2.14	2.85	3.05
	1.5	2.2	2.8			
	2.14	3.45	2.75			
10% MK	2.5	3.0	3.4	2.7	3.5	3.6
	2.9	3.6	3.6			
	2.7	3.4	3.8			
15% MK	2.6	3.6	3.0	2.6	3.65	3.82
	2.1	3.75	3.9			
	3.1	3.5	4.52			

When 5% cement is replaced my metakaolin, it gives splitting tensile strength of 2.14, 2.85 and 3.05 N/mm^2 at the age of 7, 28 and 56 days respectively. At replacement level of 10%, splitting tensile strength comes to be 2.7, 3.5 and 3.6 N/mm^2 at age of 7, 28 and 56 days respectively.

At age of 28 days there is 18 to 40% increase in splitting tensile strength, when compared to control mix at same age. Control mix has strength of 2.6 N/mm^2 at the age of 28 days.

When replacement level is further increased to 15% by weight of cement, there is further increase in splitting tensile strength. It comes out to be 2.6, 3.65 and 3.82 N/mm^2 at the age of 7, 28 and 56 days respectively. When compared to control mix at 28 days there is increase of 50%. While there is increase of 27% and 7% splitting tensile strength at 15% replacement, when compared to 5 and 10% replacement levels.

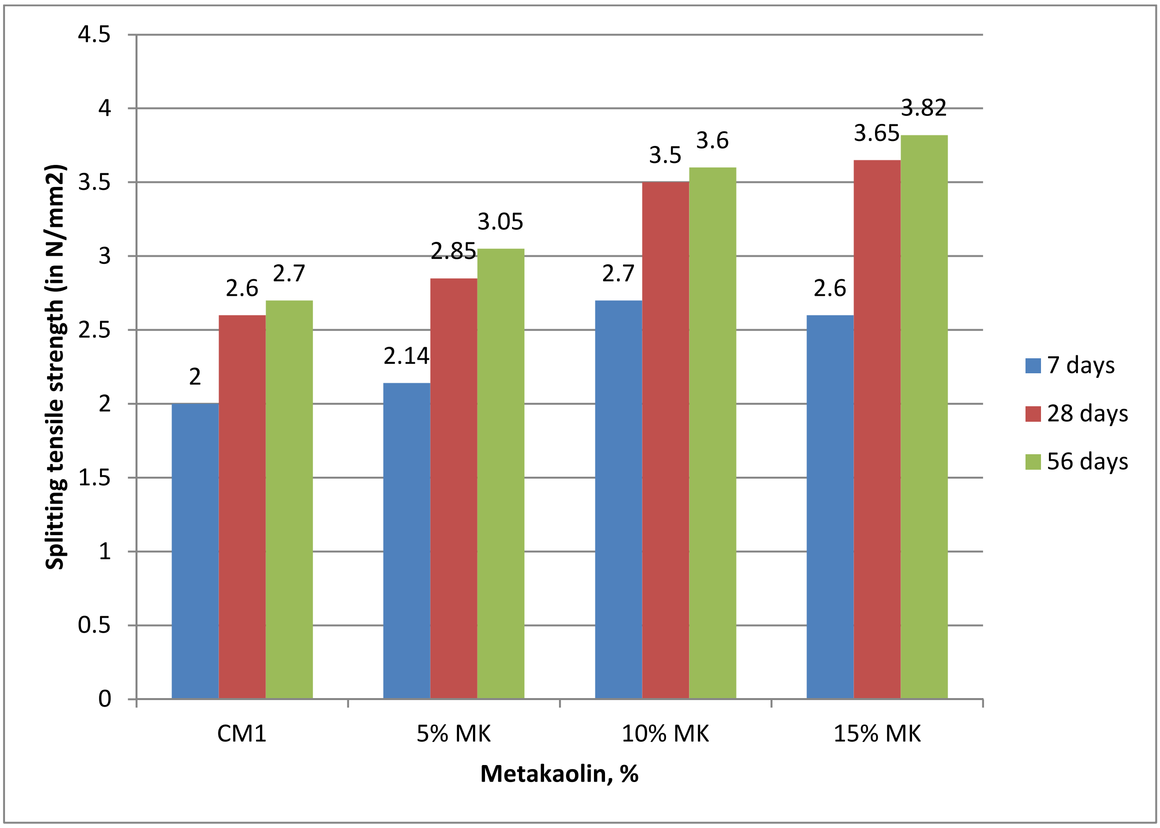

Fig. 4.9 Splitting tensile strength of concrete mixes incorporating 10% foundry sand mixed with metakaolin at various ages.

The percentage of metakaolin replaced by cement is 5%, 10% and 15% and the water cement ratio is kept at 0.5. The test results indicated that there was an increase in the splitting tensile strength of all concrete mixes on increasing the metakaolin content from 5 to 15% in cement except when we increased the metakaolin content from 10% to 15% at 7 days.

Highest Splitting tensile strength value of 3.82 N/mm^2 was obtained in the 15% Silica fume concrete mix at 56 days testing.

4.3.3 Sulphate Resistance

Sulphate resistance of concrete was measured by performing the compressive strength test after 7, 28 and 56 days of immersion period in sulphate solution, the results of which are given in Table 4.6 and fig. 4.10.

When this compressive strength is compared with compressive strength of specimens cured in water at same ages, it is found that there is loss of compressive strength. After immersion in solution for 28 days, there is 1.5% loss of strength for control specimen. Control specimen simply cured in water has 56 day strength of 35.2 N/mm^2 while those immersed in solution have strength of 34.5 N/mm^2. When cement is replaced by metakaolin, this loss tends to decrease. Strength results are represented below in Fig. 4.10 and given in Table 4.6. At the replacement level of 5% metakaolin, loss of strength is 8%. Strength of 5% MK specimen after immersion in $MgSo_4$ solution for 28 days is 40.0 N/mm^2, while of the specimen that is cured in simple water is 38.1 N/mm^2. With increase in replacement level upto 10%, loss of strength becomes 7%. When replacement level is further increased, loss of strength decreases further. Strength of 15% replacement specimen after immersion for 28 days in MgSo4 solution is 48.8 N/mm^2. Its loss of strength is approximately 6%, when compared with strength of 15% MK sample cured in water for 56 days. Loss of strength of specimens after immersion for 28 days in $MgSo_4$ solution, when compared with same replacement level specimens cured in simple water for 56 days are represented graphically below in figure 4.10.

Table 4.6 Compressive strength of concrete mixes, incorporating 10% foundry sand, mixed with Metakaolin immersed in $MgSO_4$ solution at 7, 28 and 56 days.

MIX	Compressive strength (N/mm^2) (After 28 days curing)			Average Compressive Strength (N/mm^2) (After 28 days curing)		
	7 days	28 days	56 days	7 days	28 days	56 days
CM1 (10% FS)	32	34	34.7	32.1	33.9	34.5
	29.9	33.8	34.3			
	32.1	33.9	34.5			
5% MK	36.1	39.5	42.8	36.2	40.0	42.9
	36	40	42.9			
	36.4	40.5	43			
10% MK	41.3	42.8	46.4	41.5	46.8	48.4
	41.7	47.8	50.2			
	41.5	49.8	48.6			
15% MK	44.0	49.0	51.5	44.2	48.8	50.8
	44.6	49.6	51.1			
	44.4	47.8	49.8			

The highest compressive strength reading of 50.8 was obtained in 15% metakaolin concrete mix after 56 days of sulphate solution immersion (after 28 days of water curing).

The decrease in compressive strengths of concrete mixes at all ages after immersion in sulphate solution confirms the deteriorating effect of sulphate solution on concrete.

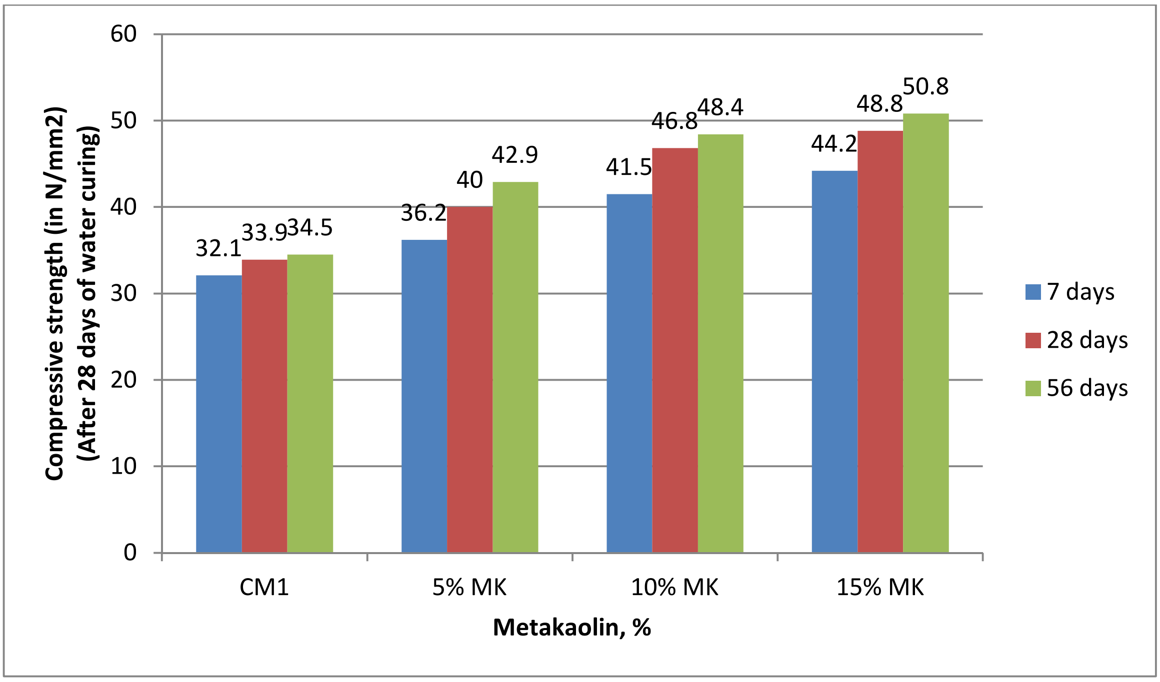

Fig. 4.10 Compressive strength of concrete, incorporating 10% foundry sand, mixed with metakaolin at various ages after immersion in 5% $MgSO_4$ solution.

CONCLUSIONS

Following observations have been made from the study of using silica fume and metakaolin in concrete:.

- The concrete mixes developed compressive strength of 27.1, 29.5 and 27.7 N/mm^2 at 7 days with silica fume replacement of 5, 10 and 15% respectively. While it achieves strength of 38.1, 42.7 and 38.9 N/mm^2 at age of 28 days. At age of 56 days, strength achieved is 42.1, 46.9 and 42.8 N/mm^2 respectively. When metakaolin is used as supplementary material in proportions of 5, 10 and 15%, compressive strength achieved at age of 7 days is 26.2, 30.6 and 32.7 N/mm^2. At age of 28 days strength is 37.0, 44.1 and 46.9 N/mm^2 respectively. While at age of 56 days strength achieved is 41.4, 47.8 and 47.9 N/mm^2. Compressive strength increases with increase in percentage of metakaolin while in case of silica fume strength increases upto 10% replacement level and then starts decreasing.

- Splitting tensile strength developed from 2.2 to 2.6 N/mm^2, 3.0 to 3.4 N/mm^2 and 3.2 to 3.6 N/mm^2 at 7, 28 and 56 days respectively with increase in silica fume content from 5% to 10% and The Concrete mixes developed Splitting tensile strengths ranging from 2.14 to 2.6 N/mm^2, 2.85 to 3.65 N/mm^2 and 3.05 to 3.82 N/mm^2 at 7, 28 and 56 days respectively with increase in metakaolin content from 5% to 15%.

- Use of silica fume and metakaolin as replacements of cement improves sulphate resistance of concrete. When measured in terms of compressive strength loss, there is loss of 1, 0.7 and 0.8% strength for 5, 10 and 15% replacement level of silica fume respectively. While in case of metakaolin loss of strength is 0.8, 0.7 and 0.6% for replacement level of 5, 10 and 15% respectively.

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