SOME STUDIES ON CEMENT AND HIGH DENSITY CONCRETE

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BY

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DECLARATION

I, Shraddha Chakurkar hereby declare that this thesis represents work which has been carried out by me and that it has not been submitted, either in part or full, to any other University or Institution for the award of any research degree.

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CERTIFICATE

I hereby certify that the work was carried out under my supervision and may beplaced for evaluation.

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Shraddha Chakurkar

(Shraddha Mahadev Anawkar)

“A Ph.D. is not a destination; it is journey of continuous learning, growth and self-discovery”

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ABSTRACT

Cement is the most used building material on the planet earth and is responsible for 8% of the total CO_2 released. With the estimated increase in population in the coming decades and infrastructure needs, a surge in cement demand is expected. Approach while solving this problem needs to be restructured to enhance the quality while meeting the demand and keeping the carbon foot-print intact or lower for a given volume of production. This work introduces a novel approach to increase efficiency of the cement with particle size engineering. Since raw materials used in cement production vary in quality across the globe, the only feasible method of enhancement is through modification of mechanical parameters such as particle size and their distribution as one of the cost effective mechanisms. The study details an experiment on cement and concrete using a "Stimulator" to OPC. It's content was varied between 0 and 100 per cent in the intervals of 10%.

The optimum dosage of the stimulator was found from the results of tests conducted, such as water requirement, setting times, the heat of hydration, chemical shrinkage and compressive strength parameters. Despite the reduction in the average particle size of cement by 38 to 47%, water requirement has shown a nominal rise of 10 to 17% for S40 to S50 cement mixes, respectively. For a known strength of concrete, 20-25% lesser quantity of stimulated cement was required (optimum dosage of stimulator) as against the concrete containing convetional OPC. This reduction in the requirement of cement quantity for a given volume leads to lower cost and lower amount of CO_2 released. Implementation of this method of cement production does not require the introduction of new machinery. Stimulated cement portrayed a double peak PSD arrangement unlike OPC with a single peak, thus representing the participation of 2 sets of particles in the process of enhanced hydration. The outcome of the study was used in developing High density and high strength concrete. The single specific notable achievement is its early strength to an extent of 80% in a single day over the OPC. Durability tests conducted on concrete samples with the optimum dosage of stimulator and OPC have depicted better durability than concrete samples with conventional OPC.

Keywords: Efficiency, High density concrete, Particle Size Distribution, Stimulator, Sustainability.

Chapter 1

Introduction

1.1 Background and Introduction

Concrete is the second most consumed material after water on the planet and involves the consumption of natural resources. The Construction industry is anticipated to grow by 6.6% by 2028 because of the expected increase in population and demand for infrastructure. This surge in demand for infrastructure leads to the increased demand of cement. The construction industry uses many kinds of cement-based materials majorly masonry blocks, precast elements and other concretes. Although India has an abundant natural resources, it is high time to use them judiciously as they are depleting very fast. Hence, efficient and sustainable use of these materials in the construction industry becomes mandatory.

Concrete technology has witnessed many inventions and modifications since its inception to meet the demand for various kinds of structures as the need arose. The quality of concrete is majorly controlled by the quality of binding material used and aggregates quality. Apart from aggregates, cement affects the quality of the concrete matrix in particular. Several researchers developed the concrete to their needs however, the mother binding material the OPC is not changed. This study of engineering of particle size is an attempt towards enhancing the quality of cement by maximizing its efficiency in the physical and chemical properties.

1.2 Cement – Industry Background and Current Scenario

Cement production in India started in the year 1904 and boomed only after 1989 due to the decontrolled policy of cement production (M. S. Shetty and A. K. Jain, 2005). Cement and steel are the most used construction material which contributes to anthropogenic CO_2 released into the atmosphere. Globally, India ranks second in the production of cement which amounts to 545 Million tons (Indian Cement Industry Analysis, 2022) which is

about 8% of total global capacity. It is predicted in 2025 the demand for cement in the country might rise to 550-600 MT. This growth amounts to a 10% average increase annually. Thus, it is the most consumed material in the construction industry hence, it becomes necessary to maintain the quality and its cost of production. Cement as a binding material has witnessed many changes concerning mechanical and chemical properties.

The research has emerged continuously in its quality tuning to the requirements. The development of high early strength is one of the challenges that the cement industry faced. High early strength saves anticipated costs of formwork and other associated mandatory requirements. Meeting such requirements needs the optimization of cement quality as a modern material. This study is towards its quality improvement with a specific note towards the behaviour of fine and coarse particles in the cement matrix. (Chakurkar & Guptha, 2021)

1.3 Sustainability- Current scenario of the cement industry

The Kyoto Protocol focused on sustainability considering the production of cement while controlling the emission of CO_2. During the production level, methodology and raw materials used in production significantly influence the carbon footprint and energy requirement of cement. The assessment of the type of fuel, machinery used and quality of raw materials available to achieve the reduction in carbon footprint and energy requirements is mandatory.

World Business Council for Sustainable Development (WBCSD) outlined 4 levers to mitigate CO_2 emission: (i) Improving energy efficiency (accounting for a 12% potential reduction of direct CO_2 emission of global cement manufacture), (ii) Switching to alternative less carbon-intensive fuels (42% potential reduction), (iii) reducing the clinker to cement ratio, namely using blended materials (128% potential reduction) and (iv) using emerging and innovative technologies to adopt excess heat recovery and to integrate carbon capture into the cement manufacture process (166% potential reduction)(Sandra Nunes a, 2021).

Focusing on the third suggestion cited above, this study focused on the approach to reduce clinker to cement ratio by increasing the efficiency of cement, which also leads to carbon

capture during the manufacturing process, thus, achieving sustainability at the production level.

1.4 Effect of Particle Size Distribution (PSD) on Efficiency of Cement

The efficiency of cement can be improved chemically, either at the clinker formation level or by using mineral admixtures. However, researchers noted its properties across the globe have made standardization difficult (K., 2005),(Mehairi & Husein, 2020),(Arteaga-Arcos et al., 2013). Further, they believed that modification of physical properties is the most sustainable method to achieve high-performance cement in a standard manner across the globe.

Cement is composed of a wide variety of particles ranging from 3 to 85µm in commercially available cement. The efficiency and performance levels of the materials depend on the particle size (Nguyen et al., 2021). The cement industry in the recent past has introduced activators to enhance regional-specific requirements in production.

Currently, OPC is used in combination with pozzolanic materials to meet expectations of high-strength concrete, sustainability and durability. The literature categorizes the particles into 4 different combinations viz., < 3, 3-9, 9-25 and >25µm. (Nguyen et al., 2021), (Bentz, 2010), (Kuri et al., 2022). Finer particles in the range of 3µm have a greater affinity towards the water and contribute to 1-day compressive strength and particles between 3-9µm and 9-25µm contribute to 28 days strength. However, the particles coarser than 25µm do not contribute much to compressive strength either due to non-participation in the hydration process or their size being inactive to hydration and thus they remain as fillers in the matrix.(H., 1997)

Various parameters of cement which are vital in determining the final properties of concrete, such as fluidity of the paste, average particle size, particle packing, pore size distribution and density of hydrated product are affected by the range of PSD of the cement sample (Lee S.H., H.J. Kim, E. Sakai, 2003) Cement with wider PSD has better particle packing as compared to one with narrow PSD due to the sequential participation during the process of hydration (Lee S.H., H.J. Kim, E. Sakai, 2003), (Arteaga-Arcos et al., 2013). A synergetic effect of increased fineness with wider PSD is observed in the current

experimental investigation achieved through the engineering of PSD. An increase in the fineness of cement has increased affinity towards water. However, the specific observation by EC Higginson (E.C.Higginson, 1970) indicated that a range of fineness of cement from 4000 to 4800cm^2/g showed an inverted bell-like relation with water requirement. Further, he elaborated that for a given water requirement there is 2 corresponding fineness. This unique concept of cement fineness observed specifically in the study enhances the process of hydration entirely different from the typical conventional one. A few researchers also stated that increased fineness helps to improve the absorption of admixture and reduction in crack width, as well. (Nguyen et al., 2021), (Divanedari & Eskandari-Naddaf, 2020) (Adu-Amankwah et al., 2021). This study is also following their findings and is in good agreement with their results.

1.5 Aim of research

The present research aims to engineer the particle size distribution of cement to enhance efficiency without significant change in water demand to achieve a higher early compressive strength of cement. Concrete with engineered PSD of cement resulted in the expected guidelines in achieving strength and cost of the concrete too. This novel approach has not only improved the strength but also the cost and sustainability requirements. Concrete composed of aggregates with high density and engineered cement were the outcomes of the work in show causing strength, durability, sustainability and performance.

1.6 Objectives of the Research

The study proposed has the following objectives.

1. To analyze the range of particle size in the commercially available OPCs in the market.
2. The blending of micro-fine particles as "Stimulator" in predefined dosages aimed to achieve an engineered PSD of cement and marginal positive enhancement on the properties of cement.
3. Optimizing the engineered PSD based on the hydration process by sequential participation of the cement in combination with the stimulator.
4. To determine the effect of optimal dosage of stimulator on the properties of concrete.

5. To compare the cost analysis and sustainability assessment of engineered cement.
6. To study the microstructure and particle packing of concrete using VCCTL and its validation.

1.7 Scope of study

In the world of sustainability, the material management and conservation of resources plays an important role. Enhancement of load-carrying capacities while achieving durability and strength is the key factors in the design mixes. Precast structural elements are becoming popular in place of low-grade concretes and conventional concretes. Structural concretes with high-density materials are considered futuristic materials. Their size and efficiency of structures with high density prompted the slenderness while achieving the performance. This modern concrete has its applications widely in the commercial sectors too.

Construction of nuclear power plants, radiation rooms in hospitals, counterweights for bridges, slender structural members, ballast blocks for washing machines etc. are some of the applications of high-density concrete. Cement is the only binding material in the concrete. Reduction in the consumption of cement for a given volume plays an important role in the design of mixes. Higher consumption of cement leads to several unanticipated problems with the structure. Efficient cement not only helps in improving the properties as a binder material but also the durability too.

1.8 Outline of research

To achieve the aim and objectives of the study stated in articles 1.2 and 1.3, the following step-by-step procedure is adopted.

1. To perform a literature review to define the methodology to be adopted for the engineering of PSD of cement and understand its effect on various parameters of cement and concrete thus produced. The effect of cement fineness on the cost and environmental impact of cement and concrete is also summarized.
2. Perform comprehensive literature analysis to determine the usage and application of high-density concrete in various sectors of the construction field and also determine the suitability from durability and environmental aspect.

3. Material allocation and conducting preliminary tests to determine the quality as per Indian standards norms. Also, design the detailed methodology of the experimental work to be adopted.
4. Engineer the PSD of cement and find its optimum dosage of the stimulator to achieve higher compressive strength without significant change in the water requirement of cement mortar.
5. Design the mix proportions of cement mortar, normal concrete and high-density concrete for comparison
    a) Cement mortar to find out the optimum dosage of stimulator and OPC knowing its compressive strength, water requirement and set times.
    b) Design normal concrete using this engineered cement mix containing the optimum dosage of stimulator and OPC
    c) Design high-density concrete using the engineered cement mix of "Stimulator" and OPC
6. To carry out a cost analysis and sustainability assessment of cement and concrete at the production level

1.9 Novelty of research

Increasing cement fineness can no longer be considered a novel approach since a lot of research is carried out in the field of cement in redefining the fineness of cement to achieve desired results. Change in cement fineness affects many properties such as water requirement, setting times, compressive strength and microstructure. However, the majority of studies are focused on using either coarse or fine cement and not specifically engineering the Particle Size Distribution. Few researchers tried to study the effect of using microparticles of cement on cement properties but failed to locate the exact particle size range and their precise effect on all the parameters of cement and concrete. This study focused on the engineering of PSD to obtain the efficiency of cement. The approach was to engineer the PSD to obtain higher compressive strength with the least effect on water requirement. It was also intended to find the effective particle size range of cement primarily believed to increase the efficiency of cement alone and its performance of concrete as overall. Separating cement particles based on their sizes is a very difficult and tedious job however this study proposes an approach of engineered PSD by blending the stimulator (micro fine particles) with OPC.

This method envisages almost double efficiency in terms of strength and durability for a given quantity of cement as compared to conventional and commercially available cement.

1.10 Structure of thesis

The thesis has been structured as follows:

Chapter 2: Literature Review

A comprehensive review of literature on the following aspects includes

a) History and background of the cement industry
b) Current scenario of the cement industry
c) Current scenario of sustainability
d) Effect of using cement fineness on various properties of concrete
e) Effect of fineness on water requirement
f) Effect of cement fineness on hydration mechanism of cement and shrinkage cracks and porosity
g) Effect of cement fineness on the heat of hydration
h) High-density concrete

Chapter 3: Materials and Mix Proportions

This Chapter deals with materials used in the experimental study and their related properties following the codal provisions wherever applicable. Physical and chemical properties influence the performances while designing the mixes.

Chapter 4: Methodology

Standard operating procedures have been followed as per the relevant Indian Standards while establishing the properties. The methodology to be followed for the micro-fining of cement using a jet mill is explained. The activator and admixtures referred were tested confirming the relevant codes.

Chapter 5: Test Results and Discussions

This Chapter contains test results conducted on cement, cement mortar and concrete samples. The comparison is based on the efficiency of the controls and that of the modifications brought in the cement and the stimulator. The results were analyzed and interpreted with the reasons for the behaviour.

Chapter 6: Mathematical Model and Validation

This Chapter describes the analysis and validation of experimental results with the mathematical model developed by the software VCCTL. The interdependency of the properties and their final influence on the strength were also estimated. The combination of the mathematical model and its validation was tested.

Chapter 7: Sustainability and cost analysis

The influence of sustainability and environmental impact of cement production at source and the detailed cost analysis at the production level is compared in this chapter. Carbon footprint concerning its sustainability along with the cost is compared with the engineering of PSD of cement.

Chapter 8: Conclusions

This Chapter includes major conclusions drawn from the study. The contributions of the current work along with the future scope of research are listed for reference.

The **References** follow in successions.

The above Chapter covers an introduction to the research topic, aim, objectives, scope and structure of the thesis. The next Chapter describes a detailed literature review carried out and the research gap identified for the study.

Chapter 2

Literature Review

This Chapter describes a detailed literature review summarizing the history and background of the cement industry, the current status of the cement industry, and the present scenario of sustainability. This Chapter also briefs about the effect of cement fineness on various properties of cement, concrete and high-density concrete elaborating on the recent advances and research. The Chapter also concludes by identifying the research gap.

2.1 Cement

In the most general sense of the word, cement is a binder, a substance which sets and hardens independently, and can bind other materials together. The word "cement" traces to the Romans, who used the term "opus caementicium" to describe masonry which resembled concrete and was made from crushed rock with burnt lime as binder. The volcanic ash and pulverized brick additives which were added to the burnt lime to obtain a hydraulic binder were later referred to as cementum, cimentum, cäment and cement.

2.2 History and background of the cement industry

The cement industry is one of the largest industries in construction and ranks next to the steel industry. History of construction and binding material dates back to 3000BC to the Roman Era. The modern-day cement which is popularly known as Ordinary Portland Cement (OPC) was invented in the year 1824 by Joseph Aspdin, Post-invention of Portland cement many countries took up cement production. However, many scientists contributed to developing cement to rise to the current standards from various countries.

The first cement plant in India was set up in the year 1914 at Porbandar in Gujarat following year cement plants were set up at many other locations in the country. Associated Cement Companies Ltd. (ACC) was formed which led to regulation in the

production of cement quantity and quality proven to be an important step in the history of cement. After the formation of ACC, many other companies were set up leading to a boom in the cement industry. PSC and PPC were introduced in the year 1980 which also gave a new path towards sustainable cement manufacturing.

The main components of standard Portland cement are listed below.

a) Lime (calcium oxide; 66%) in the form of limestone

b) Silica (silicon dioxide; 22%) is a component in most quartz, which forms the particles of clays

c) Aluminium oxide (4%) is found in large quantities in many clays. The proportion of alumin oxide in the clay can be adjusted by the addition of bauxite, which is mainly water-soluble aluminium oxide.

d) Iron oxide (3%) found in iron ore and clay

e) Magnesium oxide (2%)

f) Sulphur dioxide (2%)

g) Miscellaneous components (1%)

The manufacturing process aims to produce a material with a high content of tricalcium silicate, usually 55 to 62% of the crystals in the clinker. Other crystals formed are about 15% dicalcium silicate, (the same component as the hydraulic binder in hydraulic lime), 8 to 10% tricalcium aluminate and 9% tetra calcium aluminate ferrite commonly known as Bogue's compounds.

2.3 Current scenario of the cement industry

Currently, various types of cement are available in the market which are listed below.

a) Portland cement: Cement is manufactured by heating limestone with small quantities of other materials (such as clay) to 1450°C in a kiln. The resulting hard substance, called 'clinker', is then ground with a small amount of gypsum not exceeding 5% by weight cement into a powder to make 'Ordinary Portland Cement'. The addition of gypsum will control the setting times.

Portland cement is a basic ingredient of concrete, mortar and most non-speciality grout. The most common use for Portland cement is in the production of concrete. It is a composite material consisting of aggregates (gravel and sand), cement, and water. As a construction material, it can be cast in almost any shape desired, and once hardened, can become a structural (load-bearing) element. Portland cement may be grey or white.

b) Portland cement blends: These are often available as inter-ground mixtures from cement manufacturers, but similar formulations are often also mixed from the ground components at the concrete mixing plant.

c) Portland Blast furnace Cement: contains up to 70% ground granulated blast furnace slag, with the rest Portland clinker and a little gypsum. All compositions produce high ultimate strength, but as slag content is increased, early strength is reduced, while sulfate resistance increases and heat evolution diminishes. Used as an economic alternative to Portland sulfate-resisting and low-heat cement.

d) Portland Fly-ash Cement: contains up to 30% fly-ash. The fly ash is pozzolanic so that ultimate strength is maintained. Because fly-ash addition allows lower concrete water content, early strength can also be maintained. Where good quality cheap fly ash is available, this can be an economic alternative to ordinary Portland cement.

e) Portland Pozzolon Cement: includes fly ash cement, since fly ash is a pozzolan, but also includes cement made from other natural or artificial pozzolans. In countries where volcanic ashes are available (e.g. Italy, Chile, Mexico, and the Philippines) these cements are often the most common form in use.

f) Portland Silica Fume Cement: The addition of silica fumes can yield exceptionally high strengths, and cement containing 5-20% silica fume is occasionally produced. However, silica fume is usually added to Portland cement at the concrete mixer.

g) Masonry Cements: are used for preparing bricklaying mortars and stuccos, and must not be used in concrete. They are usually complex proprietary formulations containing Portland clinker and several other ingredients that may include limestone, hydrated lime, air entrainers, retarders, waterproofers and colouring agents. They are formulated to yield workable mortars that allow rapid and consistent masonry work. Subtle variations of

Masonry cement in the US are Plastic Cements and Stucco Cements. These are designed to produce a controlled bond with masonry blocks.

h) Expansive Cement: contains, in addition to Portland clinker, expansive clinkers (usually sulfoaluminate clinkers), and are designed to offset the effects of drying shrinkage that is normally encountered with hydraulic cement. This allows large floor slabs (up to 60 m square) to be prepared without contraction joints.

i) White blended cement: may be made using white clinker and white supplementary materials such as high-purity metakaolin.

j) Colored cement: is used for decorative purposes. In some standards, the addition of pigments to produce "coloured Portland cement" is allowed. In other standards (e.g. ASTM), pigments are not allowed constituents of Portland cement, and coloured cement is sold as "blended hydraulic cement".

k) Very finely ground cement: made from mixtures of cement with sand or with slag or other pozzolan-type minerals which are extremely finely ground. Such cement can have the same physical characteristics as normal cement but with 50% less cement particularly due to their increased surface area for the chemical reaction. Even with intensive grinding, they can use up to 50% less energy to fabricate than ordinary Portland cement.

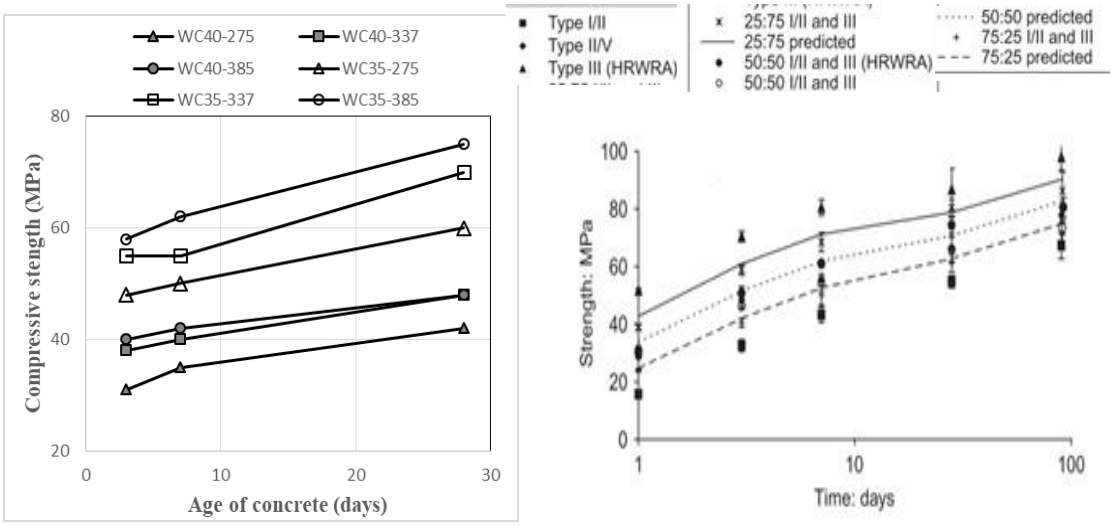
2.4 Current scenario of sustainability

With the surge in infrastructure demand increase in cement is bound to happen. Cement production is responsible for the release of CO_2 into the atmosphere; hence, sustainability in the production of cement has been a topic of research for many years. At the production level, methodology and raw materials used in production significantly influence carbon footprint and energy requirements. The assessment of the type of fuel, machinery used and quality of raw materials available to achieve the reduction in carbon footprint and energy requirements is mandatory. It is already described in article 1.3 regarding the importance of Bogue's compound while attaining sustainability.

2.5 Effect of fineness of Cement on properties of concrete

Cement production is a very tedious procedure and many factors affect the quality of cement produced. Hydration is a prolonged process which depends on the particle size of cement. (J. Talaber, 1982). It was observed and concluded that the rate of hydration increases with the fineness of cement (Sajedi and Razak, 2011), Coarser particles consume more time to hydrate completely as compared to finer particles. Finer cement helps to achieve early strength but does not contribute to later compressive strength. The fineness of cement has the added advantage of reducing the bleeding of concrete. (Sajedi and Razak, 2011) Along with the particle size range the particle size distribution also affects the compressive strength value.

Cement with the same specific surface when compared to PSD has a different effect on compressive strength. Cement with narrow PSD achieves higher compressive strength as compared to coarser cement with wider PSD. (J. Talaber, 1982), (Bentz, 2010), (Bentz, 2010) presented experimental results showing the effect of increased fineness on compressive strength. The experimental results obtained are presented in Figure 2-1 D. P. Bentz (2010) and Yang Kai (2017) et al used cement with fineness 302, 387, 613 m^2/kg and 275,337 and 385 m^2/kg respectively.



(a) Experimental Compressive strength **(b) Comparison of experimental and theoretical compressive strength**

Figure 2-1: Relation between compressive strength and Cement fineness for different W/C ratios as per D. P. Bentz (2010)

Arteaga-Arcos et al., 2013 studied and observed that wider PSD means higher packing density of powder and better mechanical properties of hardened cement. Cement with equal PSD but different PSD implies that finer cement contains smaller particles which fill the interstice between large particles without forcing the large particles apart in turn even smaller particles can be selected to fit into remaining pores, giving a corresponding improvement in packing density. Apart from overall Particle size distribution specific particle size ranges affect properties significantly.

Zhang et al., 2011 noted that particles larger than 45 microns are difficult to hydrate and particles larger than 60 microns have only a filling effect and practically no contribution to compressive strength. Cement particles finer than 3 microns are responsible for various problems such as high early strength, high water requirement, undesirable volume changes and deterioration in rheological properties.

J. Talaber, 1982. Talaber observed and suggested specific particle size ranges responsible for compressive strength. It was observed that particles finer than 3 microns contribute to 1-day compressive strength and cement particles ranging from 3-25 microns are responsible for 28 days of compressive strength. He also observed and stated that the process of strength is slower for 3-25 microns size but is crucial for the final 28 days of compressive strength.

Sajedi and Razak, 2011 compared the use of normal OPC available in the market and finer cement production after grinding OPC for 6 hours. It was observed and concluded that the rate of hydration increases with the fineness of the cement. However, when choosing fine cement, the cost of grinding and the effect of fineness on other properties should also be studied. Finer cement helps to achieve early strength but does not contribute to later compressive strength. The higher fineness of cement increases water requirement and can reduce later compressive strength. The fineness of cement has the added advantage of reducing the bleeding of concrete.

2.6 Effect of fineness on water requirement

On a broader scale increase in cement fineness must lead to an increase in water requirement however some researchers noted that for a specific range of cement, fineness has shown a dip in water requirement despite the increase in fineness. Experimental observations made by some researchers have been presented below.

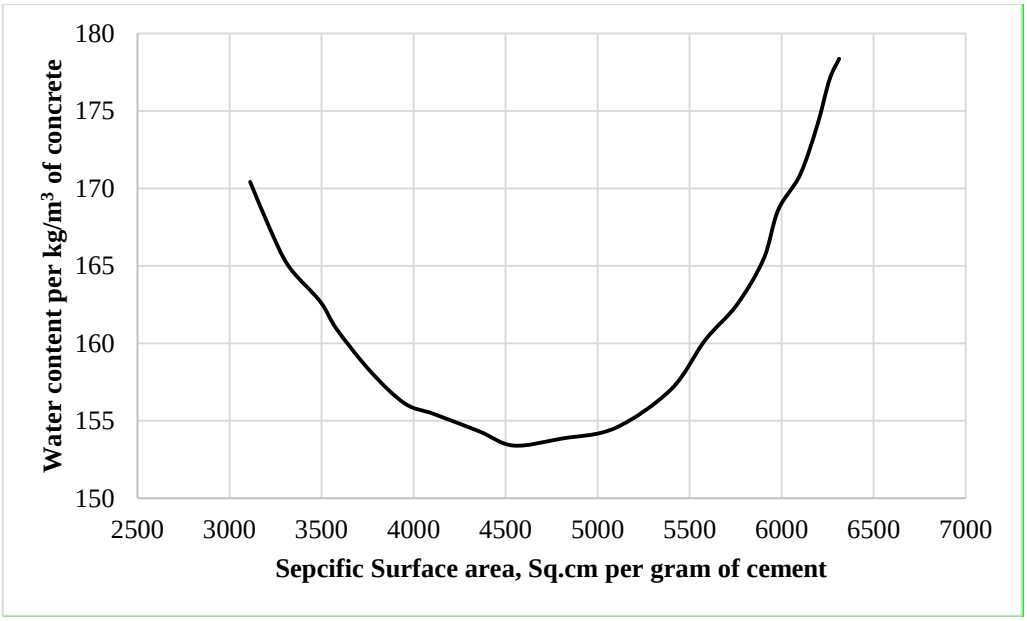


Figure 2-2 Relation between the fineness of cement and water demand as per E.C. Higginson

As per E.C. Higginson, the water demand decreased as the fineness increased up to 4000 cm^2/g where it remained constant until a fineness of 4800 cm^2/g and it began to increase beyond 4800 cm^2/g. These values are presented graphically above in **Figure 2-2.** Daegeon Kim observed and presented that water required slightly decreased with an increase in the fineness of cement and then a sudden increase in water demand was seen beyond FMC of 5. **Figure 2-3** and **Figure 2-4** show the trend of change in water demand with change in specific surface area and FMC respectively. (Daegeon Kim, 2018)

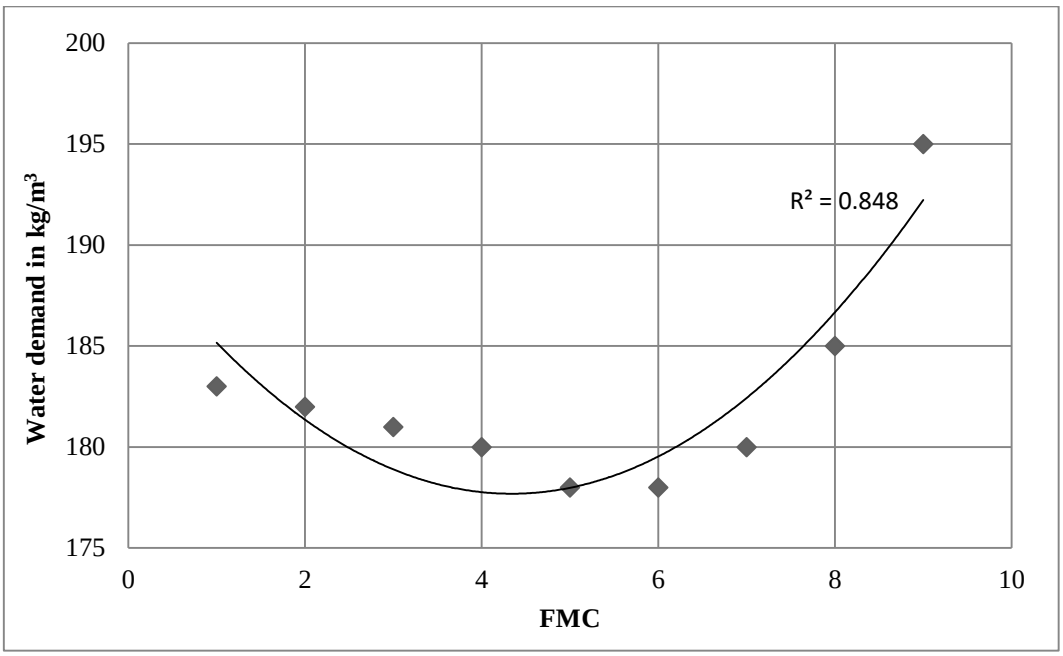


Figure 2-3 Water requirement v/s FMC of cement samples studied by Daegeon Kim

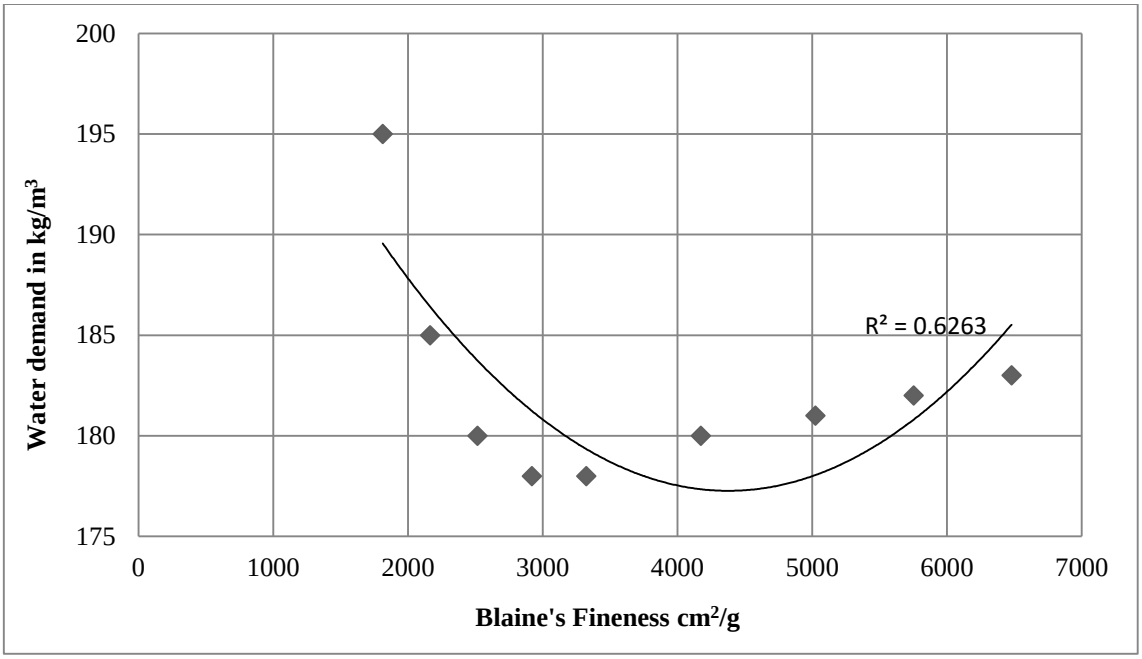


Figure 2-4: Water requirement v/s Blaine's Fineness of cement samples studied by Daegeon Kim

2.7 Effect of cement fineness on hydration mechanism of cement and shrinkage cracks and porosity

The total value of heat of hydration is directly related to the chemical content of cement however the rate of release of this heat is dependent on the cement fineness.

(Hu et al., 2014) also stated in their study that coarser cement exhibited a slower rate of release of the heat of hydration however a lower W/C ratio showed a higher value of heat of hydration. The total heat of hydration in 24 hours is the same regardless of W/C. To keep the rate of release of heat of hydration on the lower side the fineness of cement should be controlled. The excessive heat of hydration leads to the formation of cracks leading to the disintegration of cement and concrete matrix. Use of fine cement leads to decreased crack width whereas crack tortuosity to find path growth was increased.

(Divanedari and Eskandari-Naddaf, 2020)**,** (Nguyen et al., 2021) Cement hydration depends on spatial distribution, the shape of particles and the particle size distribution of cement. The hydration and property development of cement paste and mortars can also be 'tailored' by carefully controlling the PSD of cement and the relative sizes of the cement and relative sizes of the cement and mineral additives.

The mechanical properties of cement-based materials are inversely proportional to porosity. (Liu et al., 2022) (Cao et al., 2019), Researcher reviews the effect of particles sizes on cement matrix and states that macro particles act as only fillers, microparticles not only fill the voids but accelerates the hydration process thus affecting workability, mechanical property, and durability through dilution and nucleation. However, nano-sized particles agglomerate and reduce their overall efficiency hence despite their faster reactivity their use is discouraged. Hence it can be concluded that amongst all sizes micro particles serve the purpose of enhancement of strength. Some researchers also attempted the use of Ultra-fine powders were used to fill microvoids and improve the particle packing density of the cementitious system and its mechanical property and durability.

As observed from various experimental data, coarser cement exhibit lower compressive strength than finer cement at all ages of curing. Also, coarser cement exhibits a lower risk of cracking at an early age, the reason can be attributed to lower compressive stress developed. Due to coarser particles, the system develops a compressive stress thus exhibiting lower residual stress development and a lower risk of cracking at early ages compared to the fine cement system. Finer cement generally will increase both thermal and autogenous deformation contribution to early age cracking.

(Bentz et al., 2008) **and** (Ardoğa et al., 2019) explained the fact that a higher specific surface area of cement leads to decreased porosity of ITZ hence higher compressive strength. The hydration and property development of cement paste and mortar can also be tailored by carefully controlling the PSD of cement and the relative sizes of the cement and mineral additives. It was also observed that Increased fineness leads to an increased volume of hydrates thus higher early strength

2.8 Effect of cement fineness on the heat of hydration

W/C for High-performance concrete is kept lower however in the course of duration after the initial stage of hydration of cement water which is not consumed in the hydration process evaporates. This evaporated water creates empty pores and this pressure due to empty capillary voids induces shrinkage. Hence high-performance concrete is susceptible to early-age cracking. The induced capillary pressure is directly proportional to Relative humidity. As the cement paste hydrates capillary pore size reduces hence this pore space is no longer connected thus reducing permeability with the progress of hydration. The

disconnection of pores restricts the transportation of moisture. Gel pores which are smaller than 10nm are always filled with water/moisture at an RH value of 50%. As per the author for RH value lies between 50-100 the moisture is trapped in the pores, thus helping the hydration of cement**.** (Bentz et al., 1997)

2.9 High-density concrete

(Naqi and Jang, 2019) 50% of the world's OPC is consumed to make 11 billion tonnes of concrete per year and it is anticipated that by 2050 the demand for concrete would rise to 18 billion tonnes per annum.

(Özen et al., 2016) studied the properties of heavyweight concrete by using various heavyweight aggregates such as iron ore, steel mill scale, two types of barite, and steel slag and magnetite. Concrete specimens with different coarse aggregates content and constant W/C ratio were cast and tested for compressive strength, splitting tensile and bending strengths, moduli of elasticity, fracture energies, and linear attenuation coefficients. Aggregates less than 8mm were used since the size of aggregates affect the homogeneity of the concrete mix. Totally 6 different mixes were cast for experimental testing of the above said tests. A concrete mix containing Iron ore aggregates showed maximum mechanical strength as summarized in Table 2-1

Table 2-1: Mechanical and fracture properties of concretes tested by Suleyman Ozen Cengiz ,Sengul (2016)

Mixture code	Cylinder compressive strength (MPa)	Modulus of elasticity (GPa)	Splitting tensile strength (MPa)	Bending strength (MPa)	Fracture energy (J/m^2)	Linear attenuation coefficient (*) (1/cm)
CSM	78.6	42	6.9	9.2	156	0.265
CIO	130.8	60.8	8.4	10.7	101	0.255
CB1	79.1	33.9	4.9	6	72	0.244
CB2	57.9	34.5	3.8	5	81	0.239
CSI	98.4	51.4	7.2	9.5	159	0.224
CMA	80.7	51	–	8	156	-

As per the author Iron ore had a comparatively lesser amount of Fe_2O_3 as compared to magnetite, still mill scale, and steel slag, however regular particle size of iron ore was the reason attributed to increased compressive strength. Results summarized also show that aggregates containing Fe_2O_3 show higher modulus of elasticity as compared to other aggregates. It can also be observed that the modulus of elasticity increases with the grade of concrete. The presence of Fe_2O_3 also affects splitting tensile strength and bending strength. It is also observed that the linear attenuation coefficient increases with increasing unit weight of concrete.

(Esen and Doğan, 2018) investigated various properties of concrete made using barite aggregates. In this paper mechanical and physical properties of concrete specimens produced by replacing natural coarse aggregate with barite aggregate at 0, 10, 20, 30, 40 and 50% by volume were investigated. Specimens were cast to determine the unit weight, water absorption percentage, modulus of elasticity, ultrasound pulse velocity (UV), Schmidt hardness (SH) and compressive strength.

Compressive strength increases at 10% of barite content however as barite content increases further strength decreased. Results of the compressive strength test have been graphically represen**ted in** Figure 2-5. It was observed that 10% barite content showed a maximum compressive strength of 59.68MPa. Tensile strength also showed a similar trend as compressive strength i.e. Maximum value of 9.23MPa at 10% of barite content and decreased further with an increase in barite content in concrete. However, unit weight and modulus of elasticity increased with barite content. One of the important parameters was thermal conductivity which was tested for 100% replacement of natural aggregates by barite aggregates. An increase of 16% in thermal conductivity was observed due to the addition of 100% barite content. Hence it was concluded by the author that thermal conductivity and density are directly proportional.

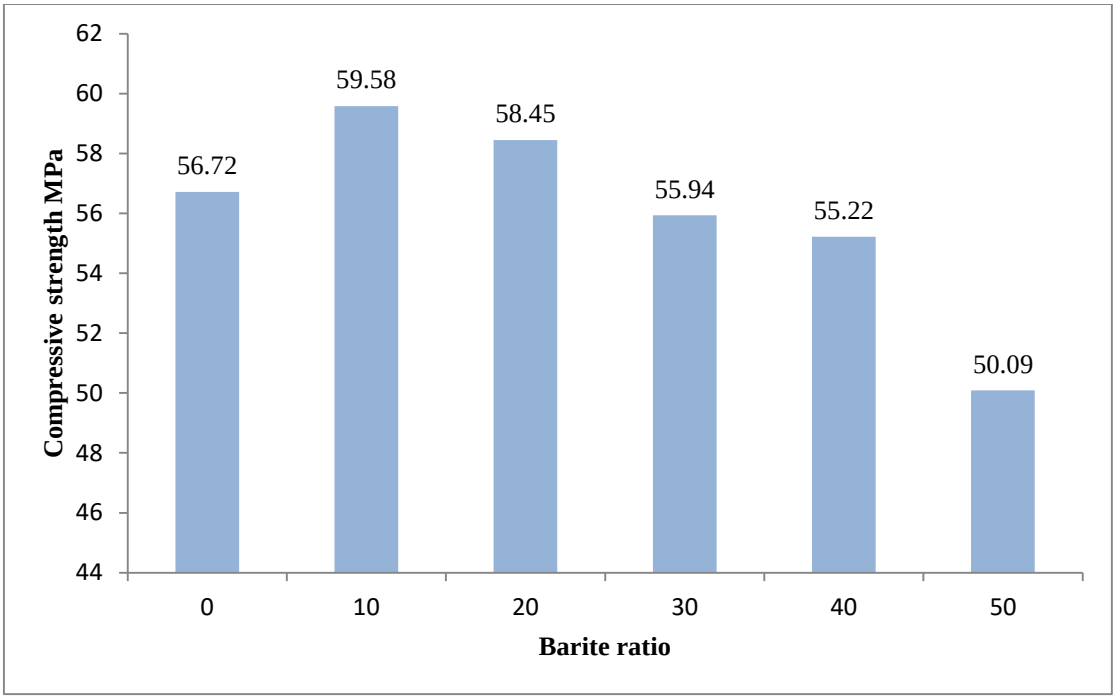


Figure 2-5: Compressive strength result with varying barite ratio observed by Yuksel Esen

It was found that unit weight, modulus of elasticity, ultrasound pulse velocity (UV) and thermal conductivity coefficient values of 400 dosage barite aggregated concrete with a W/C ratio of 0.60 increased in parallel to the increase in barite ratio; however Schmidt hardness (SH), water absorption (%), compressive strength and tensile strength values were found to decrease with increasing barite content.

(Janis Kazjonovs, Diana Bajare, 2014) designed heavy-weight concrete using steel waste by replacing natural aggregates with 50 and 100%. Iron dross which is obtained as waste during the steel rolling process and steel punching in cylindrical form was used. Two concrete mixes were prepared with 50% and 100% steel treatment waste as a replacement for natural aggregates. Results of density, water absorption and open porosity are given in Table 2-2 below.

Table 2-2: Physical Properties of concrete mixes used by Janis Kazjonovs etal (2014)

Property	**S50**	**S100**
Density(kg/m^3)	3520	4640
Open porosity %	4.9	5.1
Water absorption %	1.4	1.1
Compressive strength, MPa		
7days	35.4	32

	28 days	40.7	36.6
Tensile strength, MPa		4.1	4.15

The density of concrete mix increased due to the inclusion of steel treatment waste to 4640kg/m^3 However, increasing steel treatment waste led to decreased compressive strength. This decreased compressive strength may be due to the smooth surface of steel punching creating a weak bond between cement paste and aggregates. Along with compressive strength slump also decreased with increased steel waste. Apart from compressive strength inclusion of 100% steel treatment waste produced concrete with more resistant towards freeze and thaw.

(Yu-Cheng Kan, Kuang-Chih Pei, 2003) carried out a study on the strength and fracture toughness of heavy concrete with various iron aggregates. In this study, iron ore and iron shot are adopted as concrete aggregates. Iron ore with a specific gravity of 4.8 and iron shot with a specific gravity of 7.4 were used as aggregates. The maximum particle size used was 9.5mm. Specimens for 5 concrete
mixes with varying aggregates content were cast viz. 0, 10, 20, 30, 40, 48.8% to test compressive strength, splitting tensile strength, modulus of elasticity, rupture modulus, bond strength, fracture toughness and wave velocity. Table 2-3 shows the details of concrete mixes which were used in the study.

Table 2-3: Physical Properties of concrete mixes studied by Yu-Cheng Kan etal (2004)

Mix	**I**	**II**	**III**	**IV**	**V**
Aggregate content (%)	0	10	20	40	48.8
Compressive strength (MPa)	51.6	50.6	50.9	54.4	51
Tensile strength (MPa)	3.42	2.9	2.98	2.49	2.42
Modulus of elasticity (GPa)	20.9	25.9	26.9	34.2	31.1
Rupture modulus (MPa)	6.84	8.05	6.84	6.91	6.84
Bond strength (MPa)	10.4	12.1	8.46	9.35	10
Wave velocity (m/s)	3580	3458	3385	3225	3245
Fracture toughness (MN/m^3/2)	0.986	1.031	1.012	1.105	1.041

The addition of iron aggregates did not show a significant increase in compressive strength. Maximum compressive strength of 54.4MPa was obtained at the replacement of natural aggregates with 40% metallic aggregates. Like compressive strength, splitting

tensile strength experienced no significant change due to the addition of metallic aggregates. As aggregate content increased wave velocity decreased thus depicting increased air content with iron content in the mix. The modulus of elasticity of concrete obtained was 1.5 times that of the controlled mix. The reason for the lesser effect of the addition of metallic aggregates on compressive strength was attributed to the crack pattern of specimens subjected to compressive loading. Crack of failed specimens occurred along the aggregates rather than through as shown in Figure 2-6.

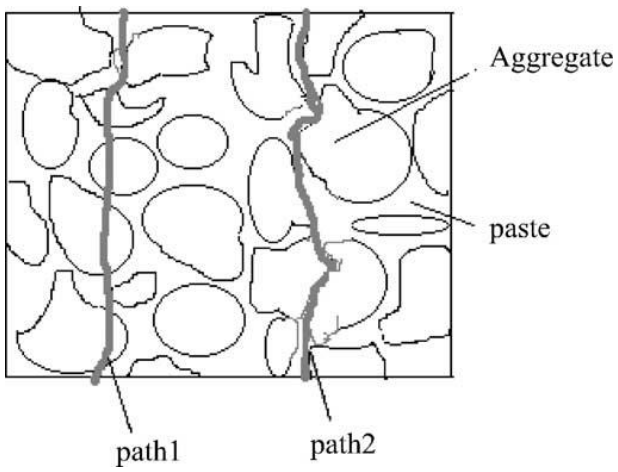


Figure 2-6: Crack pattern of failed specimen studied by Yu-Cheng Kan etal (2004)

(K. SAKRA, 2005) studied the effect of the addition of rice husk ash and silica fumes to heavy-weight concrete. Specimens for 3 different concrete mixes were cast and tested for compressive strength, permeability, Infrared properties and thermal investigation. In general, it was observed that density was less affected by the addition of rice husk ash and silica fumes however it changed with the type of aggregates. The ilmenite concretes showed higher compressive strength, indirect tensile strength, flexural strength, bond strength, modulus of elasticity, and attenuation coefficient values as compared to the barite and gravel concretes.

Microstructural investigation showed that the addition of silica fumes has better results than the addition of rice husk ash. However, the addition of either silica fumes or rice husk ash has not had much effect on the concrete attenuation coefficient. Infra-red investigation showed that Ilimenite concrete is much denser than barite and gravel concrete. The addition of silica fumes and rice husk ash showed a significant effect on the densification of concrete. Thermal investigation studied weight loss due to increased temperature of concrete specimens. With the increase in silica fumes and rice husk ash content weight loss observed decreased due to decreased porosity.

Table 2-4: Mechanical Properties of concrete mixes observed by K. Sakra, E (2005)

Type of concrete	Mechanical properties				
	Compressive strength (Fc) [MPa]	Tensile strength (Ft) [MPa]	Flexural (bending) strength (Ff) [MPa]	Bond strength (Ff) [MPa]	Modulus of elasticity [GPa]
Gravel	44	2.55	3.43	5.4	20.79
Baryte	47	2.85	4.42	5.89	29.03
Ilmenite	51	3.53	5.4	6.87	34.43

(Ouda, 2015) carried out experimentation using 4 different heavyweight aggregates procured from different sources namely magnetite(3.48), barite(4.04) and goethite(2.88) and Serpentine(2.79). Ternary mix constituting 10% SF, 20% FA and 30% GGBFS as a partial addition to OPC was used in this study. Overall 12 mixes were cast to determine Compressive strength, Density of concrete, and Radiation attenuation coefficient.

The density of concrete produced is directly proportional to the specific gravity of the aggregates used. concrete specimens made of barite coarse aggregate with 10% SF (B1), 20% FA (B2) and 30% GGBFS (B3) as additives to OPC exhibited the highest values of density of fresh or hardened concrete. Compressive strength results show an increase in compressive strength with the curing period and concrete mixes M1, M2 and M3 (containing magnetite aggregate) are significantly higher than those containing barite, goethite and serpentine at the age of 7 days.

(Gökçe and Andiç-Çakır, 2019) studied bleeding characteristics of high-consistency heavyweight concrete mixtures. Samples for 35 concrete mixes were cast and tested for compressive strength, 28-day water absorption, density and fresh concrete properties. Cement content was varied in all mixes i.e. 350, 450 kg/m^3. An increase in cement content showed a decrease in compressive strength and water absorption values however density decreases with cement content. Silica fumes showed a significant effect on the reduction of bleeding of concrete.

Bleeding is affected by many properties of concrete such as cement content, W/C and mineral admixture. It was observed that with an increase in cement content bleeding decreased. Results of bleeding characteristics showed that a decrease in the W/C ratio decreased the bleeding rates. Silica fume showed a remarkable increase in strength by up to 54% at high replacement levels. However maximum density was obtained for a mix containing 350kg/m^3 with a reduced water-cement ratio of 0.48.

(Topçu, 2003) studied the properties of heavy-weight concrete produced with barite. The experimental study of heavyweight concretes produced with barite is described in this section. Information about physical and mechanical experiments on heavyweight concrete is also provided. The physical, mechanical, and petrographic structure of the barite mineral that is used in heavyweight concrete production was examined with seven different W/C ratios and two different types of cement used in experiments. At the end of the experiments, the appropriate W/C ratio and cement dosage for heavyweight concretes were obtained. The mix design of concrete mixes used in this study has been shown in following Table 2-5.

Table 2-5: Amounts of heavyweight concrete composition used by Ilker Bekir Topc

W/C	Amount of water kg/m^3	Amount of cement kg/m^3	Amount of aggregates kg/m^3	Absorbed water kg/m^3	Slump in cm
0.3	105	350	3038	24.3	0.5
0.35	123	350	2967	23.7	1
0.4	140	350	2876	23	1.5-2
0.45	158	350	2835	22.65	2.5
0.5	175	350	2756	22	4-5.0
0.55	193	350	2683	21.46	6-7.0
0.6	210	350	2615	20.92	7

Various specimens were cast using the above mixes and their results obtained are shown in Table 5. The compressive strength obtained in mechanical experiments reveals sufficient values for aggregates by using barite as an aggregate in concretes. The unit weight of produced concretes was 3203–3288 kg/m^3 at PKC 32.5 and 3278–3359 kg/m^3 at PC 42.5. The unit weight of heavyweight concrete was 25% higher than the unit weight of normal concrete. Slumps were not high enough without the use of a plasticizer. With the addition of plasticizer, the workability of the concrete improved. It was observed that when the W/C ratio increased, the compressive strength decreased. It can be observed that said that heavyweight concretes obtained from PC 42.5 and PKC 32.5 and from 0.40 W/C ratios have properties of high compressive strength and appropriate workability.

Table 2-6: Mechanical Properties of concrete mixes studied by Ilker Bekir Topc

Cement type	W/C ratio	Unit weight (kg/m^3)	Resonance frequency (kHz)	Ultrasound duration (ms)	Schmidt hardness	Compressive strength (MPa)
PKC 32.5	0.3	3288	3.08	68.9	36	31.8
	0.35	3280	3.1	69.1	34	31.1
	0.4	3272	3.1	68.2	34	31.2
	0.45	3247	3.09	69.4	35	29.2
	0.5	3227	3.13	70.5	35	28.6
	0.55	3220	3.14	70.8	33	27.6
	0.6	3203	3.14	70.6	33	26
PC 42.5	0.3	3359	3.1	69.1	44	40.1
	0.35	3356	3.11	69.5	43	39.4
	0.4	3346	3.11	69.5	42	42.6
	0.45	3322	3.13	69.9	40	35.9
	0.5	3309	3.15	70.8	40	35.6
	0.55	3296	3.15	71	39	33.8
	0.6	3278	3.16	71.2	39	32.5

As the W/C ratio increases, because of increased voids of concrete, ultrasound duration also increases. Therefore it can be concluded that as the W/C ratio increases porosity increases.

2.10 Research Gap

Extensive research has been done on modifying the fineness of cement to improve its compressive strength of it at an early and later age of its curing. However, earlier studies carried out had certain shortcomings viz., increased water requirement with fineness, increased cost of production due to grinding and issues of sustainability. Efforts were also made to change the chemical composition rather than engineering PSD. Hence there is a need to address the issues as reported earlier without cost implications or with marginal cost implications. More specifically, the research gap identified based on the literature review is:

1. Micro fining is not the solution to be looked at but the engineering of PSD needs to be addressed in the improvement of cement quality.
2. Observations generally confirmed that water content increases with the fineness. Referring to the Figure 2-2 and with the support of literature there exists a research

gap on the right part of the curve to keep the same water content for higher fineness.

3. Optimization of Engineered PSD to bridge the gap in the improvement of cement quality in terms of performance.
4. With the enhanced fineness to suite predetermined stimulator to impact the sustainability aspects and to improve the performance of cement at no cost addition.
5. Contribution towards high strength high density concrete with improved PSD is the need of the industry.

However, based on the gaps the work is proposed to be undertaken to address the issues. Accordingly, the next Chapter deals with materials used for the experimental study under the title materials.

Chapter 3

Materials

3.1 General

The quality of the materials always plays an important role in deciding the quality of concrete. Codal provisions need to be followed while selecting the materials. This Chapter deals with a detailed description of the materials used in the study and properties of the same.

1. Binders
    a. Ordinary Portland Cement
    b. Stimulator
2. Water
3. Standard sand for mortar testing
4. Fine and Coarse aggregates for concrete
5. Activator
    a. Dynamon
    b. Endura
6. Curing Compound

3.2 Binders

Ordinary Portland cement of 53 grade of ACC make and 43 grade of JK cement make was used. This cement was blended with the stimulator of a known fineness. Commercially available OPC is ground to a fineness of particles ranging up to 25 microns and is termed a “Stimulator”. A jet mill was used for grinding. The property of the material is presented in Table 3-1 and diagrammatically in Figure 3-3 and Figure 3-4 respectively.

IS 269:2015
CM/L 2023022

ACC
ACC Limited
Wadi Cement Works
WORK TEST CERTIFICATE

IS / ISO 9001
QMS

53 Grade Ordinary Portland Cement

Despatched during Week No.		3	From	15-01-2019	To 21-01-2019
Test Certificate no.		3	Reference No.		WCW/IMS/QA/FM/08
Dated		29-01-2019			
S. N	Parameters	Results obtained	Requirements as per IS269: 2015 (Variety: OPC 53)		
	Chemical Composition				
	Lime saturation Factor (CaO - 0.7*SiO2) /(2.8*SiO2 + 1.2 Al2O3 + 0.65* Fe2O3)	0.91	Not greater than 1.02 not lepss than 0.8		
	Ratio of % Alumina to that of Iron Oxide Al2O3/ Fe2O3	1.28	Not less than 0.66		
	Insoluble Residue (% by mass)	0.5	Not more than 5.0 %		
	Magnesia (% by mass)	1	Not more than 6%		
	Sulphuric Anhydride (% by mass)	2.9	Not more than 3.5%		
	Total Loss on Ignition	1.6	Not more than 4.0 %		
1	Chloride content (%)	0.01	Not more than 0.1% for general purpose and not more		
	Physical Analysis				
	Fineness				
2	Blaine's Specific Surface Area (m2/kg)	306	Not less than 225		
	Compressive strength (Mpa)				
	72 ± 1h (3 Days)	38	Not less than 27.0		
	168 ± 2h (7 Days)	47.5	Not less than 37.0		
3	672 ± 4h (28 Days)	Awaited	Not less than 53.0		
	Setting Times (Minutes)				
	Initial	150	Not less than 30		
4	Final	205	Not more than 600		
	Soundness				
	Le Chateliers Expansion (mm)	1	Not more than 10.00		
5	Auto Clave Expansion(%)	0.1	Not more than 0.8		
	Normal consistency (%)	27.25			
6	Temp. During Testing (°C)	27	(27 ± 2) °C		

The above cement complies with the requirement of IS 269: 2015 (Variety :OPC 53) for 53 Grade Ordinary Portland Cement

Manager (QA)

Figure 3-1 Certificate of ACC 53 grade

IS 269 :2015

CM/L - 8217772

TC-6535

J.K.SUPER CEMENT
J.K.CEMENT WORKS-NIMBAHERA

J.K.Cement Works Quality Control Laboratories
Kailash Nagar No.1- 312617
NIMBAHERA, Dist.-Chittorgarh (RAJ.),
Phone-01477-220087, 220098, 220562, E-mail: jkc.nbh@jkcement.com

INTERIM TEST REPORT

Description: 43 Grade Ordinary Portland Cement as per IS: 269: 2015

Customer Name & Address: Packing Plant JKCW-NBH/Marketing — **Sample ID: 430PC/w35/53**

Unique Lab Report: TC 65351900000000 103P — **Sample received Date: 02.09.2020**

Date of eprformance of Testing : 02.09.2020 to 09.09.2020 — **Date of reportoing: 09.09.2020**

Location of Test Performed: Chemical Lab & Mechanical Lab ɔtion of sample: 24kg (Approxi)

The test report pertains to the average sample of week no. 35 (26.08.2020 to 01.09.2020)

CHEMICAL ANALYSIS OF CEMENT

S. N	Sample test Type	UOM	Test Results	Requirements as per IS269: 2015	IS METHOD
1	LOI (% by mass)	(%)	3.06	Max 5.00	IS 4032:1985
2	Insoluble residue (% by mass)	(%)	2.94	Max 5.00	IS 4032:1985
3	SO_3 (% by mass)	(%)	2.56	Max 3.5	IS 4032:1985
4	MgO (% by mass)	(%)	0.81	Max 6.0	IS 4032:1985
5	Na_2O (%)	(%)	0.12	-	IS 4032:1985
6	K_2O	(%)	0.46	-	IS 4032:1985
7	Chloride (% by mass)	(%)	0.02	Max 0.1	IS 4032:1985
8	Lime saturation Factor	(%)	0.86	0.66 . 1.02	
9	A/F		1.42	Max 0.66	
10	Performance Improver (Lime stone) by	(%)	4.96	Max 5.00	
MECHANICAL TESTS OF CEMENT					
1	Fineness	m^2/Kg	291	Min 225	IS 4031 P - 2:1999
2	Standard Consistency (%)	(%)	28	-	IS 4031 P - 4:1988
3	Soundness	(%)			
a	Le Chateliers Expansion (mm)	mm	1	Not more than 10.00	IS 4031 P - 3:1988
b	Auto Clave Expansion(%)	(%)	0.03	Not more than 0.8	IS 4031 P - 3:1988
4	Setting Times (Minutes)	(%)			
	Initial Setting Time	Minutes	140	Min 30	IS 4031 P - 5 1988
	Final Setting Time	Minutes	180	Max 600	IS 4031 P - 5 1988
5	Compressive strength (Mpa)	(%)			
	72 ± 1h (3 Days)	(%)	33.5	Min 23	IS 4031 P - 6 1988
	168 ± 2h (7 Days)	(%)	43	Min 33	IS 4031 P - 6 1988
	672 ± 4h (28 Days)	(%)	UT	Min 43 and Max 58	IS 4031 P - 6 1988
6	Normal consistency (%)	(%)	28		

Remarks:
1. Environmental Condition: Temperature: - 27±2°C; Humidity: - Not less than 65%
2. The results listed refer only to the tested sample(s) and parameter(s).
3. This report is not to be reproduced wholly or in part and cannot be used as evidence in the Court of law and should not be used in any advertising media without our special permission in writing.
4. ** Not in NABL Scope
5. ULR- Unique Lab Report.
6. Humidity for Fineness 65% Max

Authorized Signatory
P.K. SANCHETI
(TM / QM)
(Chemical & Mechanical Section)

Figure 3-2 Certificate of JK Cement 43 grade

Table 3-1: Properties of OPC and Stimulator (S)

	OPC	Stimulator
Physical Properties		
Specific Gravity	3.15	3.15
Blaine's Fineness, cm^2/g	3650.3	5390
Normal Consistency, NC (%)	28	41.25
Initial Setting Time, IST, Minutes	160	70
Final Setting Time, FST,Minutes	270	210
Chemical properties		
SiO_2 (%)	20.5	20.7
Al_2O_3 (%)	6.08	5.7
Fe_2O_3 (%)	3.34	3.82
CaO (%)	57.5	59.1
MgO (%)	4.23	5.61
SO_3 (%)	2.67	2.34
K_2O (%)	1.4	0.9
Na_2O (%)	0.09	0.18
SUM (%)	95.9	98.5
LSF (%)	83.1	85.4
SIM (%)	2.18	2.18
ALM (%)	1.82	1.49



Figure 3-3 : OPC used



Figure 3-4 : Stimulator used

3.3 Water

Water is the most crucial material used in mortar and concrete which controls and affects fresh and hardened properties of concrete. As per IS 456-2000, water used shall be free from any impurities and its pH should not be less than 6. The water used was confirmed to the standards prescribed.

3.4 Fine Aggregates / Standard Sand for Mortar Testing

Standard sand of specific gravity 2.46 is used. Standard sand used confirms to IS 650-1991and ASTM C778 It was used in equal proportions as per Indian standard codes. Figure 3.5 presents a pictorial view of standard sands used in the mix proportions of mortar for testing. The sand of 33.33% by weight was used passing through a 2-mm IS sieve having a specific gravity of 2.46, chlorides content nil, free from organic impurities, having loss of mass when washed with hot hydrochloric acid was not exceeding 0.25%.

The sand of 33.33% by weight was used passing through a 1-mm IS sieve having a specific gravity of 2.46, chlorides content nil, free from organic impurities, having loss of mass when washed with hot hydrochloric acid was not exceeding 0.25%.

The sand of 33.33% by weight was used passing through 500microns retained on a 90microns IS sieve having specific gravity 2.46, chlorides content nil, free from organic impurities, having loss of mass when washed with hot hydrochloric acid was not exceeding 0.25%.



(a) 2mm



(b) 1mm



(c) 500microns-90microns

Figure 3-5 : Standard sand

3.5 Fine and Coarse aggregates for concrete

Aggregates used for casting concrete ad high-density concrete samples are described in this sub-topic. The properties of aggregates used in the casting of normal concrete and high-density concrete samples have been presented in Table 3-2. The aggregates used in the casting of normal concrete are of natural origin however for high-density concrete samples waste products obtained from mine waste are incorporated. The pictures of the above-mentioned aggregates have been presented in Figure 3-6, Figure 3-7 and Figure 3-8. Figure 3-9, Figure 3-10 and Figure 3-11 shows the gradation curve derived from sieve analysis for Iron Power, Mill scale waste and fine aggregates respectively.

Table 3-2: Properties of aggregates used in concrete mix

Description	Coarse aggregates		Fine	Iron	Millscale
	20mm	10mm	aggregates	powder	waste
Specific gravity	2.88	2.87	2.75	4.38	3.97
Water absorption (%)	1.09	1.53	3.15	1.5	2.5



Figure 3-6 Mill scale



Figure 3-7 Iron powder

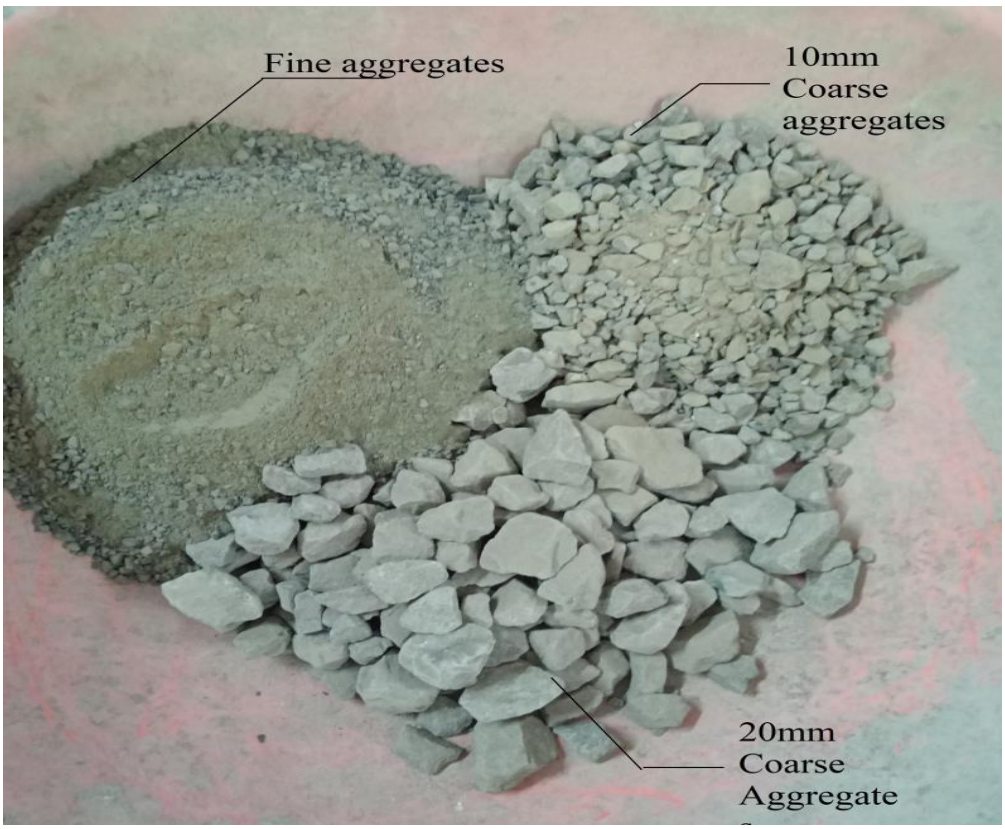


Figure 3-8: Coarse and fine aggregates used in concrete

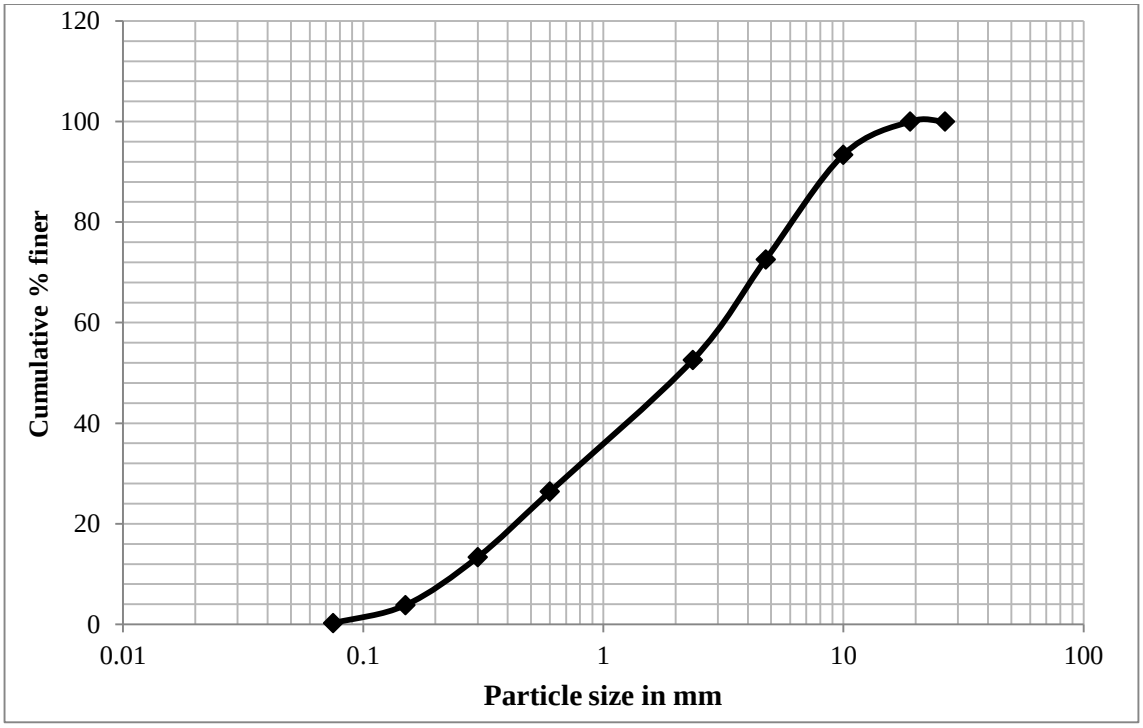


Figure 3-9: Gradation curve of Iron powder

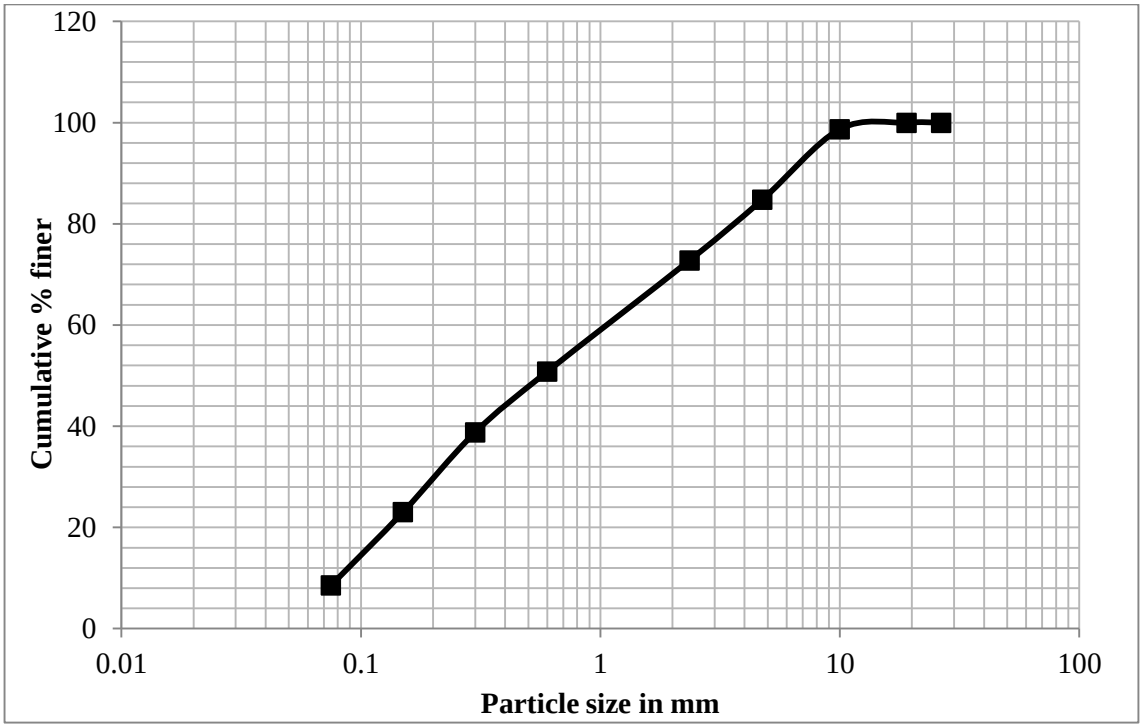


Figure 3-10: Gradation curve of Mill scale waste

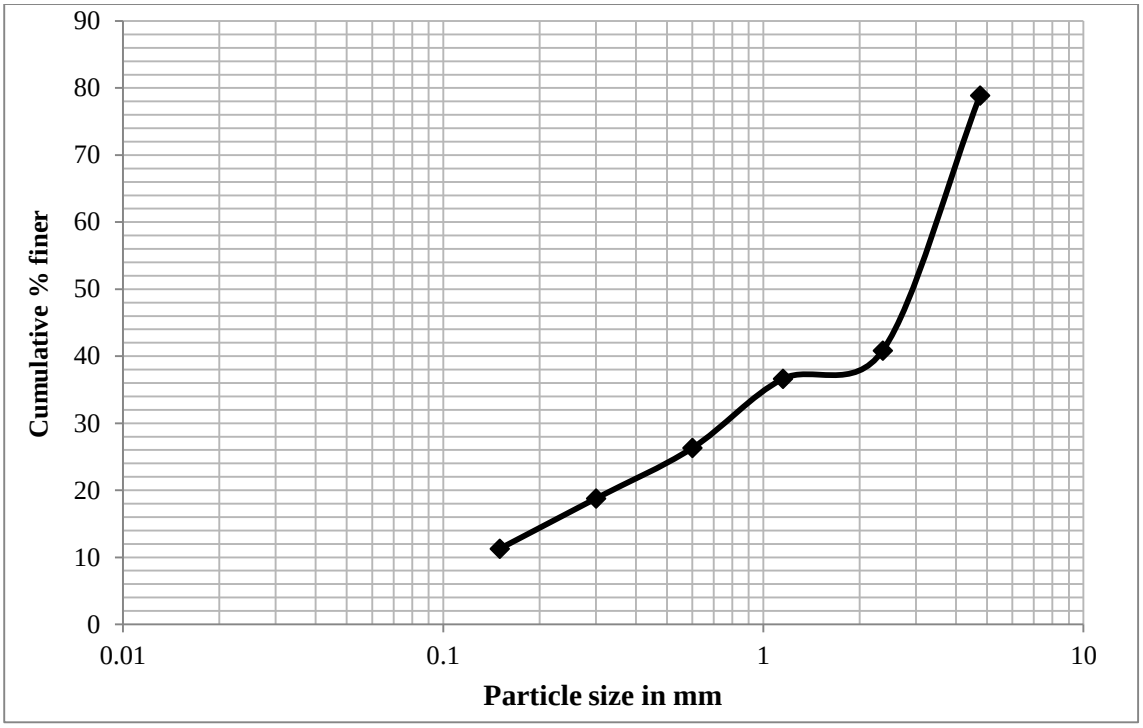


Figure 3-11: Gradation curve of Mill scale waste

3.6 Activators

1. Dynamon SX-636:

A high-end PC admixture of make MAPEI was used as an activator. The properties are listed in Table 3-3.

Table 3-3: Properties of Dyanamon SX 636

Property	Description
Consistency	Liquid
Colour	Amber
Density according to ISO 758(g/cm^3)	1.07±0.02 at +20° C
Main Action	Increase workability and/ or reduction of mixing water
Classification according to EN 934-2	High range water reducing superplasticizer, table 3.1 and table 3.2
Classification according to ASTM C 494	Type F
Classification according to ASTM C 107	Type I
Chorides soluble in water according to EN 480-10(%)	<0.1 (Absent according to EN 934-2)
Alkali Content (Na2O equivalent) according to EN 480-12(%)	<2.5
pH Content according to ISO 4316	6.5 ± 1

2. **Endura**

A high-end PC admixture of make Endura Ardex was used as an activator. The properties are listed in Table 3-4.

Table 3-4: Properties of Endura Ardex

Property	Description
Appearance	White Emulsion
Specific Gravity	1.04
Total Solids	Approximately 48%
pH	4-8
PC	High end

3.7 Curing Compound

Curing compound MONOBOND or krishnaconchem is used in the study. It is a water base, quick-drying urethane-modified acrylic solution. The properties of curing compound are presented in Table 3-5.

Table 3-5: Properties of Curing Compound

Property	Description
Composition	Urethane Modified Acrylic
VOC	<100g/L
White Base Weight Solids	52% ± 2
White Base Volume Solids	38% ± 2
Clear Base Weight Solids	34% ± 2
Clear Base Volume Solids	31% ± 2
Weight per gallon	10.5 Lbs
Finish	Smooth Flat
Viscosity	85-95 Kus
Coverage	250-400 Sq.Ft per Gallon Recommended Dry Mis 1.5-2.5 Recommended Wet Mis 4-6
Drying time	Dry to touch- 30minute Dry to top coat- 2-3 hours

3.8 Mix proportions of concrete

Mix design of concrete samples carried out as per IS: 10262-2019. The proposed mix design is presented in Table 3-6. M25 concrete without using any admixture was designed. he test cubes were cast using OPC and OPC with a stimulator. The strength was found at 1,3,7 and 28 days. The results were compared with the control while analysing the strength parameters.

Concrete grade M40 was also designed. The concrete was also dosed with admixtures to find out the compatibility. The strength of such concrete was also established and compared for 1,3,7 and 28 days. Concrete samples of 2 grades M25 (Normal Grade) and M40 (High strength concrete) are designed and specimens are cast. Under both grades of concrete two different sets of concrete specimens are cast one using only OPC and the other using stimulated cement (with an optimum dosage of stimulator). For concrete mix 20mm and 10mm size coarse aggregates were used in the proportion of 50:50. Properties of aggregates used for concrete mix design are presented in Table 3-2.

Table 3-6: Mix design of normal concrete

Sr No.	**Material**	**M25**		**M40**	
		Conventional Mix	Mix with Stimulator	Conventional	Mix with Stimulator
1	OPC	330*	165	430	182
2	Stimulator	-	135	-	148
	Total Cementitous content (1+2)	**330**	**300**	**430**	**330**
	W/C	0.5	0.5	0.3	0.3
3	Water	165	150	129	99
4	Coarse aggregates, 20mm	563	800	566	690
5	Coarse aggregates, 10mm	563	350	566	500
6	Fine aggregates	830	850	837	910

Note: All units in kg/m^3

*-Minimum cement content as per IS 456-2000

3.9 Mix proportions of High-Density Concrete (HDC)

HDC was cast using the mix design procedure for M60. The optimum contents proposed are shown in Table 3-7.

Table 3-7: Mix design used for high-density concrete samples

Sr.No.	Materials	Trial I	Final Mix
1	OPC	550	302.5
2	Stimulator	-	247.5
	(1+2) Total Cementitous content	550	550
3	W/C	0.25	0.25
4	Water	138	138
5	Iron Ore/ Iron powder	2088	2088
6	Mill scale	820	820
7	Admixture	4.4	4.4

Note: All units in kg/m^3

The subsequent Chapter describes the methodology adopted.

Chapter 4

Methodology

4.1 Introduction

This Chapter describes the overall methodology of the entire study and a detailed description of every experiment conducted following Indian standards. The summary and the sequence of operations of the study are pictorially presented in Figure 4-2. The work has been presented under 2 categories viz., the tests on cement mortar and the test on concrete.

A. Tests on cement

1. XRF Analysis
2. PSD analysis using the LASER diffraction test
3. Water requirement of various cement mixes
    a. Flow table
    b. Normal consistency
4. Setting times
5. Heat of hydration
6. Compressive strength
7. Density of mortar cubes

B. Tests on concrete

1. Compressive strength
2. Durability tests
    a. Sorptivity
    b. Permeability
    c. Acid attack

4.2 Methodology of production of "stimulator"

Commercially available Ordinary Portland cement is ground into particles finer than 25µm is termed a "Stimulator". Grinding of OPC to produce a "stimulator" is carried out using a jet mill. The diameter of the jet mill is 300mm with an input feed size of 200 mesh which maintains a gas pressure of 7-8 kg/cm^2. The principle of micronization using a jet mill is to use a high-speed jet of compressed air, gas or high-pressure super-heated steam to impact particles into each other by collision. The jet mill has no moving parts, thus eliminating contamination due to contact with external grinding media.

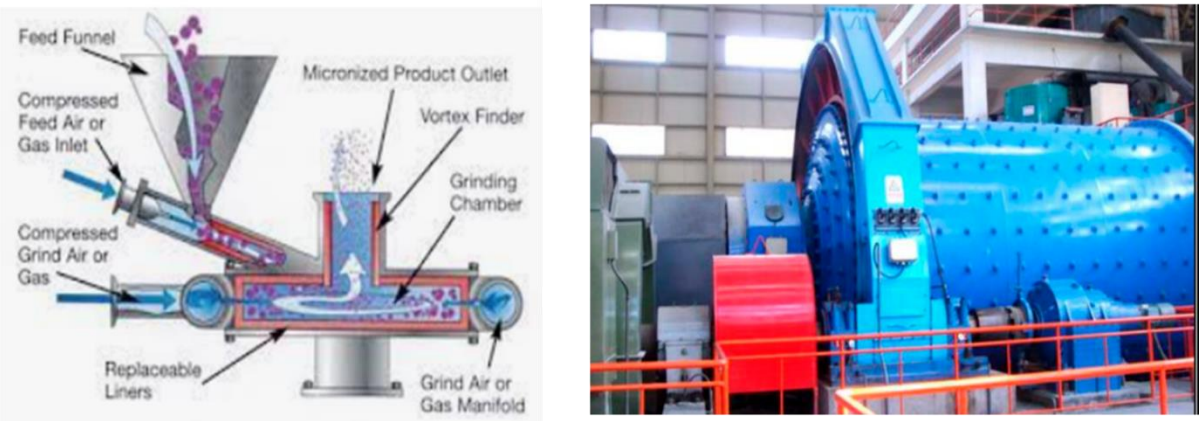


Figure 4-1 Jet mill and ball mill arrangement used for the production of micronization

Various combinations of OPC and stimulator used in the current experimental study have been presented in Table 4-1.

Table 4-1: Blending combinations of OPC and stimulator

Nomenclature	**OPC** (%)	**Stimulator** (%)
Pure OPC*	100	0
S	0	100
	Cement Mixes	
S10	90	10
S20	80	20
S30	70	30
S40	60	40
S50	50	50
S60	40	60
S70	30	70
S80	20	80
S90	10	90

*-Control Mix

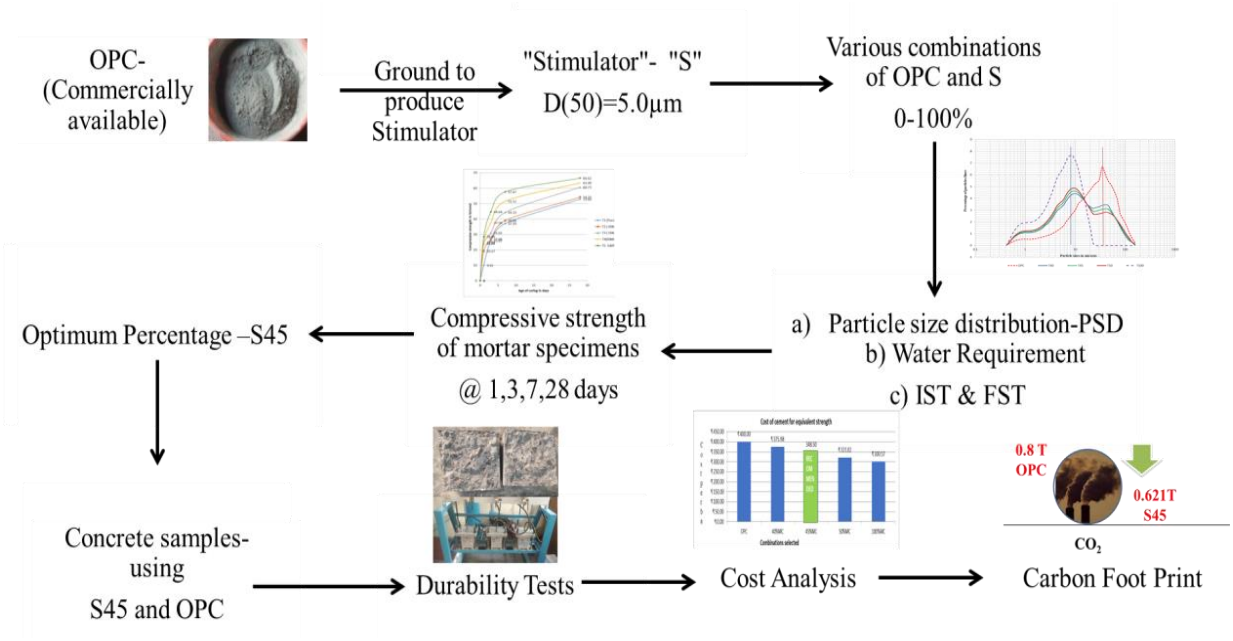


Figure 4-2: Sequential Methodology adopted for the experimental study

4.3 Tests on Cement and Mortar

Cement and mortar were subjected to the following tests to understand its behaviour for its analysis:

a. XRF Analysis
b. PSD analysis using the LASER diffraction test
c. Water requirement of various cement mixes
d. Flow table
e. Normal consistency
f. Setting times
g. Heat of hydration
h. Compressive strength
i. Density of mortar cubes

4.3.1 XRF analysis

This test was proposed to understand the structure and properties of cement and the stimulator. It confirms to IS: 12803. The results of the XRF test are discussed in subsequent articles and the corresponding experimental arrangement has been shown in Figure 4-3. The samples were dried and prepared as tablets by mixing them with wax powder.



Figure 4-3: XRF testing arrangement – *Courtesy JK Cement Plant*

4.3.2 PSD analysis using LASER diffraction technology

LASER diffraction technology is used to analyze the PSD of cement and stimulator. The principle of measurement of particle sizes is by noting angular variation in the intensity of light scattered after it passes through the dispersed particulate sample. The angle at which the LASER beam is scattered is inversely proportional to the individual sizes of cement grains. The test arrangement has been presented in Figure 4-4 which confirms ISO 13320 and ASTM-E 3340 standards (ASTM E3340-22, 2003) .The analysis permits the particles which are in the range of 0.1 – 1000µm. Analysis was repeated several times until a consistent value was obtained for the combinations referred to in Table 4-1.



Figure 4-4: LASER diffraction testing arrangement

4.3.3 Water requirement of various cement mixes

Water requirement is one of the important tests while analysing cement samples for their consistency and workability. Flow tests appear to be more rational as compared to consistency tests for higher finer cement in terms of Blaine. For a better interpretation of results, both methodologies were used.

A. Flow table method

Combinations proposed in Table 4-1 were analyzed for the flow table test. The tests enable the determination of water requirements to obtain a known flow value as per codal provisions IS 5512. The test set-up is pictorially presented in Figure 4-5. The mortars were prepared using a sand: cement mass ratio of 1:2.75 according to ASTM-C 124.



Figure 4-5: Flow table arrangement

B. Normal consistency

Combinations proposed in Table 4-1 were analyzed for the Normal consistency test. This confirms IS:4031 (Part 4) using Vicat's apparatus. The test is performed to determine the

quantity of water required to produce cement paste of standard consistency. The test set-up is presented in Figure 4-6.



Figure 4-6: Vicat's apparatus and testing arrangements

C. Setting times

It is one of the important tests conducted on cement to find its suitability for transportation and placement. The fineness of cement and chemical composition affects the tests. Setting times of cement affects many other related parameters such as workability, transportation time available, and use of admixture. The values of setting times are dependent on many factors such as the chemical composition of cement, the fineness of cement and the prevailing conditions at the time of casting. Setting times of the cement combinations stated in Table 4-1 are found by using Vicat's apparatus results which are presented in subsequent articles. Setting times are found by procedure confirming to ("IS : 4031 (Part 5)," 1988). The testing arrangement used to determine the setting time is presented in Figure 4-7.

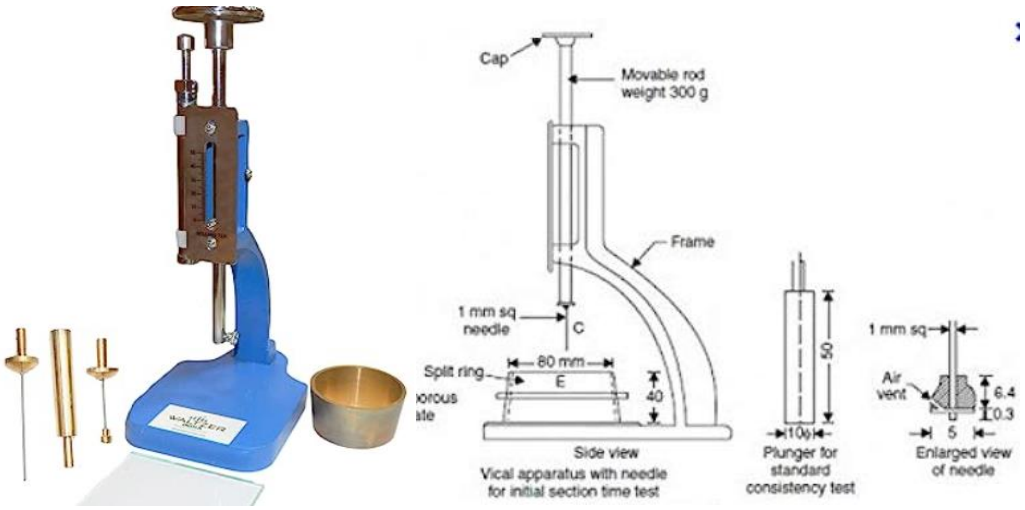


Figure 4-7: Testing arrangements – setting times of cement

D. Heat of hydration

The optimum combinations of OPC and stimulator were chosen based on the results of PSD, water requirement and set times. The values of the total heat of heat hydration were found as per IS 4031 (Part 9)(Indian, 2005) for OPC (S0), S40, S45, S50, S60, sample and pure stimulator sample (S100). Figure 4-8 shows the calorimeter used for determining the heat of hydration of cement samples.



Figure 4-8: Calorimeter used to determine the heat of hydration -Courtesy JK Cement laboratory

4.3.4 Compressive strength test on mortar

Compressive strength test is performed on 50mm cubical mortar specimens prepared following IS 4031- Part 6. The loading rate of the compression testing machine used ranges from 0.14-0.324 MPa/s. Standard sand confirming to IS 650-1991 and ASTM C778 and water confirming to quality stated in IS:456-2000 are used in the study. The specimens were left to set in a moist cabinet at 23 ± 2 ^{o}C immediately after casting and then cured after the final setting time by immersing in water at the same temperature as the moist cabinet. The compressive strength of the mortars was measured after 1, 3, 7 and 28 days of casting, using a Compression Testing Machine.



Figure 4-9: Cement mortar under compressive strength test

4.3.5 Tests on Concrete

1. Compressive strength test
2. Durability tests
   a. Sorptivity
   b. Permeability
   c. Acid attack

1. Compressive strength test

To determine the compressive strength of concrete, 150 mm cubes were prepared following IS: 516:2021 and the same were tested in Compression Testing Machine after 3, 7 & 28 days of casting. The equipment and machinery used for casting specimens and compression of concrete is shown in Figure 4-10.

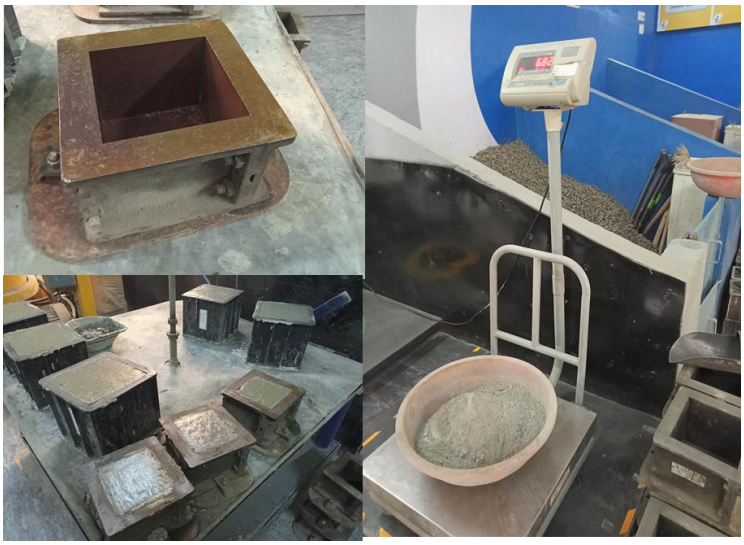


Figure 4-10 Equipment, moulds used for casting of specimens

2. Durability tests

3. Sorptivity Test

The sorptivity test was performed as per ASTM C 1585. The specimens of disk shape with 100mm diameter and 50mm thickness were prepared. The samples before testing were kept in an oven at a temperature of 50±2 ^{o}C and RH 80±3% for 3 days. The samples were covered with a thin film of epoxy on all surfaces except for the bottom. The sorptivity was measured at an interval as per ASTM C 1585. Testing arrangement of samples for determination of sorptivity is shown in Figure 4-11.

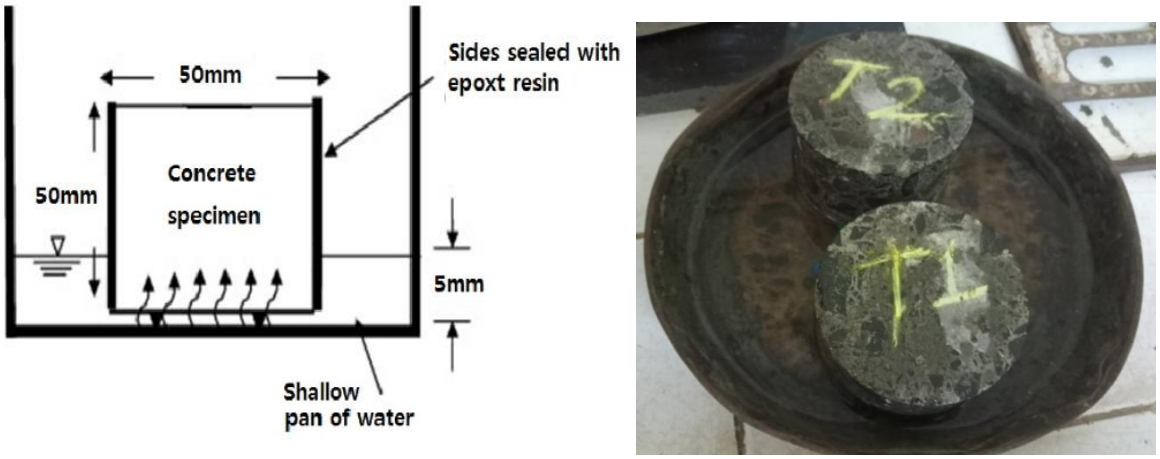


Figure 4-11: Schematic Diagram and experimental arrangement for Sorptivity test on concrete sample

4. Permeability Test

The Permeability of concrete is the critical parameter which affects durability. It can cause corrosion in reinforcement in the presence of oxygen, moisture, Carbon dioxide etc. The corrosion leads to spalling of concrete thus disturbing the integrity of concrete. Permeability tests are performed on concrete following IS 3085-1965. Concrete samples of grades M25 and M40 using OPC and S45 were cast for determination of permeability values. The concrete samples are cast as per Indian Standards and cured for 28 days before testing for permeability. The testing arrangement used for the permeability test and the samples under testing is shown in Figure 4-12.

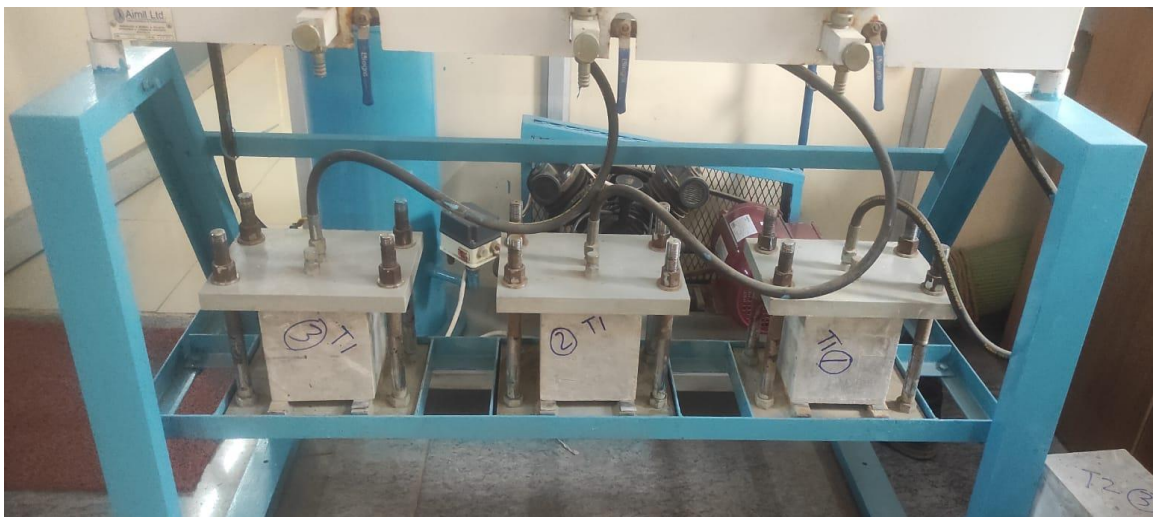


Figure 4-12: Permeability testing apparatus for concrete samples

5. Acid Attack Test

The acid attack test was performed on the concrete samples (150mm size) and observed for the mass loss after 30,60 & 90 days of acid exposure (pH=3) and compressive strength loss after 90 days of exposure (ASTM C-1898-20). Figure 4-13 shows pH value of water before and after addition of Sulphuric acid (H_2SO_4). The pH value 3.0 is to be maintained throughout the period of curing.



Figure 4-13 pH paper showing pH of water before addition of Sulphuric acid

The subsequent Chapter presents results and conclusions based on the study.

Chapter 5

Results and conclusions

Following the test procedures narrated earlier results of Ordinary Portland Cement were compared with stimulator-assisted cement samples. The properties are compared according to the results conducted on cement in the dry form and also for physical and mechanical properties listed under A. Tests on concrete specified under B were compared with conventional concrete and modified stimulator-assisted concrete.

A. Tests on Cement

5.1. XRF analysis

5.2. Particle Size Distribution

5.2.1. Particle size distribution for the combinations listed in Table 4-1

5.2.2. Summary of comparison of PSD

5.3. Water Requirement

5.3.1. Flow table method

5.3.2. Normal consistency value

5.4. Setting times

5.5. Heat of hydration

5.6. Chemical shrinkage

5.7. Compressive strength -Cement Mortar

5.8. Effect of Curing Compound

5.9. Effect of Activators

B. Tests on concrete

5.10. Compressive strength of Normal Concrete

5.11. Compressive strength of high-density concrete

5.12. Durability tests on concrete

5.12.1. Sorptivity

5.12.2. Permeability

5.12.3. Acid attack

A. **Tests on Cement**

5.1 XRF analysis:

Table 5-1 presents the comparison of chemical properties of OPC 53-grade and 43-grade cement. Stimulator-assisted cement of 43 and 53 grades are also listed.

Table 5-1: Percentage of Oxide content in cement used in the study

	SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**K_2O**	**Na_2O**	**SUM**	**LSF**	**SIM**	**ALM**
OPC 53 grade	20.58	6.08	3.34	57.53	4.23	2.67	1.4	0.09	95.93	83.11	2.18	1.82
OPC43 grade-JK cement	20.3742	6.01	3.30	56.95	4.18	2.64	1.386	0.09	94.97	82.27	2.15	1.80
100S-53 grade	20.76	5.7	3.82	59.17	5.61	2.34	0.9	0.18	98.48	85.46	2.18	1.49
100S-43 grade	21.13	6.33	4.22	55.64	4.09	2.97	1.43	0.19	96	77.18	2	1.5

$$C_3S = 4.07(CaO) - 7.60(SiO_2) - 6.72(Al_2O_3) - 1.43(Fe_2O_3)2.85(SO_3) \quad \text{Eq. 5-1}$$

$$C2S = 2.87\,(SiO_2) - 0.754(3CaO.SiO_2) \quad \text{Eq. 5-2}$$

$$C3A = 2.65\,(Al_2O_3) - 1.69(Fe_2O_3) \quad \text{Eq. 5-3}$$

$$C4AF = 3.04\,(Fe_2O_3) \quad \text{Eq. 5-4}$$

Equation 5.1 - 5.4 represents the chemical composition of cement also termed Bogue's compounds. PSD analysis of conventional and stimulator-assisted cement indicated the participation of particles in the process of hydration is in the proportion of the combinations S40 and S60, more specifically at S45. Accordingly, Table 5-3 presents the Bogue's compounds estimated for the combinations S40 - S60 along with controls OPC 53 grade and S100. Table 5-3 presents the Bogue's compounds estimated for the combinations S40 - S60 along with controls OPC 43 grade and S100.

Table 5-2: Percentage of Bogue's compound in every cement sample using 53 gr OPC

Cement sample	OPC-53 grade	S40	S45	S50	S60	S100
% of stimulator	**0**	**40**	**45**	**50**	**60**	**100**
C_3S (%)	24.5	28.47	29	29.53	29.366	32.61
C_2S (%)	40.59	37.51	37.195	36.88	37.23	34.99
C_3A (%)	10.47	9.73	9.56	9.39	9.378	8.65
C_4AF (%)	10.15	10.76	10.9	11.04	11.026	11.61
Silicates (%)	65.09	65.98	66.195	66.41	66.596	67.6
Aluminates(%)	20.62	20.49	20.46	20.43	20.404	20.26
C3S+C3A	34.97	38.2	38.56	38.92	38.744	41.26

Table 5-3 Percentage of Bogue's compound in every cement sample using 43 gr OPC

Cement sample	**OPC 43 grade**	**S40**	**S45**	**S50**	**S60**	**S100**
% of stimulator	**0**	**40**	**45**	**50**	**60**	**100**
C_3S (%)	12.11	14.61	14.40	14.19	13.78	16.28
C_2S (%)	49.34	48.76	48.81	48.86	48.95	48.37
C_3A (%)	10.35	9.93	9.96	10.00	10.07	9.64
C_4AF (%)	10.03	11.71	11.57	11.43	11.15	12.83
Silicates (%)	61.45	63.37	63.21	63.05	62.73	64.65
Aluminates(%)	20.38	21.64	21.53	21.43	21.22	22.47
C3S+C3A	22.46	24.54	24.36	24.19	23.84	25.92

Discussion

Referring to Table 5.2 and Table 5.3 it is noted that the stimulator and the conventional cements remain unaltered in their chemical composition even after fine grinding. The statement holds good for 43-grade cement too. However, very marginal changes have been noticed which is attributed to randomization of sampling. Figure 5.1 presents the compositions for the combinations considered for the analysis of Bogue's compounds.

Conventional cement and stimulator-assisted cement are chemically the same but they are different concerning PSD. Thus, particle size decides the rate of the hydration process rather than its chemical composition for a given grade of cement. It has been noted that the summation of C_3S and C_2S is constant for all the combinations

considered in Table 5.2. According to Talaber (J. Talaber, 1982), the summation of C3S and C3A is around 65% for the cement to be categorized as high early strength cement. However, in the present case, although the sum is in the range of 35 and 41% it has been witnessed to give high early strength and hence the same is attributed to the PSD but not for chemical composition.

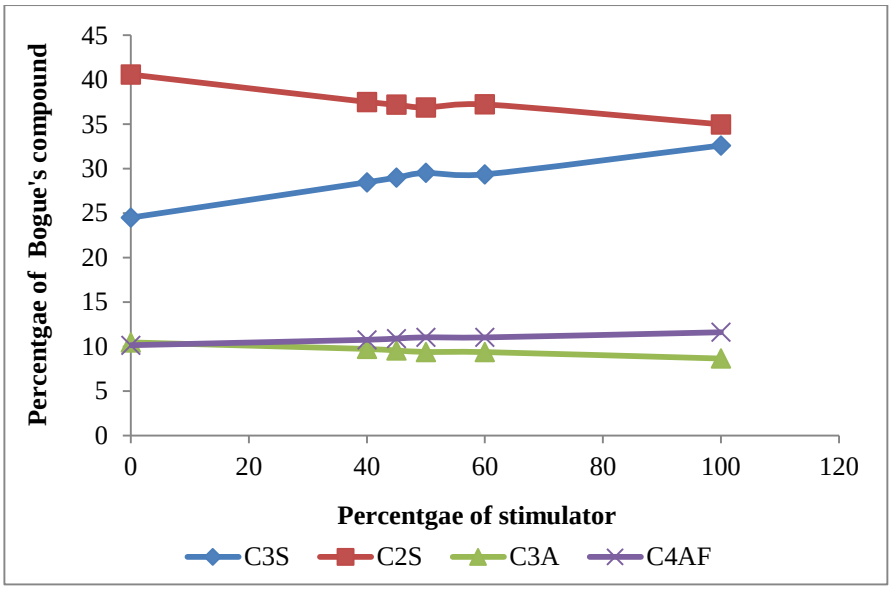


Figure 5-1: Relation between % of Bogue's compound and percentage of stimulator

5.2 Particle Size Distribution

Particle Size Distribution of cement was performed by using LASER diffraction technology. PSD of all the cement mixes stated in Table 4-1 has been presented in the following sub-article 5.2.1.

5.2.1 Particle size distribution for the combinations

PSD of the cement combinations proposed in Table 4.2 is presented in **Figure 5-2** to Figure 5-12 It has been noted that the range of particles is 0.417 – 158.49 microns. The addition of a stimulator will bring the median gradually towards the Y axis indicating the increase in fines percentage. It has been noted that the increments in finer content will also narrower the base of the bell-shaped curve in addition there is a reduction in the differential percentage of particles being lowered.

This concept is universal and applicable to any PSD of the samples. It is specifically observed that, the addition of fines led to the arrangement of particles based on PSD into two groups. A specific double peak arrangement at S30 begins and reaches its peak at S45. Further addition of fines beyond S45 gradually lowers the second peak and disappears beyond S60.

Given this, it is justified that the combination S45 is considered to be more efficient as compared to other combinations. Thus, the S45 combination is treated as an optimized combination.

PSD plays an important role in the process of hydration. Finer particles participate in hydration before the coarser particles. This fact is true for combinations up to S30 and beyond S60. The combinations up to S30 require lesser water for hydration as compared to the combinations beyond S60 which is solely attributed to the fineness. Having noted that for the combinations up to S30, the average particle size is higher and thus the finer particles participate first in the hydration process and the coarser particles remain un-participated. Hence the majority of the particles in this combination remain un-hydrated. In the case of the combinations beyond S60, the quantum of water required is more and the hydration process is active. Coarser particles remaining in the matrix work as fillers, however, they are either partially hydrated or not hydrated at all. These statements are true for the single peak systems of PSD. In the specific combinations between S40 and S60, sequential participation of the particles (both coarser and finer) is observed in the process of hydration. Thus, the strength gained is always higher in this range. The studies have shown the highest strength was observed for the optimized combination.

The analysis carried out and the statements made above clearly indicate that PSD is more important than the chemical composition of the cement. Further, particles of size less than 10 microns are hyperactive and the activeness reduces as the size grows. Several authors have indicated the addition of fines or fine grinding techniques is adopted for high-strength cement. This appears to be an expensive process. The specific combination at S45 satisfies the balancing of particles leading to the participation of the entire matrix of cement in the process of hydration with the addition of water. For any other given combination other than S40 – S60 the matrix does not participate in the process of hydration fully.

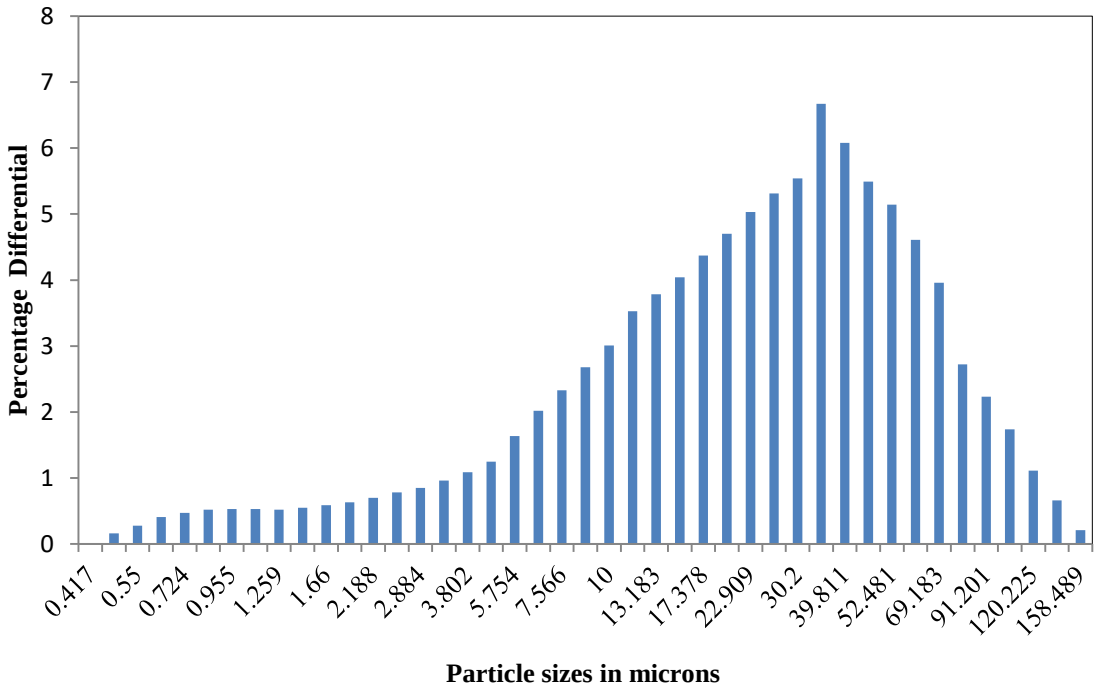


Figure 5-2: Particle size distribution of Ordinary cement sample

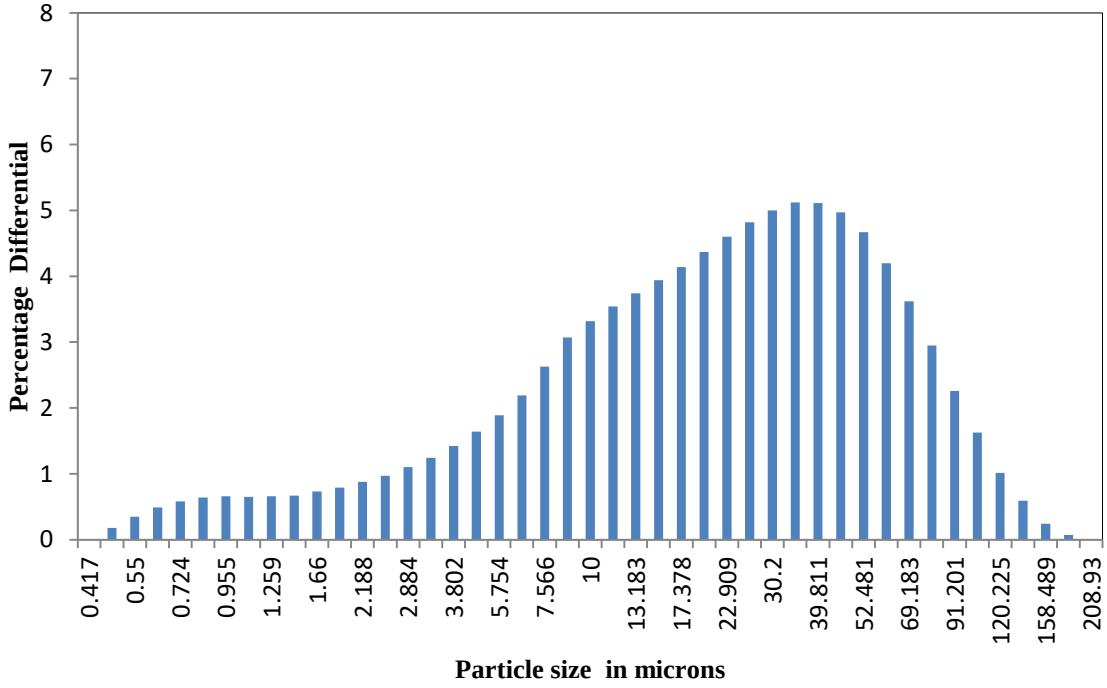


Figure 5-3: Particle size distribution of S10

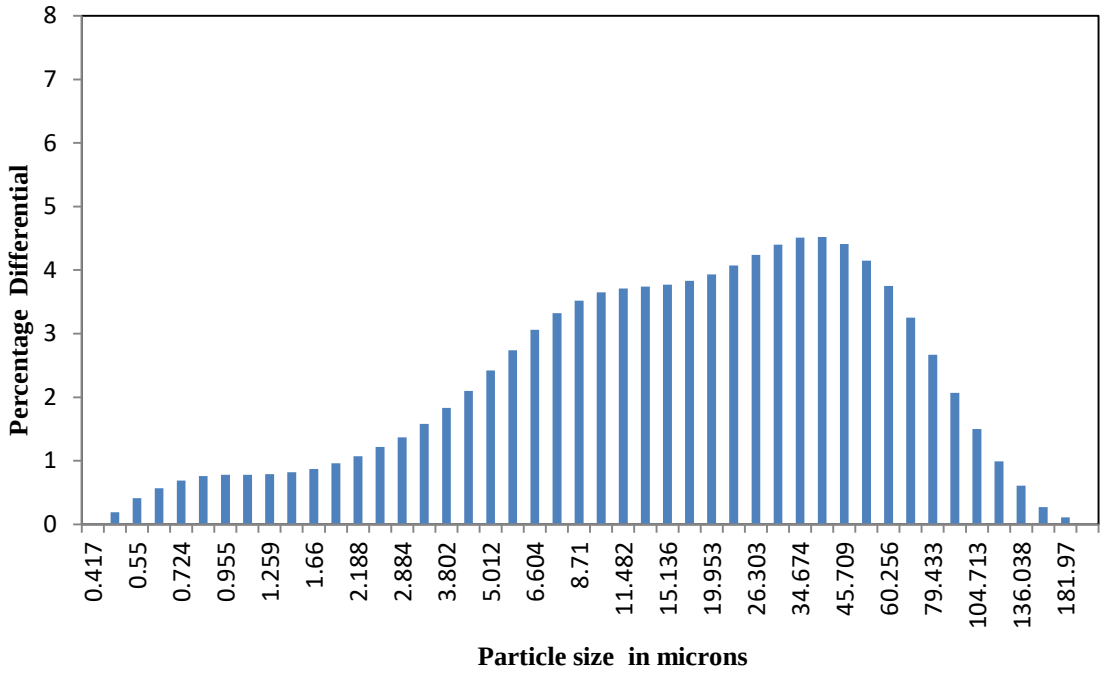


Figure 5-4: Particle size distribution of S20

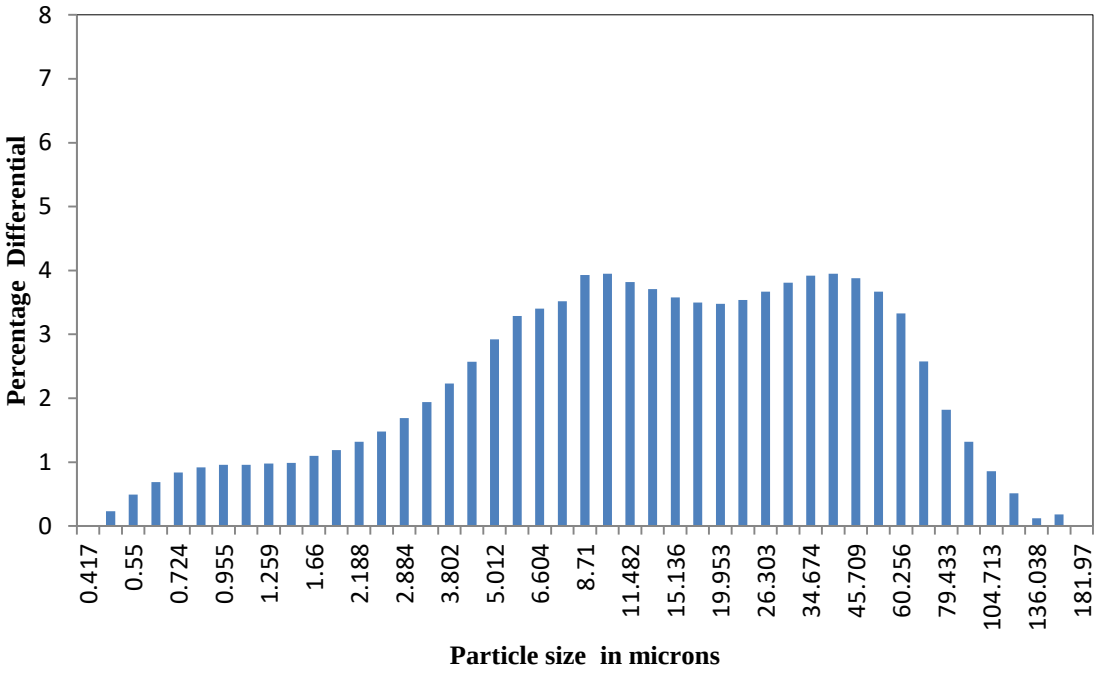


Figure 5-5: Particle size distribution of S30

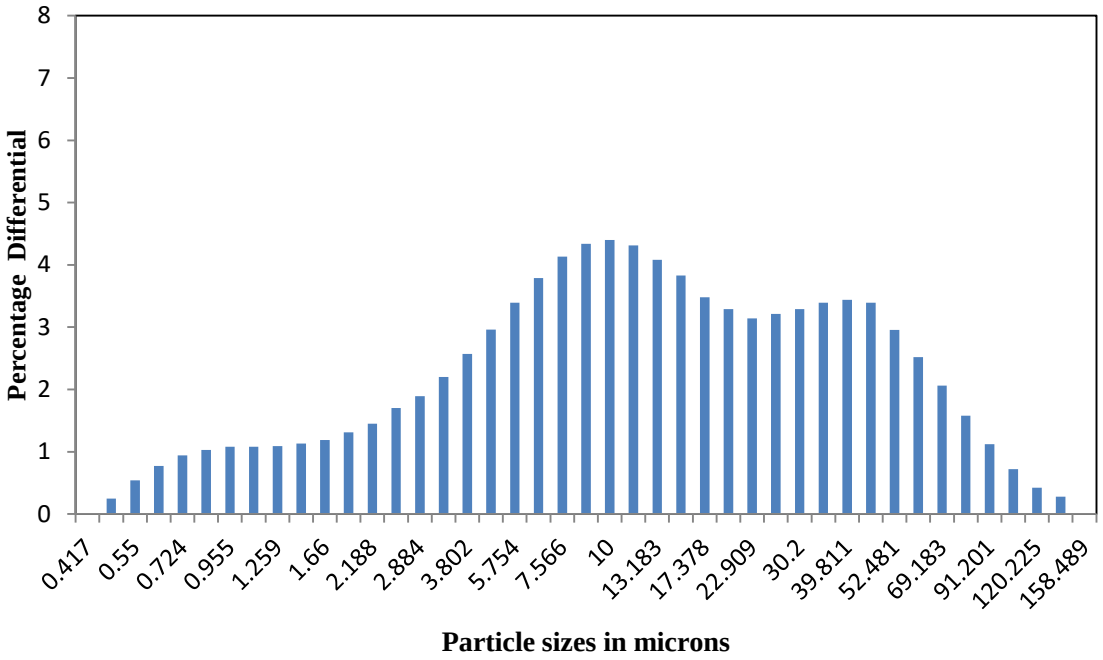


Figure 5-6: Particle size distribution of S40

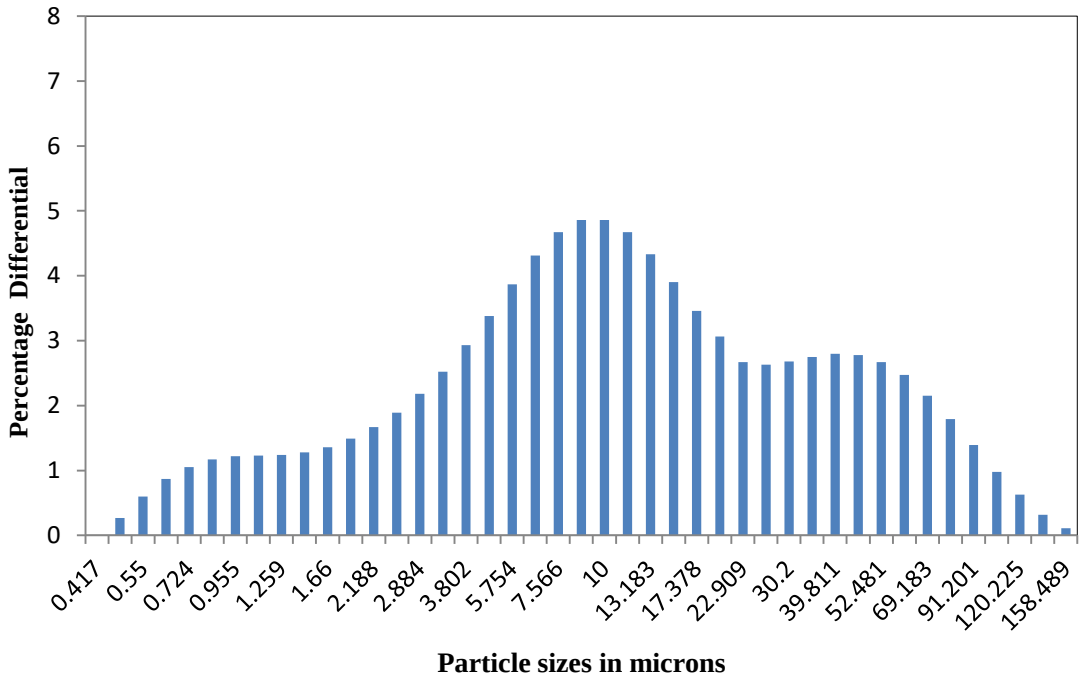


Figure 5-7: Particle size distribution of S50

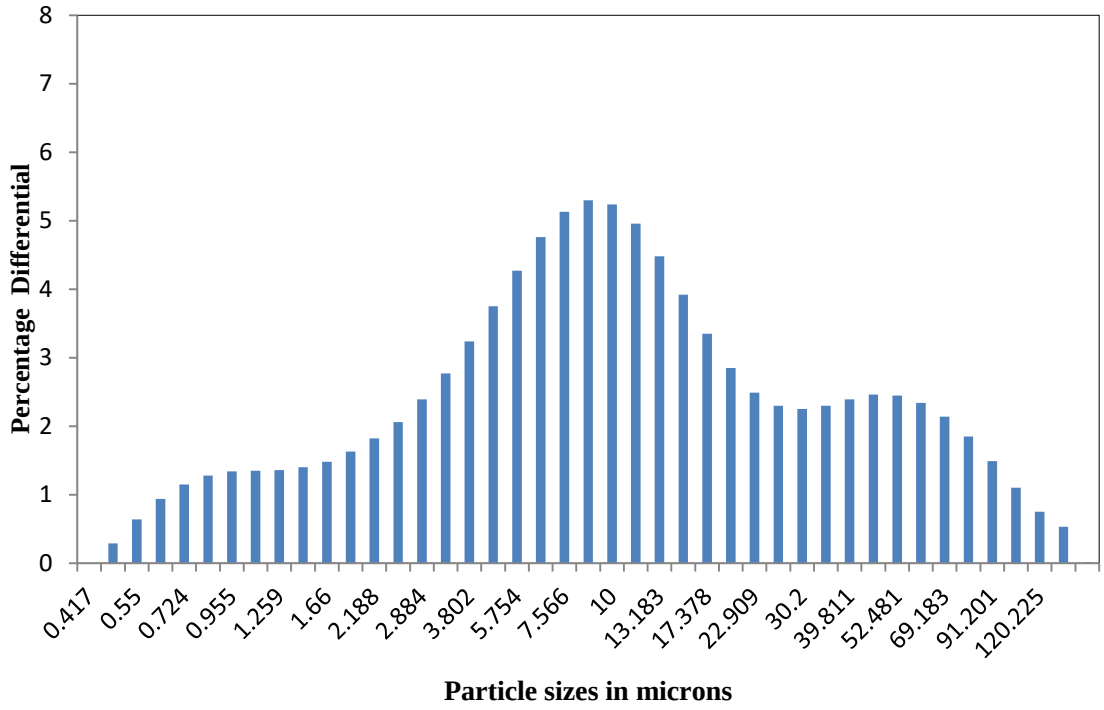


Figure 5-8: Particle size distribution of S60

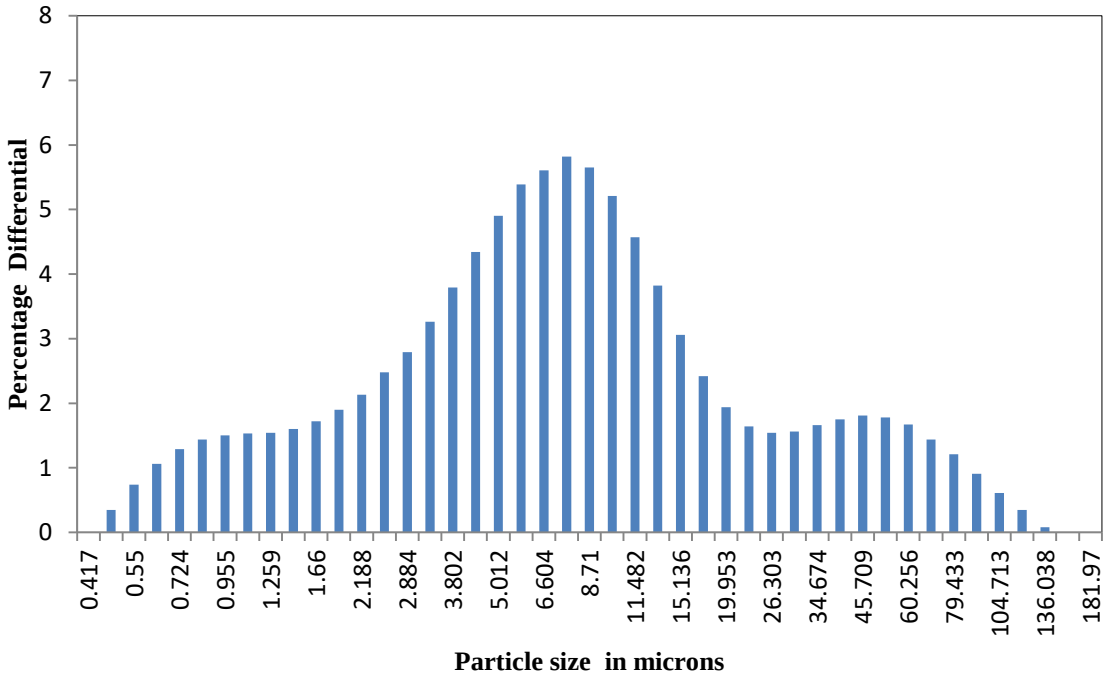


Figure 5-9: Particle size distribution of S70

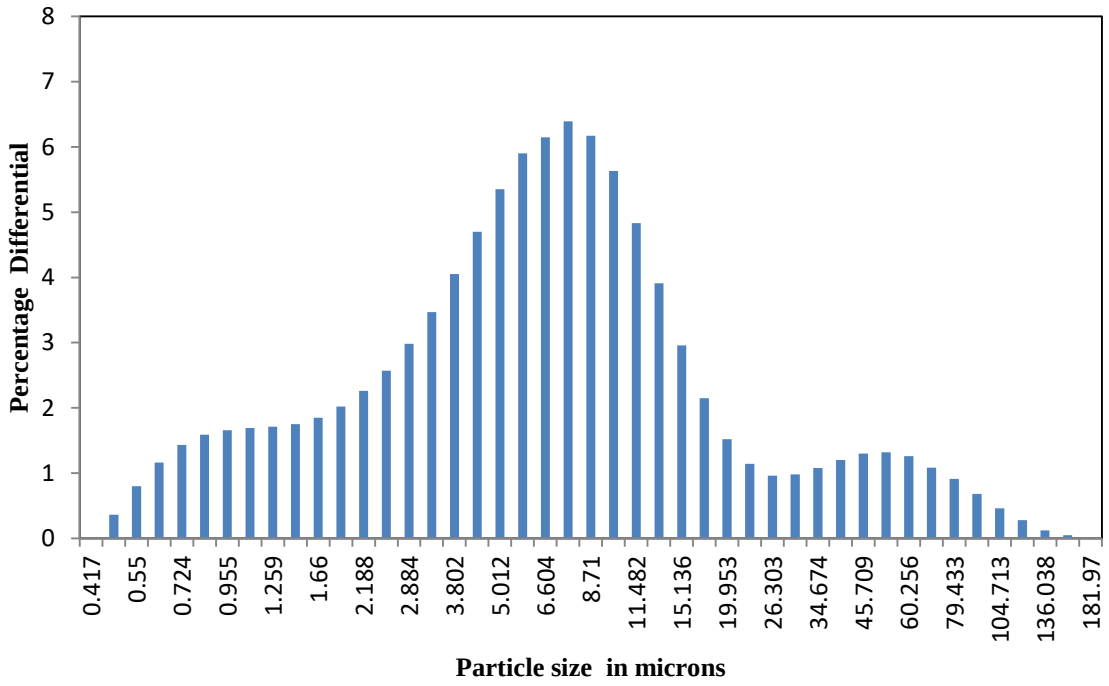


Figure 5-10: Particle size distribution of S80

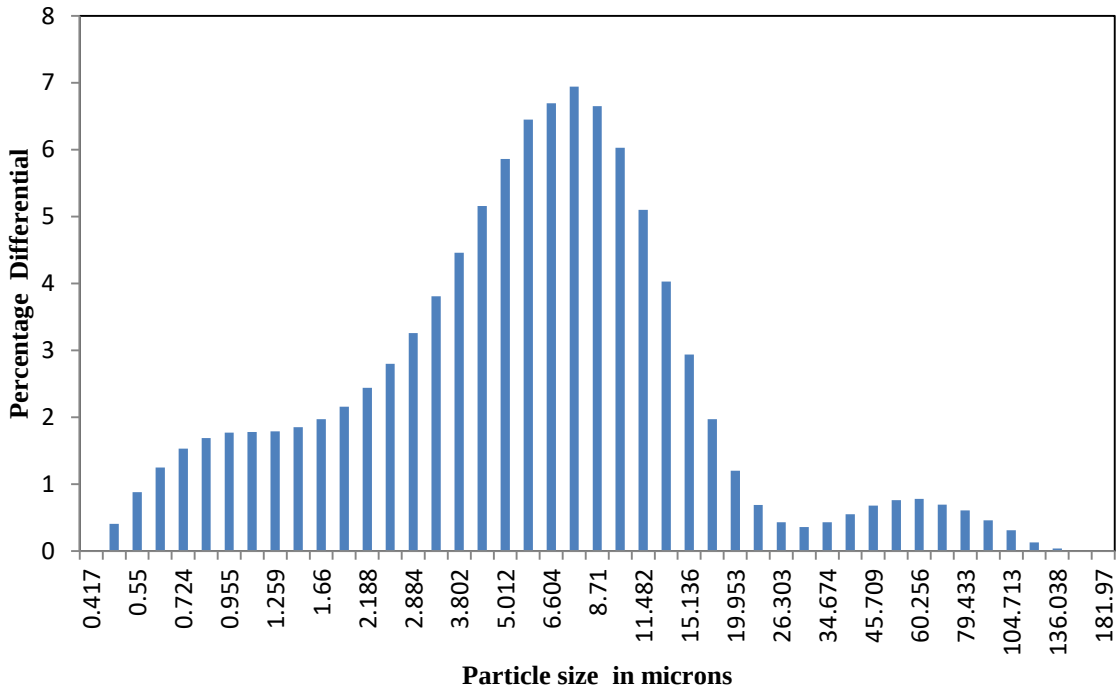


Figure 5-11: Particle size distribution of S90

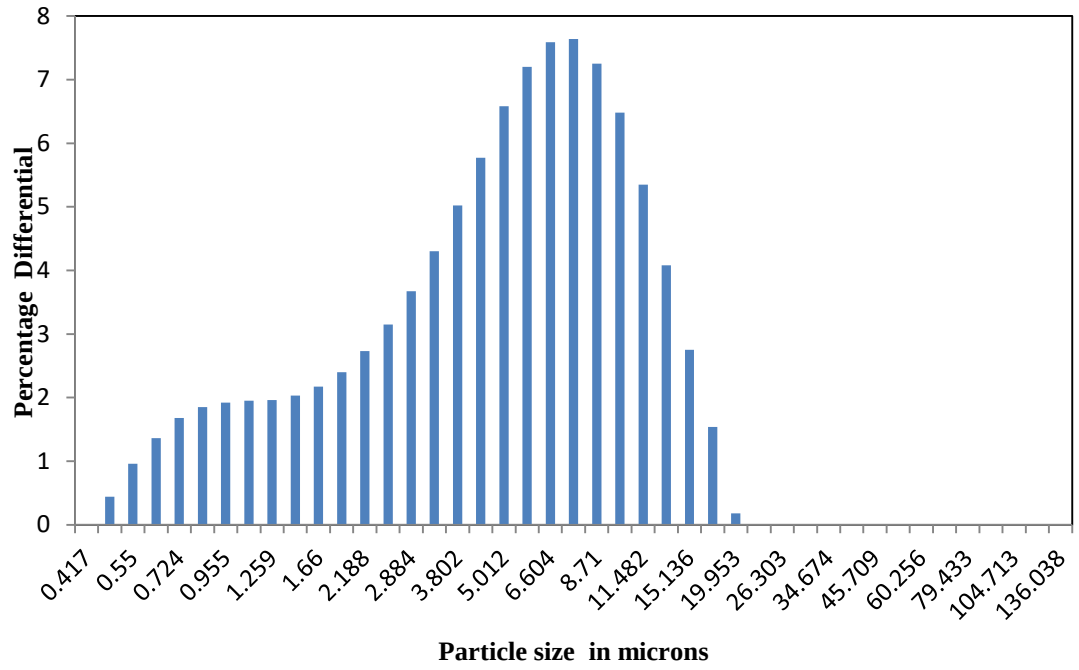


Figure 5-12: Particle size distribution of Pure Stimulator S100

5.2.2 Comparison of PSD Combinations

Figure 5-13 presents the PSD for the combinations S40 –S50 and OPC and S100. It has been noted that S45 is the optimized combination concerning PSD. Figure 5.14 represents the average particle size noted for the combinations considered in Table 4.2. Referring to Figure 5.13 the following is noted:

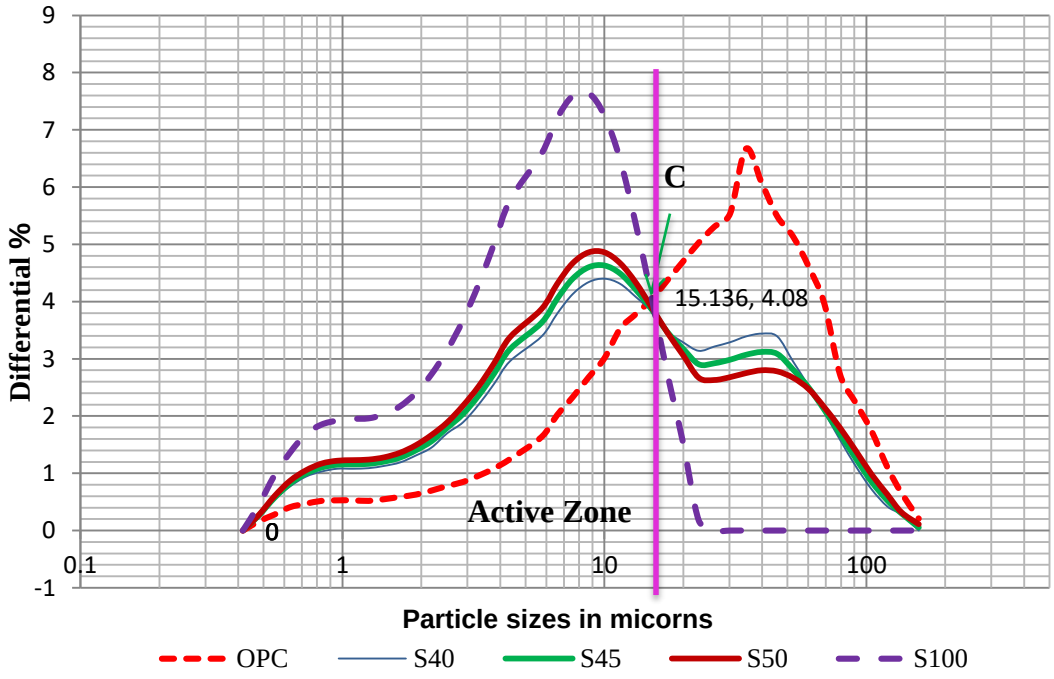


Figure 5-13: Comparison of PSD of OPC and Optimum stimulator dosage

a. The differential percentage of fines is lowered from 6.67 to 4.86.

b. The shift in the particle size peaks between OPC and that of the optimized case is (23.38-8.99) 12.87 microns

c. The differential percentage of fines is lowered from 6.67 to 3.12 in the second active phase.

d. A specific point C was observed in Figure 5.13 which is a meeting point of all the curves. This indicates the particles finer than 15 microns are the first ones to participate in the hydration.

e. Sizes of the particles beyond this either participate slowly or may not participate depending on the availability of water.

f. The percentage of particles participating in the hydration process can be found by the area under the curve by simple integration of the active zone.

Table 5-4 summarizes the percentage of particles based on their average size in the combinations considered. It has been noted that 20% of the particles are more active in the process of hydration in the combination S45 as compared to OPC. This fact enhances the strength of cement which is attributed to the particle size.

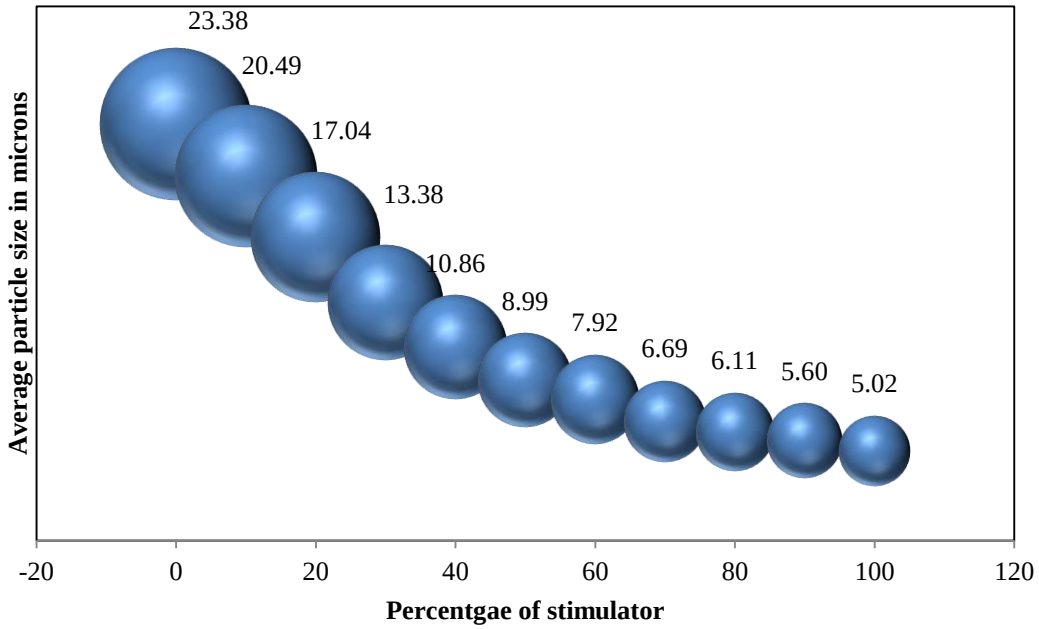


Figure 5-14: Average particle size of cement samples with varying stimulator content

Table 5-4: Percentage of particles (< 3 microns, 3-9microns and 9-25microns) of various cement samples

Particle size range	OPC	40S	50S	100S
<3µm	7.52	15.45	17.52	28.27
3.0-9.0 µm	11.96	23.38	26.52	44.1
9.0-25.0 µm	28.45	26.53	26.95	27.63
Total Particle fines between <25.0 µm	47.93	65.36	70.99	100
% increase in fines w.r.t OPC	**0.00%**	**17.43%**	**23.06%**	**52.07%**

A graphical representation of Figure 5-15 is drawn to understand the effect of PSD and the activeness of the particles in the process of hydration. The process of micronization improves by 20% in comparison to OPC with that of an optimized combination. Further, the process of micronization does not affect the particles 9-25 microns but influences the particles lower than 9 microns thereby improving the cement quality. The finer particles are also responsible for high early strength for a given content of C3S. This fact is again attributed to PSD.

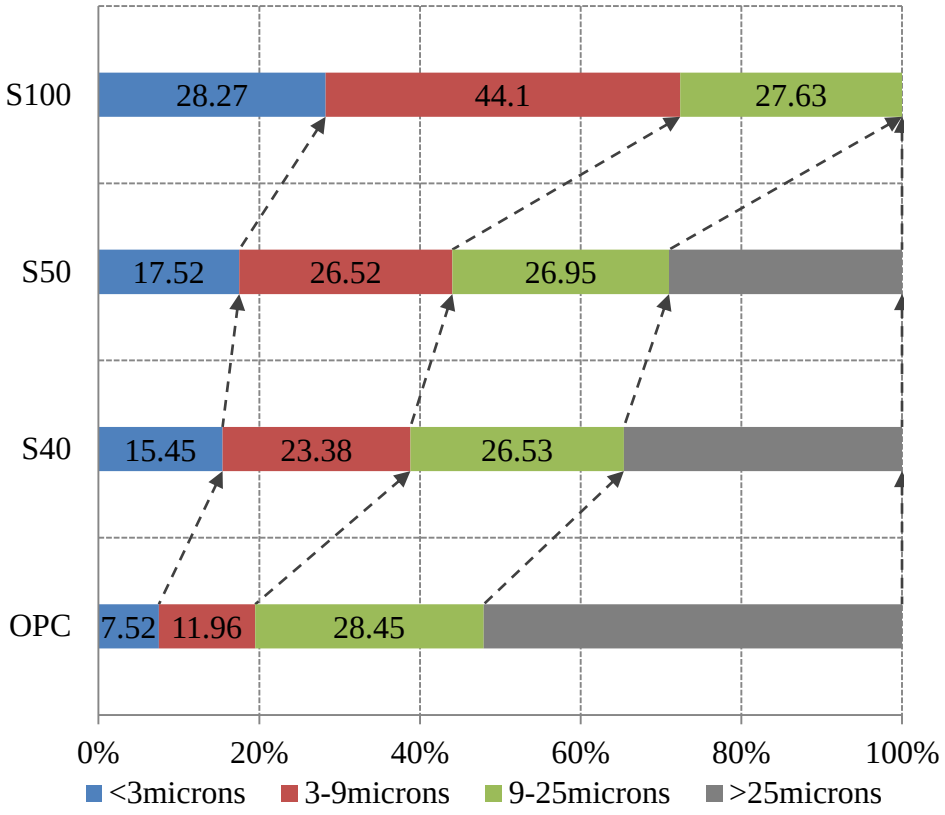


Figure 5-15: Categorization of cement particle sizes

5.3 Water requirement of cement

The amount of water added to the cement while mixing plays an important role in C-S-H gel development which ultimately crystallizes and imparts strength to concrete. The water requirement of cement affects initial setting times (IST), final setting times (FST), compressive strength values as well as other mechanical properties. Typically, with an increase in the fineness of cement, the water requirement is expected to rise linearly. However, a unique range of fineness of cement was found to be not in agreement with the above statement. This phenomenon was presented by EC Higginson. A detailed description is provided in Figure 2-2. He also stated that the relation between water content and fineness is an inverted bell.

In the present scenario, the work has been carried out on the left limb of the curve indicated by Higginson. However, the present study is on the right limb considering the unique fineness for which there is no additional water requirement beyond a certain range of fineness. Thus the concept of shifting from consistency to flow values was found to be more rational. Accordingly, referring to Figure 5-16, restricting the flow value to 110mm the water content was optimized and the corresponding average fineness required was estimated. It is noted that S45 satisfies this specific requirement.

The flow table and Normal consistency test results are presented in Figure 5-16 and Figure 5-17 respectively. Typically, it represents the right side of the inverted bell-shaped curve proposed by EC Higginson. The mixes S40 to S50 indicated a relatively flatter trend of water requirement despite a decrease in the average particle size, which is attributed to the unique particle size arrangement. Typically following the double peak arrangement. The trend lines in Figure 5-16 and Figure 5-17are in good agreement.

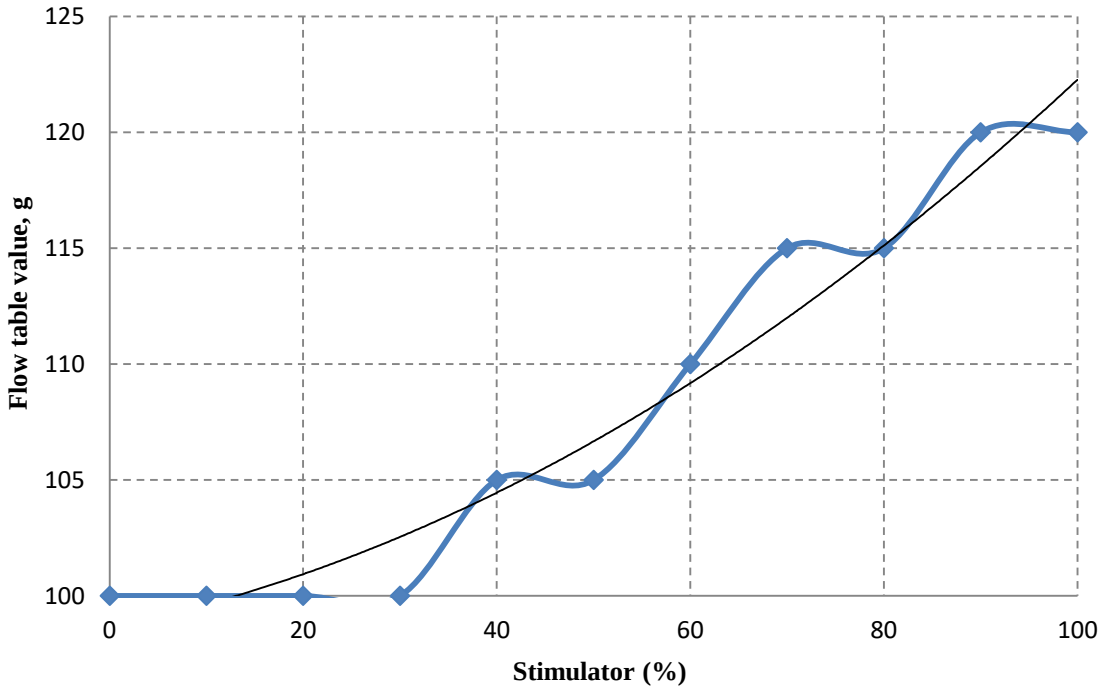


Figure 5-16: Flow table values v/s % of stimulator

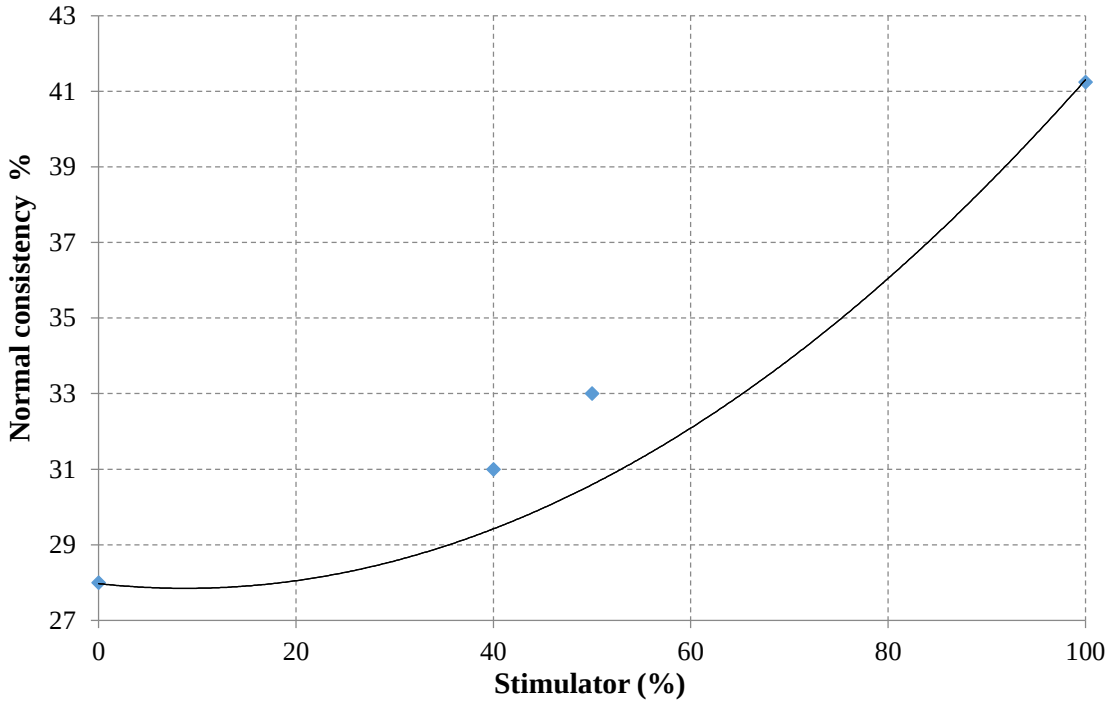


Figure 5-17: Normal consistency results in v/s % of stimulator

The relation is step-wise and indirectly promotes the reason behind the sequential hydration of cement. The normal consistency of cement depends on the particle size range as depicted in Figure 5-18.

With reference to IS 4031 part 4 the normal consistency values are plotted for the combinations of stimulated cement mixes. The NC values are least affected for particle size up to S45. However, beyond this the water requirement is exponential. Hence PSD influences NC values. Figure 5-20 shows the relation between particle size and water requirement to achieve normal consistency. The intersection point of the curves represents the optimum balance of particles for better efficiency of cement.

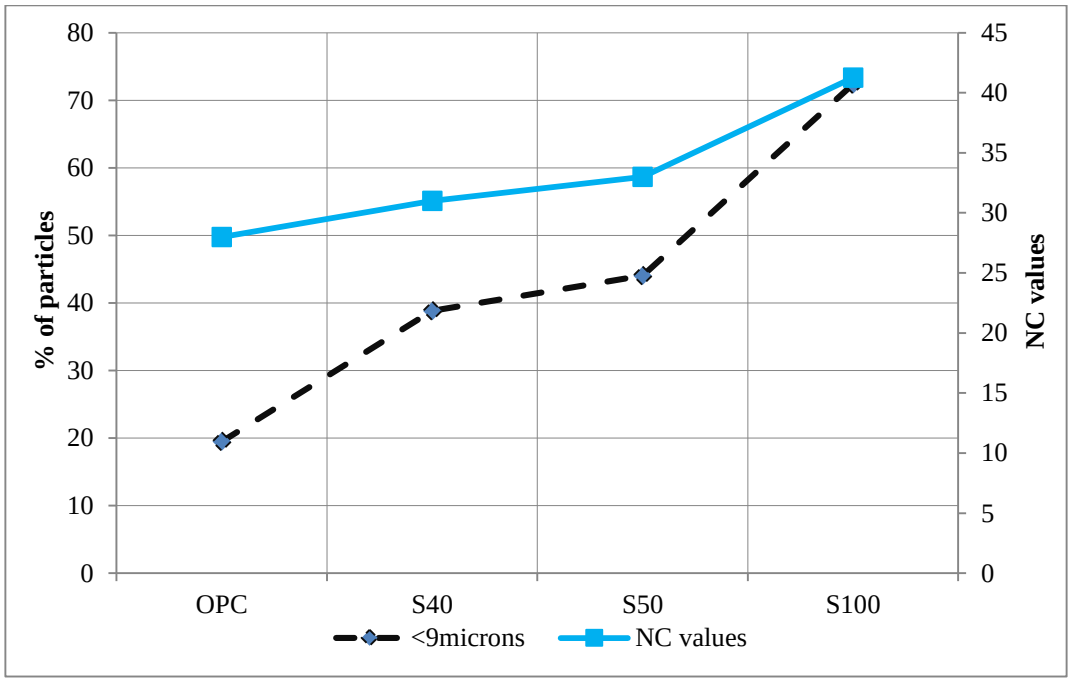


Figure 5-18: Effect of <9microns and >25 microns sized cement particles on NC value

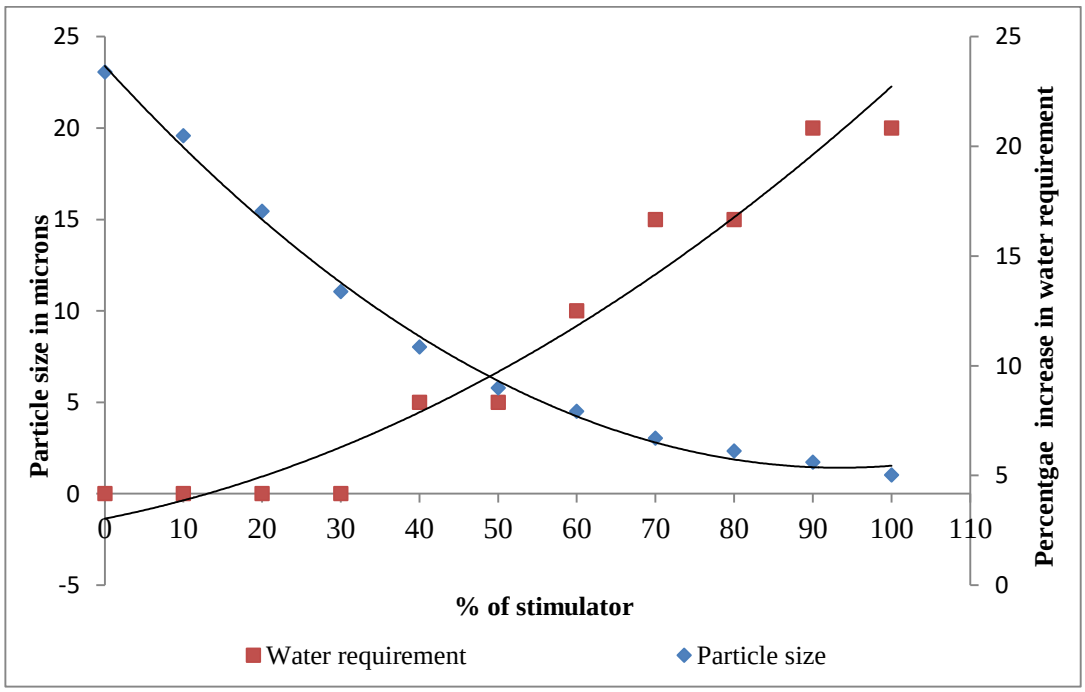


Figure 5-19: Relation between particle size and water requirement

5.4 Setting times of cement

Setting times of cement affects fresh properties of concrete such as workability, transportation and placement of concrete. The relation between the setting times of cement and the percentage of stimulators is graphically presented in Figure 5.20. The sudden drop in setting times beyond S50 is attributed purely to the early participation of micro-fine particles (from the stimulator) in the hydration process. Noting this sudden drop of IST and FST, setting time was determined for an intermediate percentage of the stimulator to locate the exact point of drop-down i.e. S45. It was then concluded that the expected trend of setting time is followed from OPC (S0) to S45 and then drops down from S45 to S50 further continuing the linear downward trend. Hence, the optimum dosage of stimulator content from the point of view of setting time is justified.

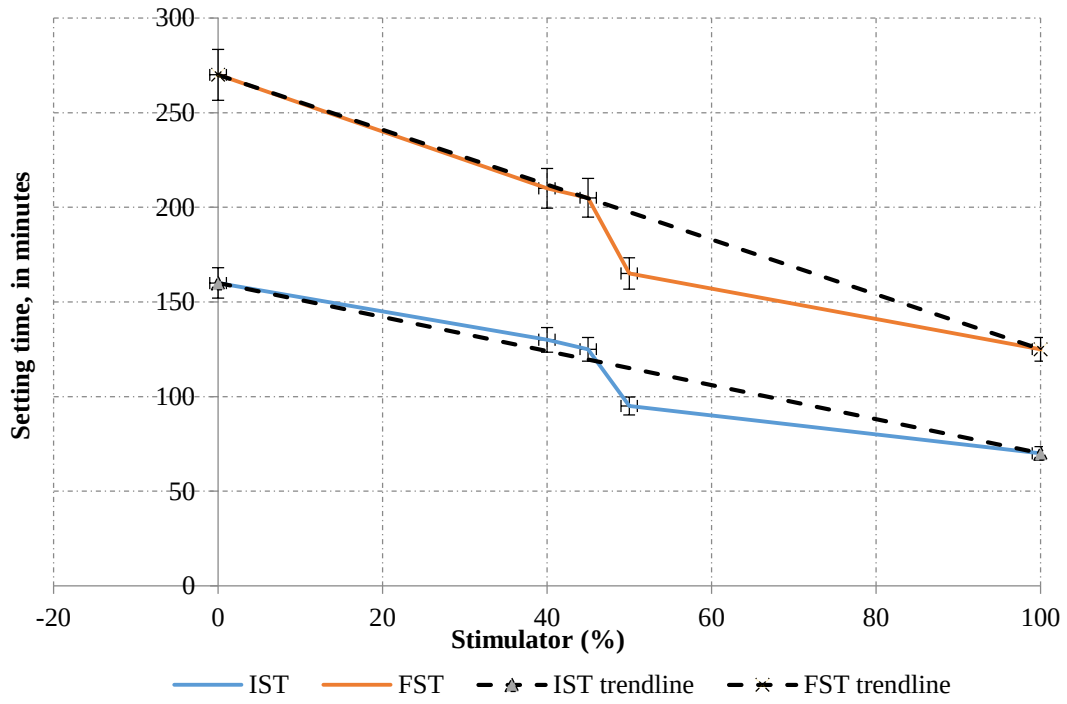


Figure 5-20: Setting times v/s percentage of stimulator

5.5 Chemical shrinkage of cement

Hydrated cement occupies a lesser volume as compared to un-hydrated cement. This reduction in the volume of cement upon hydration is referred to as chemical shrinkage. Thus, the measurement of chemical shrinkage is an alternative approach to measuring the degree of hydration of cement. Due to the increased fineness of cement particles degree

of hydration is more as compared to coarser particles of OPC and therefore the chemical shrinkage is bound to increase. Results of chemical shrinkage are presented in Figure 5-21 and Figure 5-22. The graphical representation of chemical shrinkage against time and degree of hydration varies for cement with fineness is presented in Figure 5-21 and Figure 5-22 however, the chemical shrinkage stabilizes after 48 hours of casting.

The results of the heat of hydration showed insignificant changes in values due to changes in cement fineness. The slight variation in the heat of hydration is attributed to random sampling. The values of heat of hydration for samples S0 (OPC), S40, S50 and S100 are 86.38, 88.32, 88.78 and 90.34 cal/g respectively.

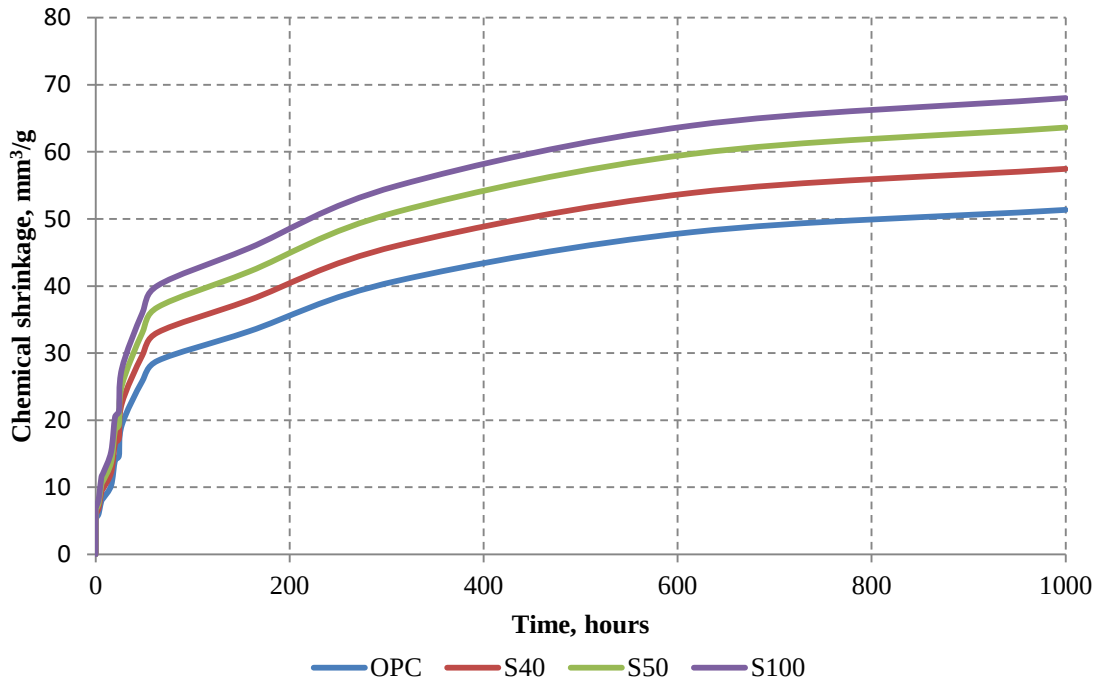


Figure 5-21: Chemical shrinkage values v/s time

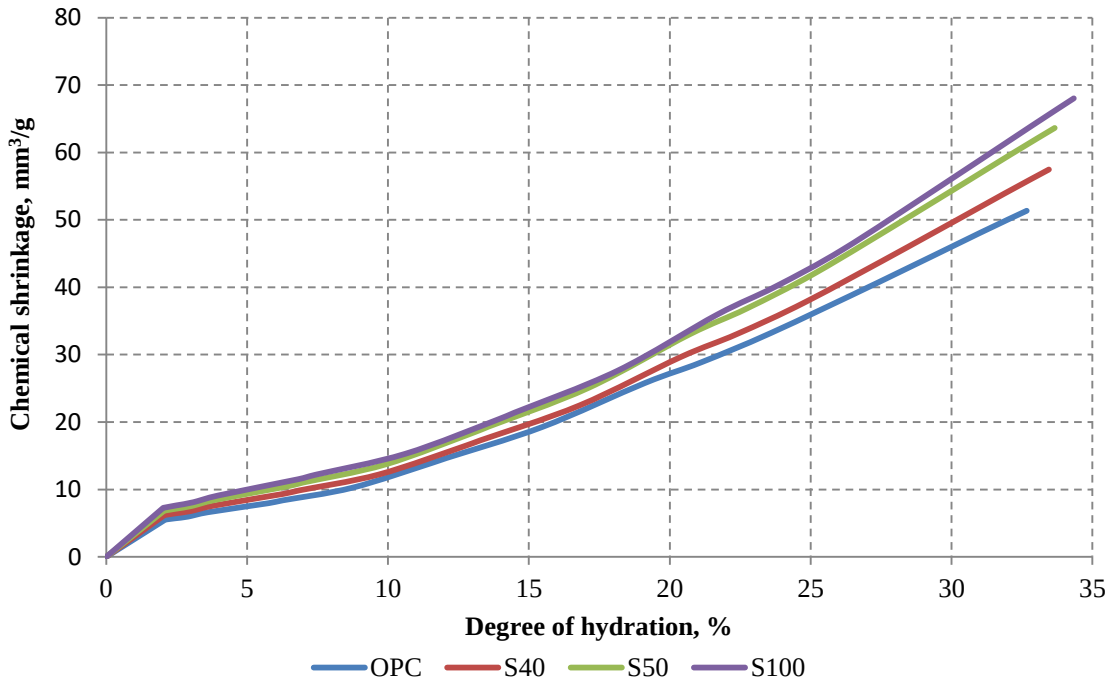


Figure 5-22: Chemical shrinkage v/s degree of hydration

5.6 Compressive strength- Cement

It is one of the important properties of cement. Bogue's compound decides the proportion for achieving the desired strength. It is cited in the literature that the presence of C3S and C3A contributes greatly to this factor. It is observed from Table 5-1 the combined summation is around 35%. High early strength in the sample is possible with a 65% of the presence of C3S and C3A chemically. The observations indicated PSD is one of the factors to improve high initial strength. The tests on OPC 53 with and without stimulators were conducted. The experimentation was also conducted on OPC 43 with and without a stimulator.

5.6.1 Strength Studies on 53 grade OPC

A comparative study of the compressive strength with age was conducted for the samples OPC, S40, S45, S50 and S100. The age varied from 1, 3, 7 and 28 days of curing. It is noted that optimized particle size has more impact in imparting strength to the cement. Faster hydration is seen in this case and almost complete hydration is seen at the end of 7 days of curing. This fact is attributed to the participation of the particles in the cement matrix completely however, it is not seen in the other combinations. This will enable the

user to take decisions on the de-shuttering process. The highest 28-day compressive strength is shown by the S100 sample i.e., 76.52N/mm^2, which is approximately 44% higher than (OPC). The 28 days compressive strength of S40 and S50 is 17.58% and 31.62% relatively higher than OPC. S45 (optimum mix) exhibited 40%, 24.79% and 24% higher compressive strength at 3,7 and 28 days of the curing period respectively in comparison with OPC.

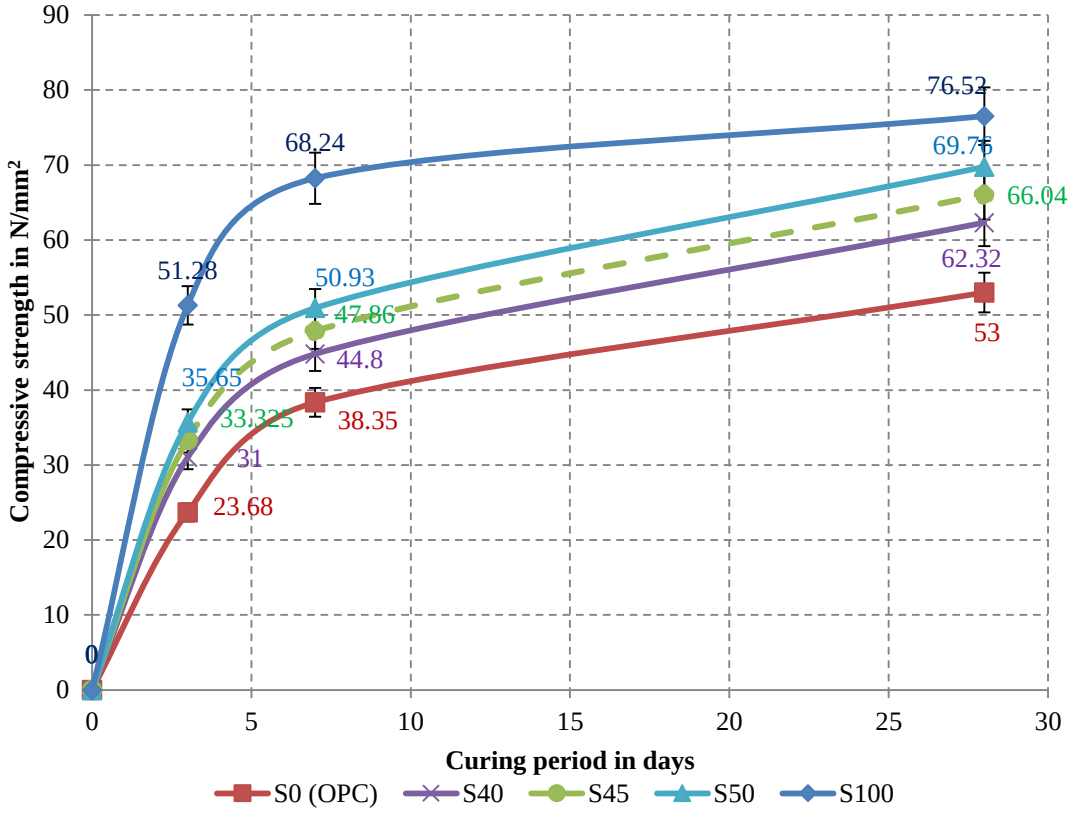


Figure 5-23: Compressive strength v/s curing period using 53 Grade OPC

Note: Error bars are used to show the fluctuation of 5% in measured compressive strength values.

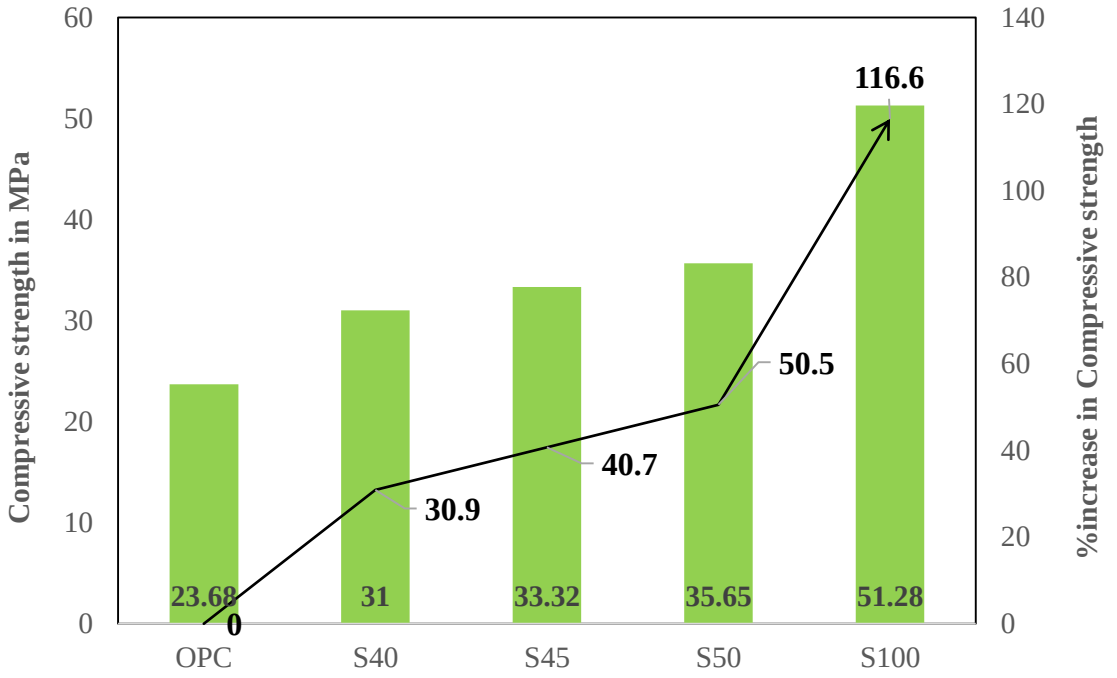


Figure 5-24: 3 days Compressive strength and % increase in Compressive strength - 53 Grade OPC

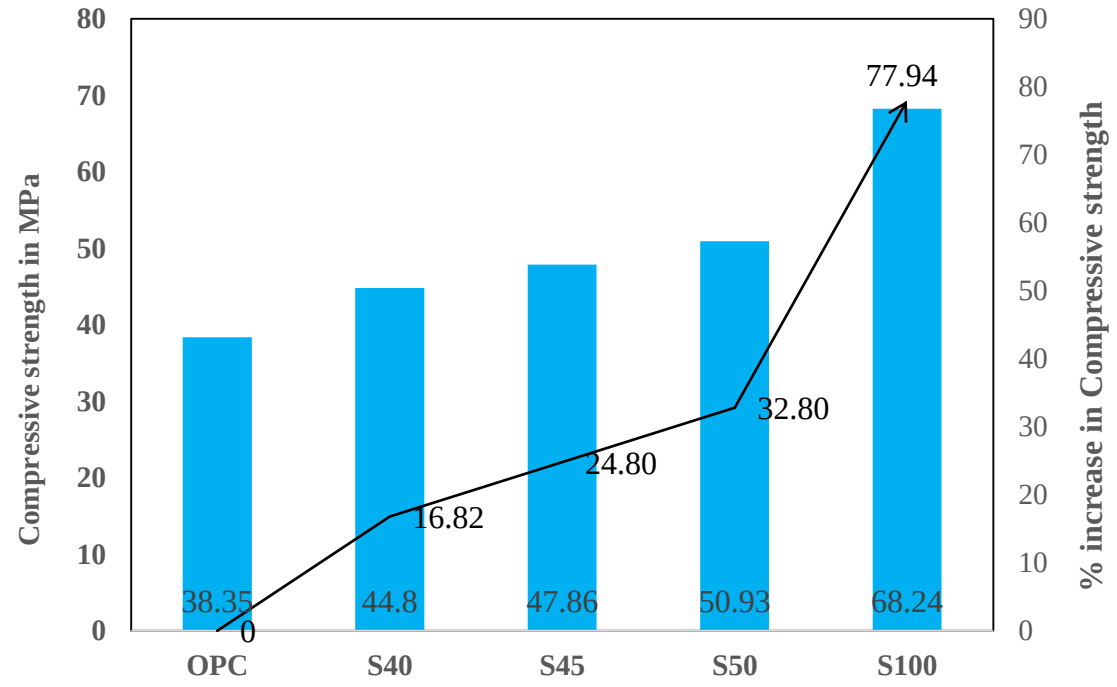


Figure 5-25: 7 days Compressive strength and % increase in Compressive strength - 53 Grade OPC

Figure 5.24 and Figure 5.25 present a percentage increase in compressive strength of 3 days and 7 days compressive strength values. 3 days of compressive strength has shown a 30 -50% increase in compressive strength values as compared to OPC samples and 7 days of compressive strength has shown a 16-32% increase.

5.6.2 Strength Studies on 43 grade OPC

A comparative study of the compressive strength with age was conducted for the samples OPC, S40, S45, S50 and S100. The age varied from 1, 3, 7 and 28 days of curing. It is noted that optimized particle size has more impact in imparting strength to the cement. Faster hydration is seen in this case and almost complete hydration is seen at the end of 7 days of curing. This fact is attributed to the participation of the particles in the cement matrix completely however, it is not seen in the other combinations. This will enable the user to take decisions on the de-shuttering process. The 28 days compressive strength of S40 and S50 is 17.58% and 31.62% relatively higher than OPC. S45 (optimum mix) exhibited 80%, 24.79% and 11% higher compressive strength at 1,3 and 7 days of the curing period respectively in comparison with OPC.

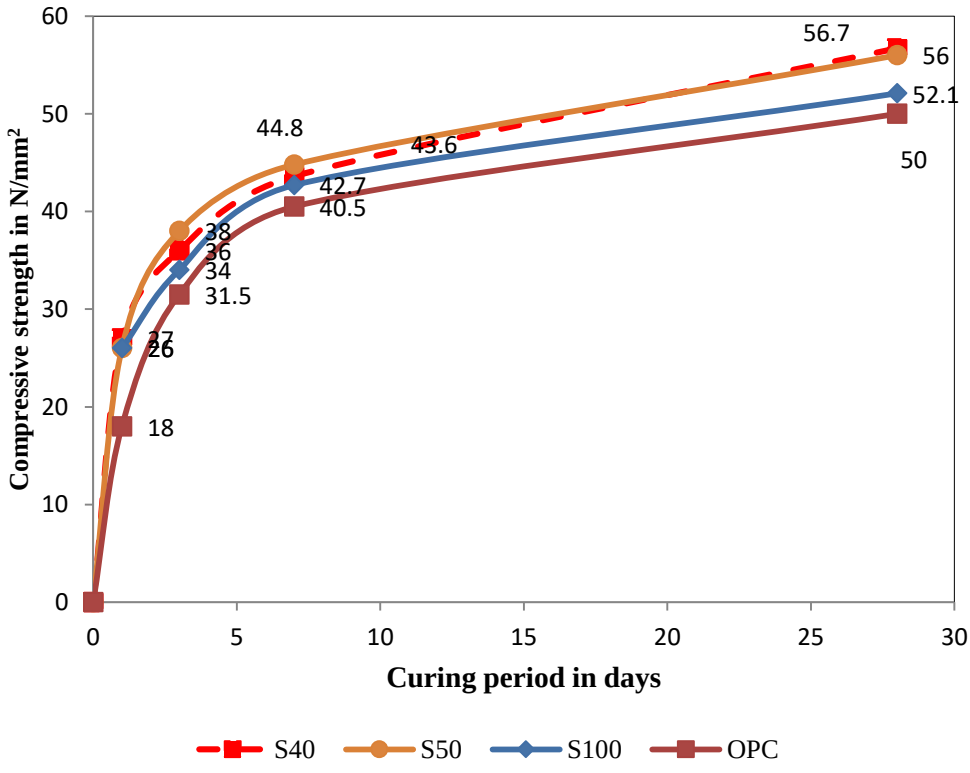


Figure 5-26: Compressive strength v/s curing period using 43 Grade OPC

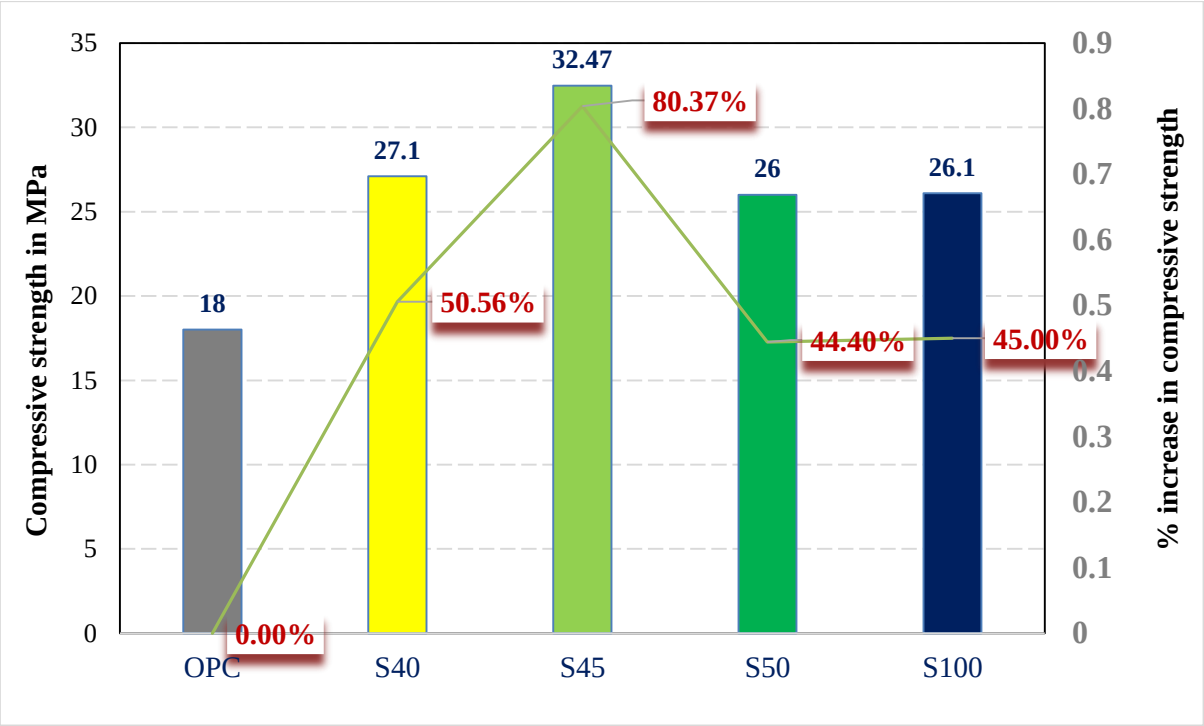


Figure 5-27: 1 day- Compressive strength and % increase (43 Grade OPC)

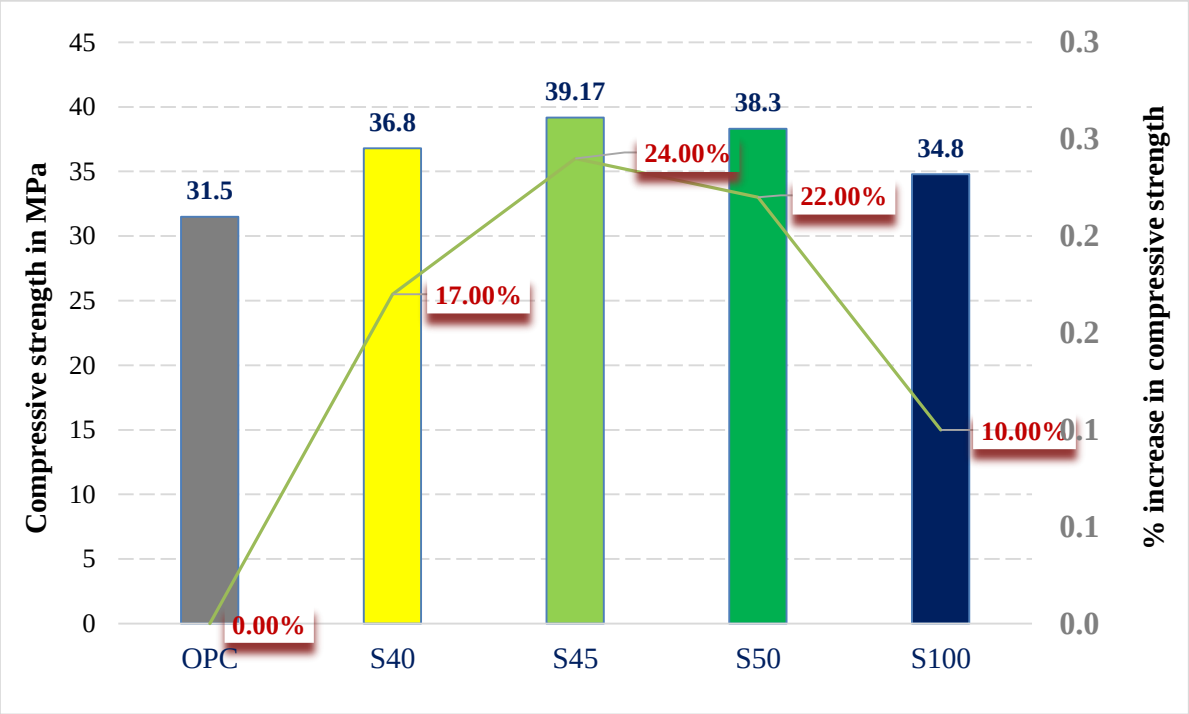


Figure 5-28:3 day- Compressive strength and % increase (43 Grade OPC)

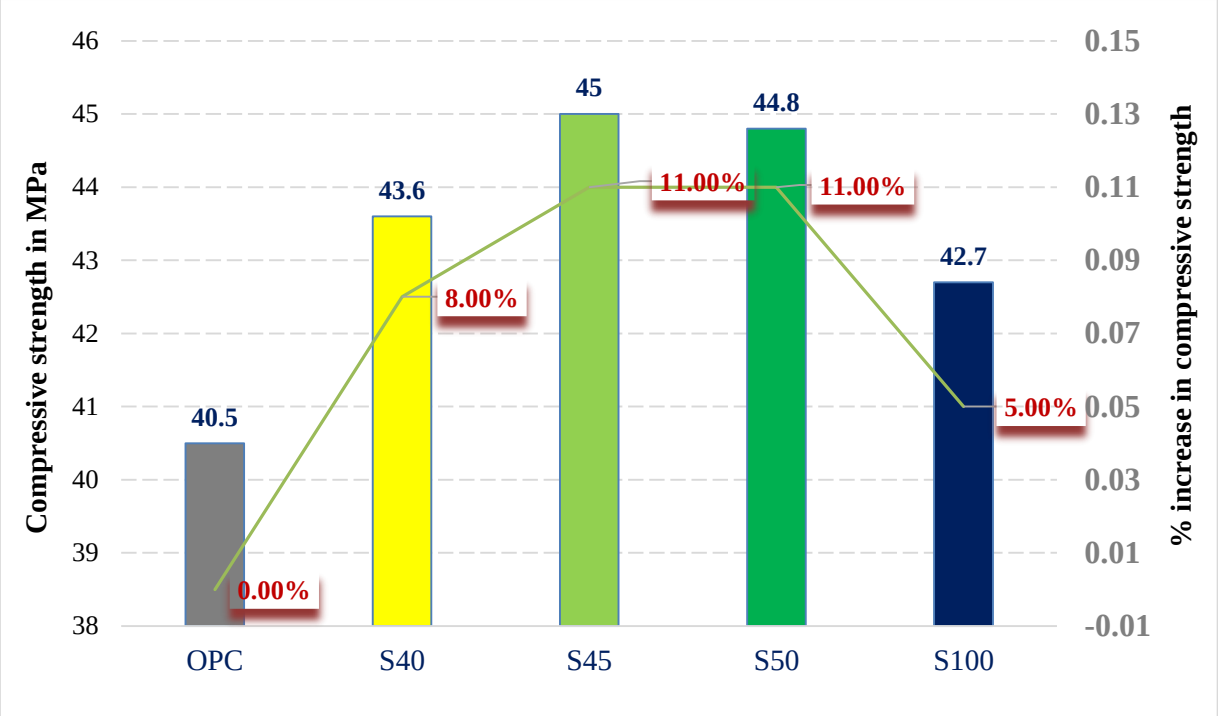


Figure 5-29:7 day- Compressive strength and % increase (43 Grade OPC)

Figure 5-26 presents the trend of compressive strength values of cement mixes using 43-grade OPC with combinations of stimulators. The trend of strength gain becomes flatter towards 28 days of curing however has shown a significant increase in early strength.

S40 –S50 cement mixes have shown about 50%, 17-20% and 8-10 % increase in 1 day, 3 days and 7 days compressive strength. Briefly, 3 days of compressive strength achieved by OPC can be achieved by S40 in just 1 day of casting. This shall thus enable to carry out early de-shutter on the site thus helping to save the cost and time on site.

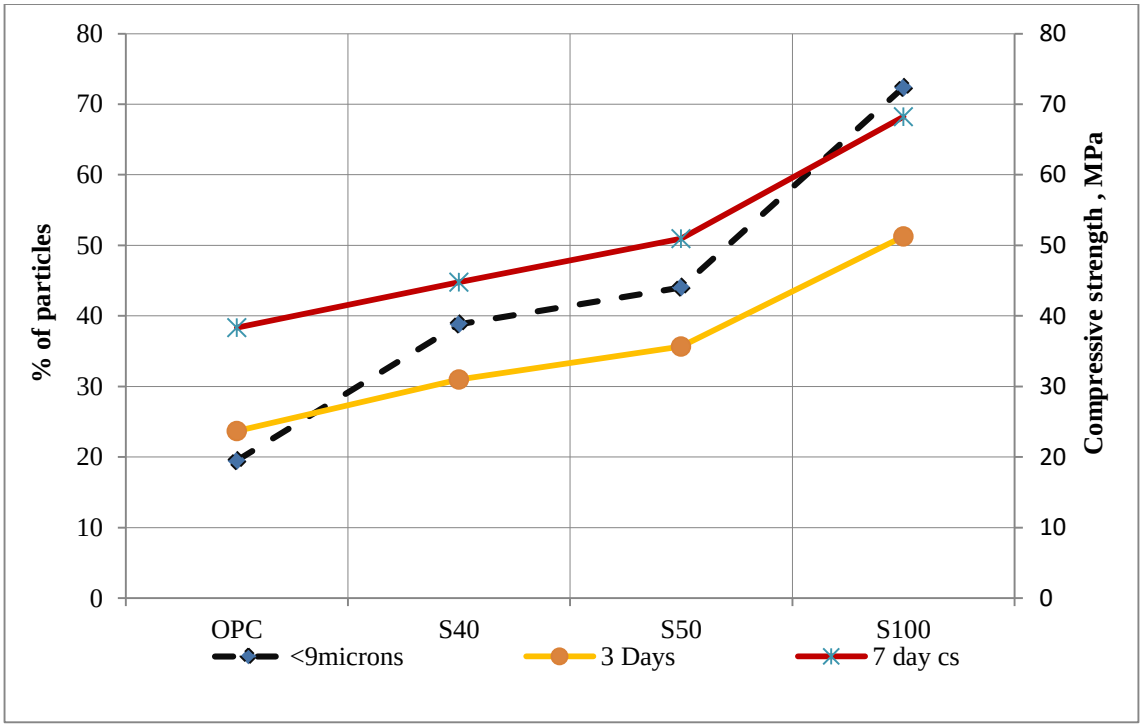


Figure 5-30: Effect of <9µm and >25µm sized cement particles on 3 and 7 Days compressive strength

Figure 5-30 presents graphical relation of particle size ranges with compressive strength values. Particles finer than 9 microns hold direct relation with 3 days and 7 days compressive strength values. However, the particles coarser than 25 microns hold indirect relation with compressive strength values. The trend of values for mixes S40 –S50 is flatter as compared to other cement mixes. This flatter trend again confirms the fact that S40 –S50 are the optimum dosage of stimulator.

5.7 Density of cement mortar

The density of cement mortar has been presented in Table 5-5 indicating a marginal increase in density with an increase in the percentage of stimulator. S40–S100 has depicted densification of the matrix due to particle packing caused by the presence of a stimulator.

Table 5-5: Density of cement mortar

Nomenclature	OPC	S40	S50	S100
Mass, g	282.83	285.8	288	289
Density, kg/m^3	2263	2286	2301	2312

5.8 Compressive strength- Concrete

One of the major constituents of concrete is cement. The study on optimized stimulator-assisted cement is presented and it is necessary to understand the behaviour of concrete. This study involved considering 2 grades of concrete M25 and M40. The grade M25 was not assisted with any admixture however; M40 was assisted with PC-based admixture for workability. The comparison of results of the respective grades was carried out with controls. The compatibility studies with admixture and the performance of the cement were compared.

Mix design calculations are presented in Table 3-6. The test results of the concrete in green state and hardened state were established. Table 5-6 presents the comparison of cement usage and corresponding improvements in strength. Concrete mixes with stimulators have recorded a 20-23% reduction in cement while achieving the desired target strength as per IS 10262. However, due to improving PSD higher early strength was achieved despite the reduction in cement quantity. The performance of optimized cement with a stimulator performed at least 1.5 times better than the normal concrete. It has been noted that a huge reduction in the cement per cubic metre of concrete.

Table 5-6: Experimental results of concrete mix designs

Parameter	**Normal Mix**		**Stimulator assisted mix**	
	M25	M40	M25	M40
Cement quantity required per cum of concrete, kg	360	430	300	330
No cement bags required per cum of concrete	7.2	8.6	6	6.6
Reduction in cement bags required per cum of concrete	--	--	1.2	2
% Reduction in cement quantity	--	20.00%	--	23.25%
Compressive strength, MPa				
1 day	7.5	13	12.25	18.3
7 day	17	27	24.6	38
28 days	33	49.5	38	52

Figure 5-31 and Figure 5-32 present 1 day and 7 days of compressive strength of concrete.

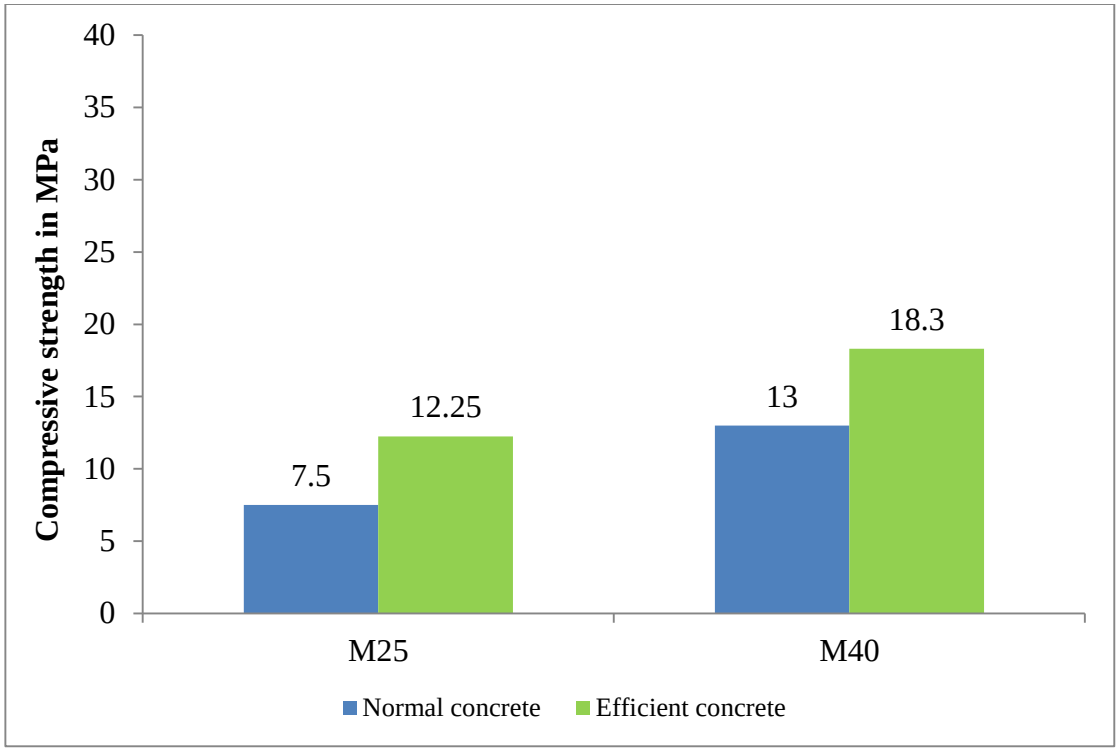


Figure 5-31: 1 day -Compressive strength of concrete

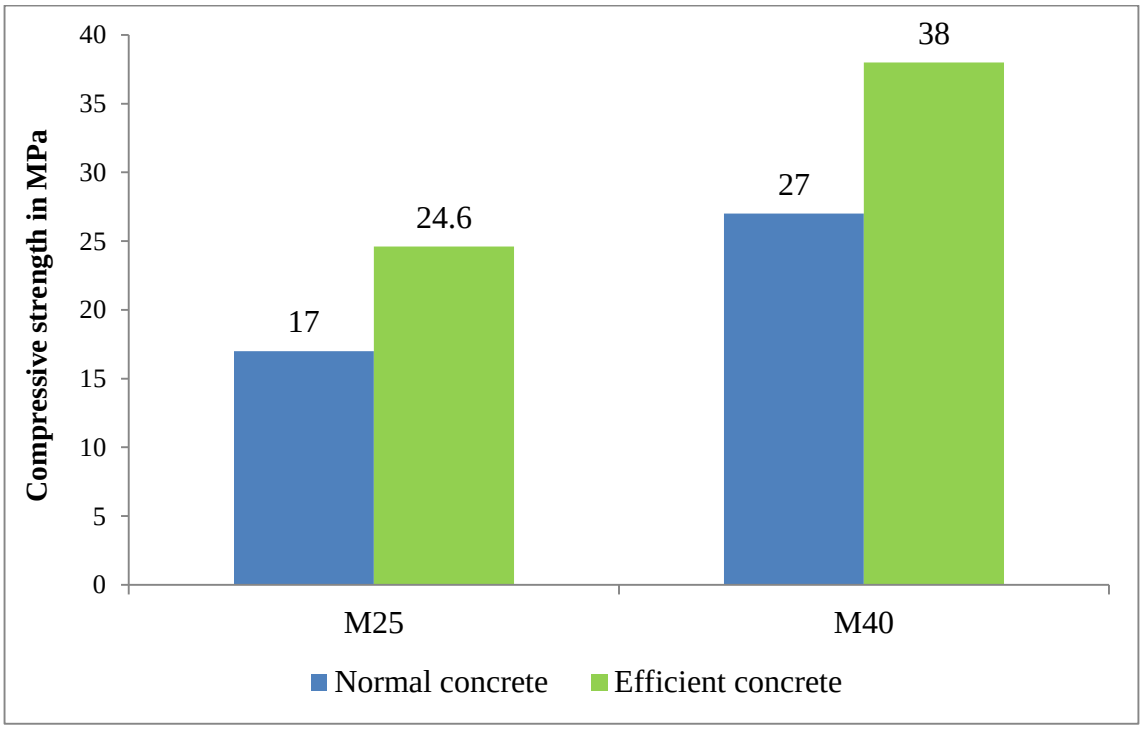


Figure 5-32: 7 day -Compressive strength of concrete

5.9 Studies on the Effect of curing compound

The usage of curing compounds is being practised nowadays to save water. Quality of the cement and compatibility of the curing compound is a must before the use of any construction chemical. Studies were carried out to find the compatibility and performance of the stimulator-assisted cement. MONOBOND of Krishna-con-chem makes was selected for application as a curing compound. Table 3-5 presents the technical data of the compound. The compound was mixed with water in the proportion of dilution ranging from 10-50% by weight. The solution so prepared was applied with a brush (single coat) and allowed for curing. Similarly, control was also used for comparison.

It has been observed that the compound is compatible and a more efficient combination was 10% dilution. Higher concentration tends to give lower compressive strength. This phenomenon is attributed to the cracking of the curing compound. It is a well-known fact that the curing compounds never be compared with total water curing. Curing with chemical are bound to give lower result up to 10% in comparison with the age to that of conventional curing. A similar phenomenon was also observed in the present case.

The application of curing compound was carried out for all the combinations S0, S40, S50, S60 and S100. The corresponding compressive strength was noted. It is concluded that a 10% diluted solution of the curing compound is efficient to an extent of 95% as compared to other curing. Figure 5-33 to Figure 5-37 shows the compressive strength observed without curing compound and with varying percentages of curing compound. A graphical representation of the efficiency of curing compound for all combinations S0, S40, S50, S60 and S100 at 10% diluted solution is shown in Figure 5-38. It is concluded that the curing compounds in general suitable for conventional concretes will also hold well with the concrete assisted with the stimulator.

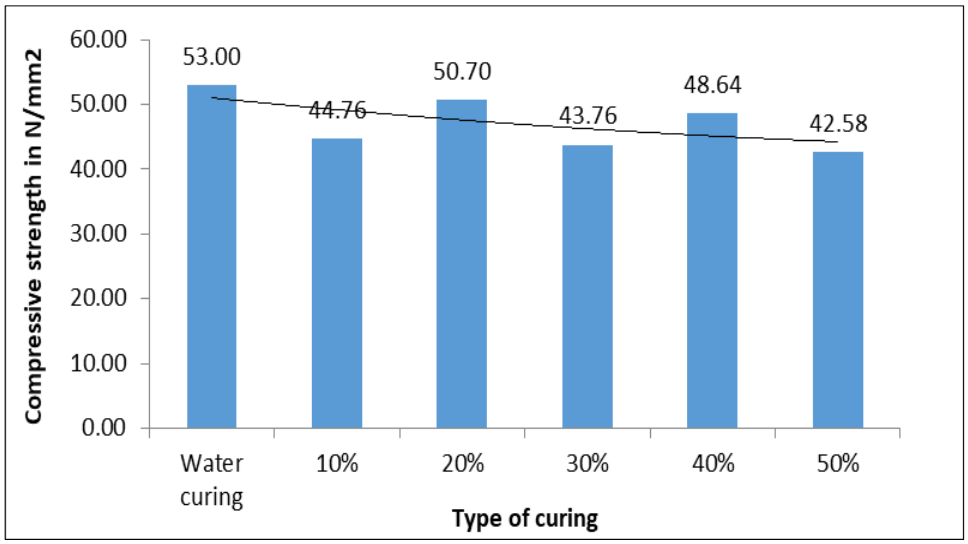


Figure 5-33: 28 days compressive strength OPC

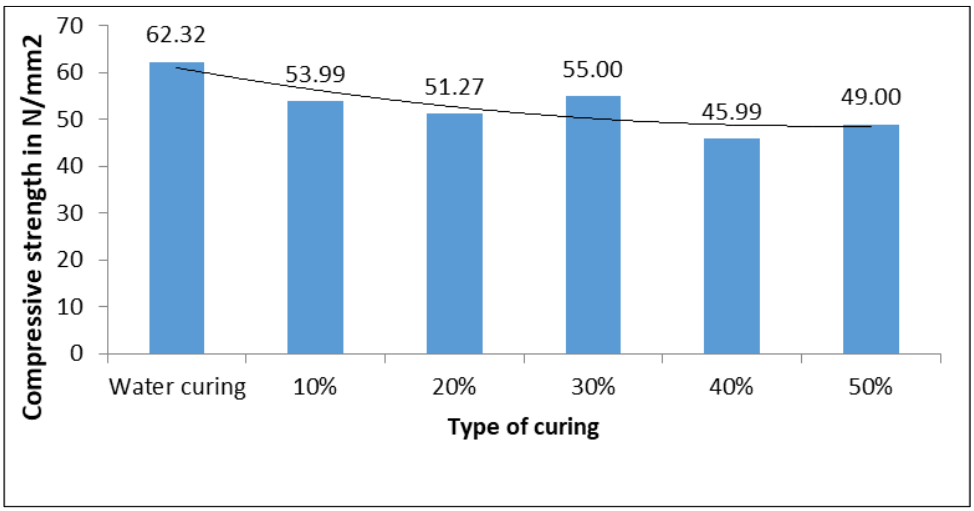


Figure 5-34: 28 days compressive strength S40

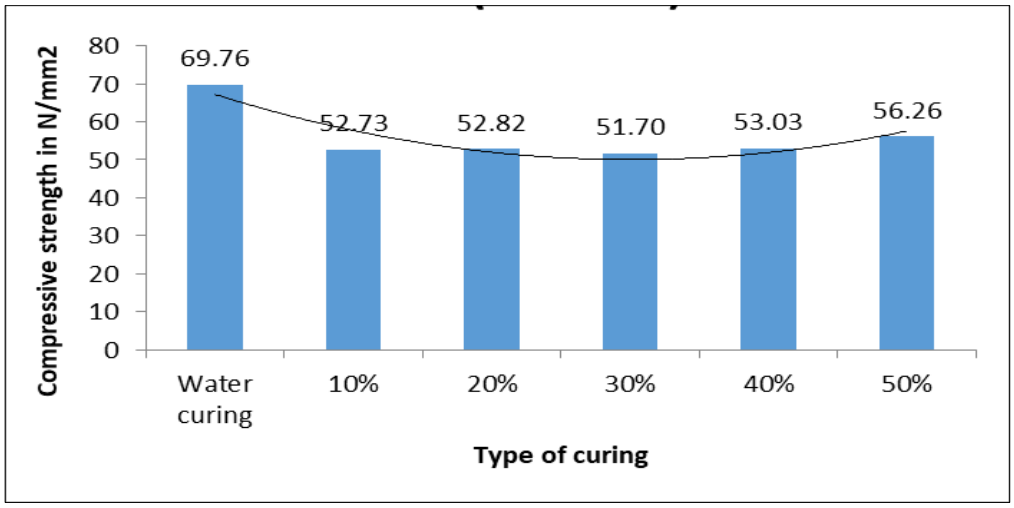


Figure 5-35: 28 days compressive strength S50

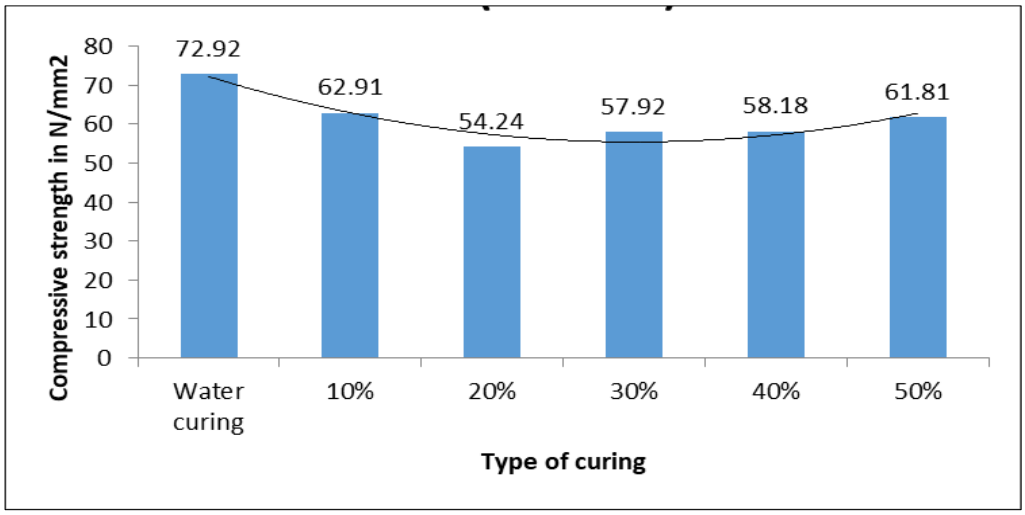


Figure 5-36: 28 days compressive strength S60

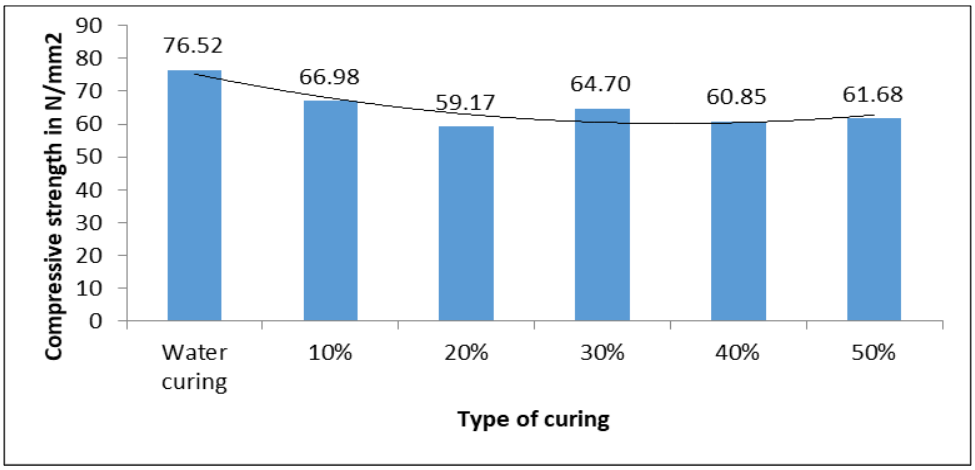


Figure 5-37: 28 days compressive strength S100

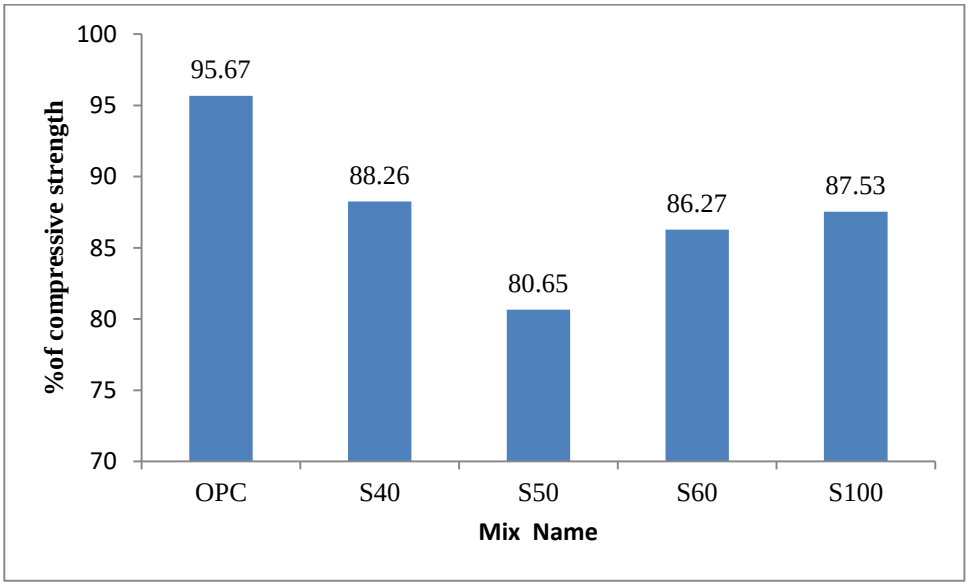


Figure 5-38: Efficiency of curing compound

5.10 Durability Results

The structure is susceptible to various chemical attacks due to which degradation of the concrete matrix occurs. The durability aspect is well accepted in t recent codes giving priority over strength. Better permeability and particle packing are essential for the improvement of the grade of concrete. Cement plays a role as a binding material in both these aspects. Measurement of resistance to durability is studied under Sorptivity, acid attack tests and permeability.

5.10.1 Sorptivity

Concrete is believed to be more susceptible to micro-cracks and thereby intervening with the durability. The volume of pores depends on the particle packing, density of materials used and degree of compaction of concrete. The porosity of concrete affects the water absorption and freeze and thaw resistance of concrete. The ability of concrete to absorb and transport water through capillary suction is termed sorptivity. Thus lower values of sorptivity indicate improved durability and resistance against fluid transportation through capillary action. Hence, concrete with lower porosity is desired to maintain durability and strength. The experimental methodology has been provided to assess the sorptivity values in the article 4.3.5 a confirming to ASTMC 1585. A graphical representation of sorptivity against the square root of time is shown in Figure 5.39 for both the grades under consideration. It is imperative to state that the relation between sorptivity and durability is inversely proportional. Figure 5-39 shows a clear indication of the sorptivity of the grades under consideration. Normally, as per the code the values of sorptivity are estimated for the part of the curve marked till the line AB. The slope of the curve till line AB indicates the rate of sorptivity of the sample at the initial stages. However, the sorptivity values remain either constant or marginally increase for the rest of the part as indicated in Figure 5.39 beyond the line AB, which is also termed secondary sorptivity. Concrete samples with OPC exhibit higher sorptivity ranging between 0.0265-0.037 mm/sec$^{0.5}$. However, for the modified cement the values are ranging between 0.004 and 0.0027 mm/sec$^{0.5}$, exhibiting the resistance to the sorptivity by 600 times. Figure 5-40 and Figure 5-41 shows the initial and secondary sorptivity values.

It has been witnessed from the behaviour of mechanical properties of cement and concrete samples when compared with the control, the samples have better packing

density and resistance to permeability. The sorptivity values indicate long-term durability.

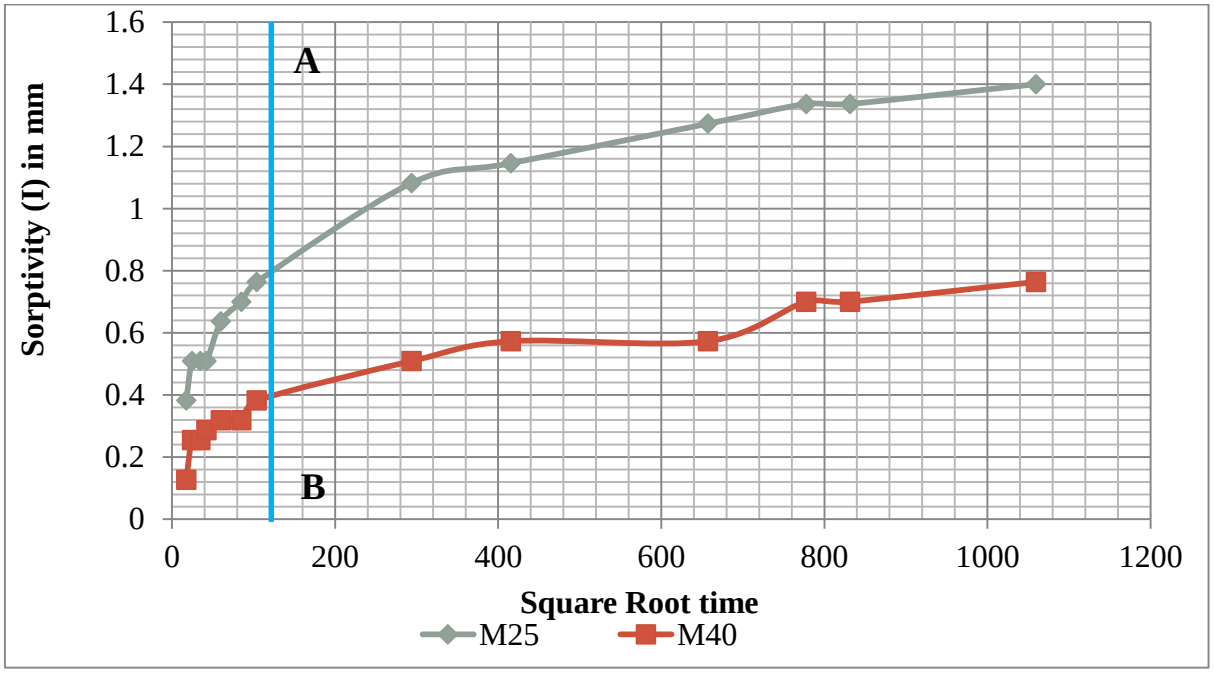


Figure 5-39: Sorptivity results for concrete specimens

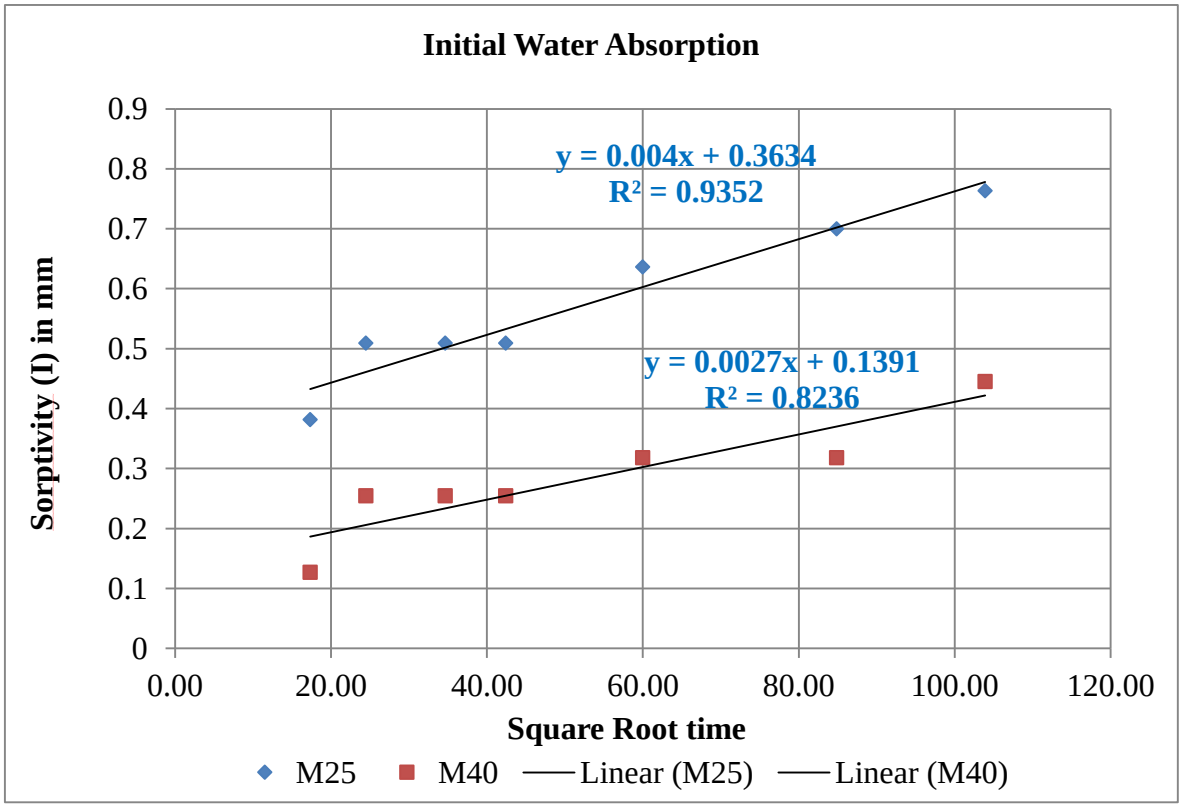


Figure 5-40: Sorptivity results -Initial Absorption

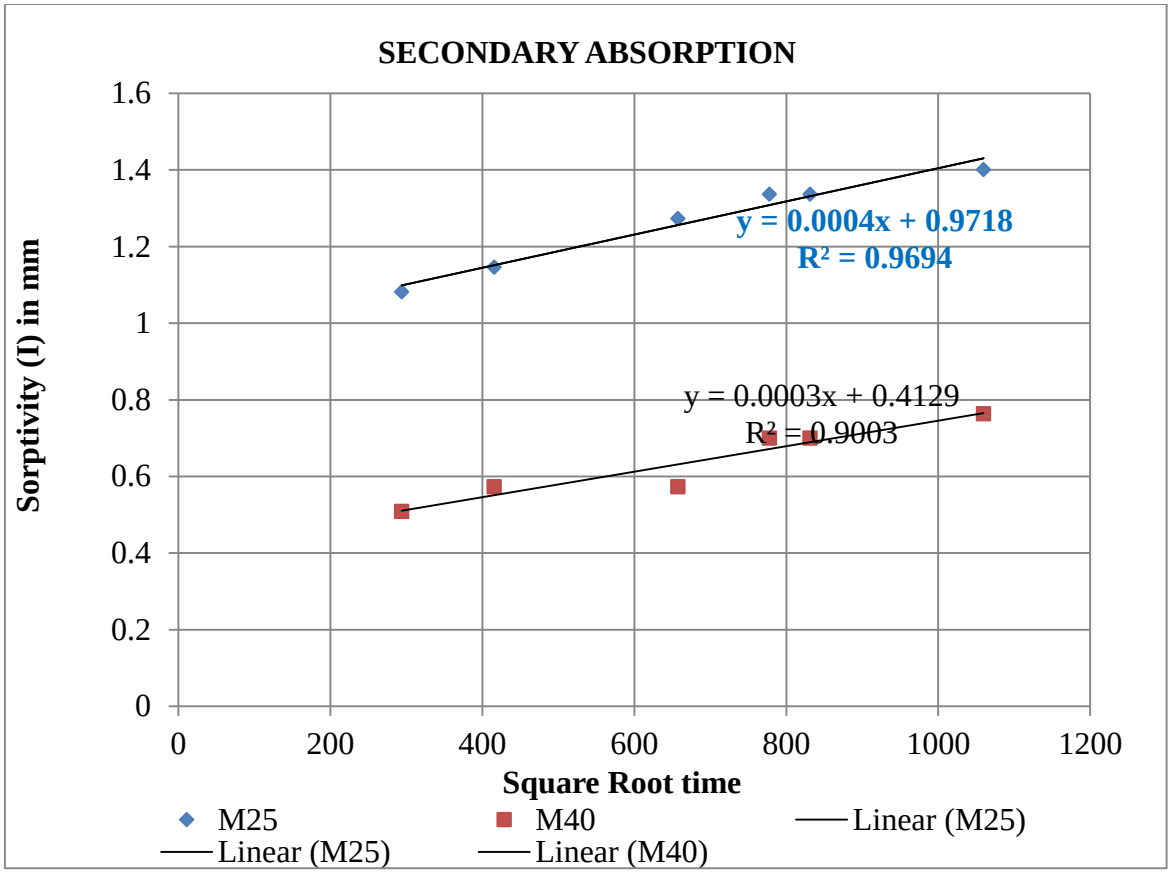


Figure 5-41: Sorptivity results –Secondary Absorption

Table 5-7: Sorptivity Values of concrete

Sr. No.	Grade of Concrete	Sample Description	Sorptivity mm/sec$^{0.5}$
01	M25	With Pure OPC-Control	0.037
02		With stimulated cement	0.004
03	M40	With Pure OPC-Control	0.0265
04		With stimulated cement	0.0027

5.10.2 Acid attack

It is reported that acids attack concrete by dissolving hydrated products and un-hydrated cement as well as calcareous aggregates from the concrete matrix. Concrete deterioration increases as the pH of the concrete decreases due to the acidic environment affecting the passivation resistance of the steel. Thus, laboratory tests were carried out as per ASTM C1898-20 (ASTM C-1898-20) provisions to create an aggressive environment. This is an experiment to estimate the long-term implications. Aggressive climate, rainfall seepage through the soil and liquid storage are some of the sources of acid attack on concrete. The acid molecules from these sources attack the hydrated compound in the matrix. The effect

of acid attack on concrete is determined based on mass loss and compressive strength loss of concrete for 30, 60 and 90 days of curing.

Loss of mass and lowering of compressive strength at the end of 90 days are the indicative parameters of the acid attack. Figure 5-42 shows a plot of the relations of the mass loss and strength loss at the end of 30, 60 and 90 days respectively. The tests were conducted on both the concrete grades viz. M25 and M40 and the results are compared with conventional concretes. It is noted that the resistance to acid attack improves by 2 times in comparison with conventional concrete.

A reduction in compressive strength at the end of 90 days of curing irrespective of the grade of concrete with modified cement is 60% lower than the conventional concrete.

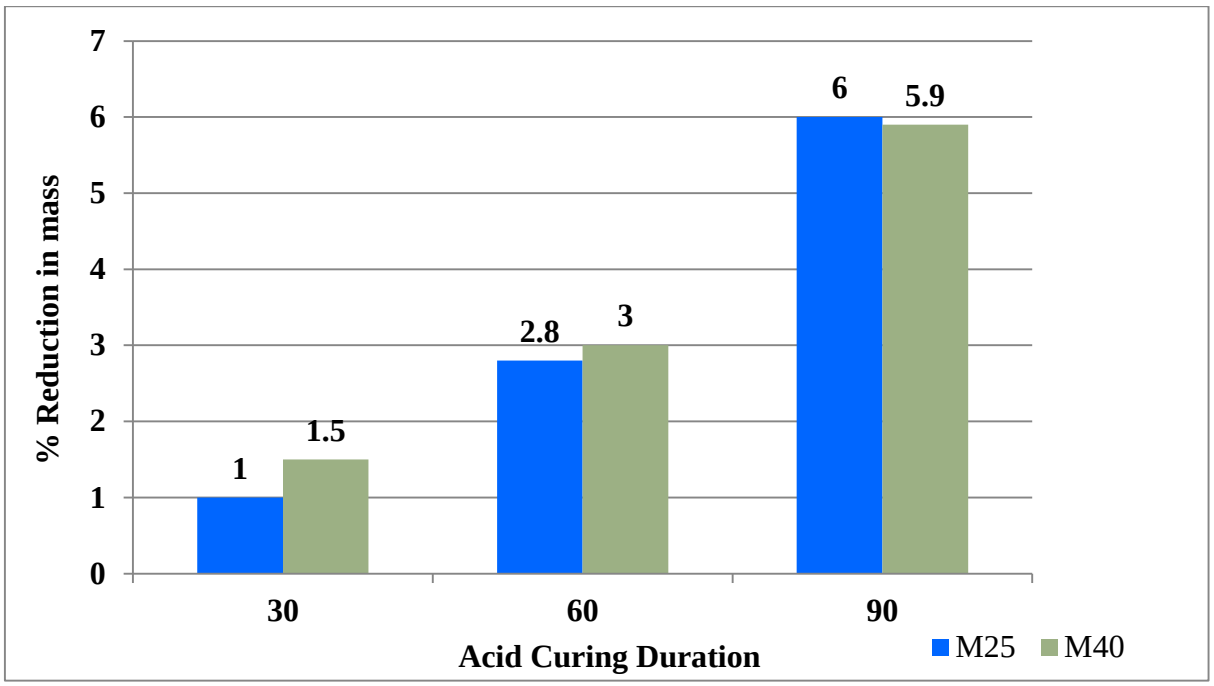


Figure 5-42: Mass loss due to Acid attack

5.10.3 Permeability

The permeability is the rate of seepage of water passing through concrete samples. The rate defines the connectivity of the pores present in the concrete matrix. Higher permeability values influence greatly the durability of concrete. It is calculated using the relation

$$K = \frac{Q}{\left(AT\left(\frac{H}{L}\right)\right)} \qquad \text{--------- Eq. 5-5}$$

Where,

Q = Quantity of water in Milliliters percolating over the entire period of the test

A = Area of the specimen in the face in cm^2

T = Time in seconds over which Q is measured

H = Ratio of pressure head to thickness ratio

L = Thickness of the specimens

Laboratory experiments conducted on permeability show the results as presented in Table 5-7. Typical images of the specimens after the test for both the grades of concrete considered are presented in Figure 5-43. A notable reduction in permeability was observed. This is attributed to better packing of particles, binding and hydration of cement based on PSD.

Table 5-8: Permeability Values of concrete

Sr. No.	Grade of Concrete	Sample Description	Permeability cm/s	Per cent Reduction of permeability with control
01	M25	With Pure OPC-Control	9.0×10^{-10}	66%
02		With stimulated cement	3.03×10^{-10}	
03	M40	With Pure OPC-Control	7.5×10^{-10}	80%
04		With stimulated cement	1.51×10^{-10}	



Figure 5-43: Specimens subjected to permeability testing

Researchers have reported that better resistance to environmental aggressions can be controlled by controlling the water cement ratio of the concrete. This statement is however true provided complete hydration of cement takes place. Keeping the water-cement ratio the same but rearranging the particles of the cement matrix is expected to do better hydration thereby better durability. This technique enhances durability at least by 60% above the conventional cement. The experimental results of sorptivity, acid resistance, and permeability exhibited the fact.

The next Chapter describes and discusses the mathematical model and the experimental validation.

Chapter 6

Mathematical Modelling and Validation of PSD

It is a well proven novel technology of understanding the behaviours of PSD of cement from the foregoing discussions. In order to establish mathematically the PSD's of the samples an excel programme was prepared to estimate particle size and there distribution theoretically. The same was validated with accurate measurement in the laboratory as seen from 5.2. The results obtained from the actual measurements for a given range of particles is in very good relation with the theoretical values generated using the software. Further, the particle packing theoretically was estimated using the software Virtual Cement and Concrete Testing Laboratory (VCCTL). The comparison of the microstructure for the combinations considered in the Table 4-1 is presented in Table 6-1.

6.1 Theoretical Modelling of PSD using Excel and its validation

Bentze 2010, used the law of combinations while estimating the theoretical PSD and its validation by actual measurement using LASER diffraction technique revealed that the results are in good agreement with the predicted ones. This law of combination stated will hold good for any number of combination as long as weighed averages are considered. The law of combination for blended components is presented in Eq. 6.1.

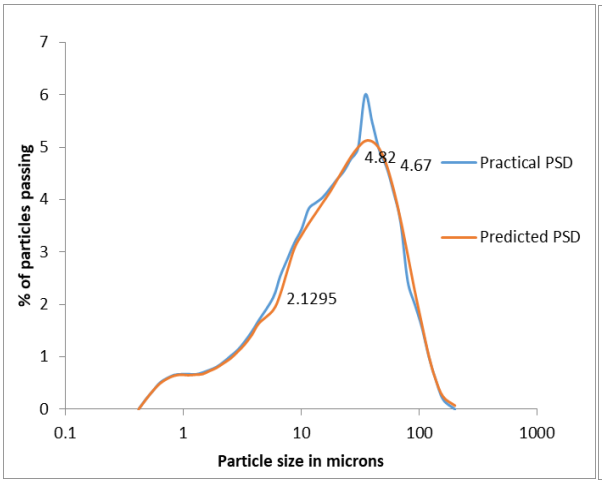
$$\mathbf{P(Blend) = P(Component\ 1)x\ mass\ fraction + P(Component\ 2)\ x\ mass\ fraction} \quad \text{.....Eq. 6-1}$$

Where,

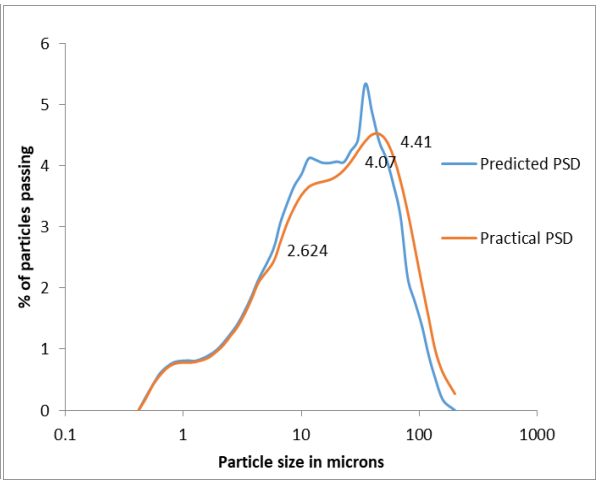
P – Property of interest (Differential volume)

Theoretical PSD was established using the relation for the combination listed in Table 4.1. Two components were considered namely OPC and stimulator. Mass fractions

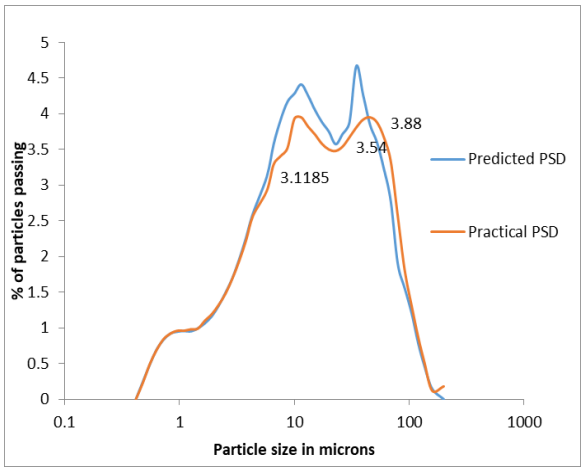
predetermined were subjected to theoretical PSD and there estimation is presented in the Figure 6-1. The results as obtained from LASER diffraction technology for the same combinations was also overlapped respectively in the Figure 6-1. It is noted that the results are in good agreement for a given range of particles. The accuracy of the results is also in the range of 95-98% confidence limits.



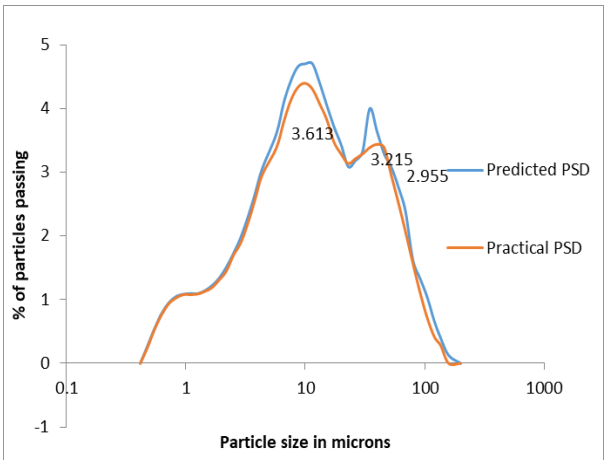
(a) S10



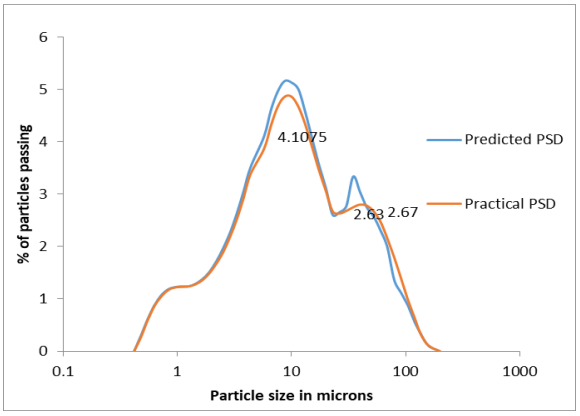
(b)S20



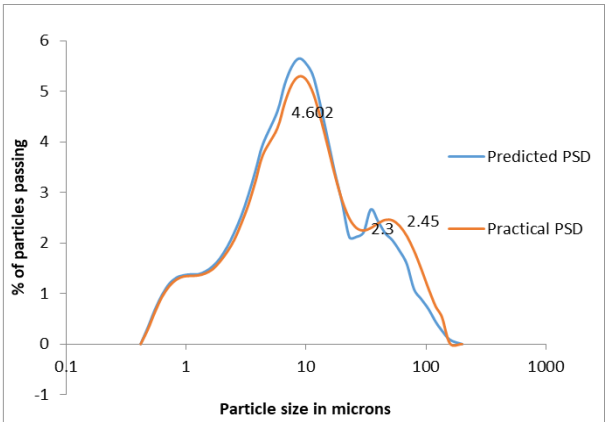
(c) S30



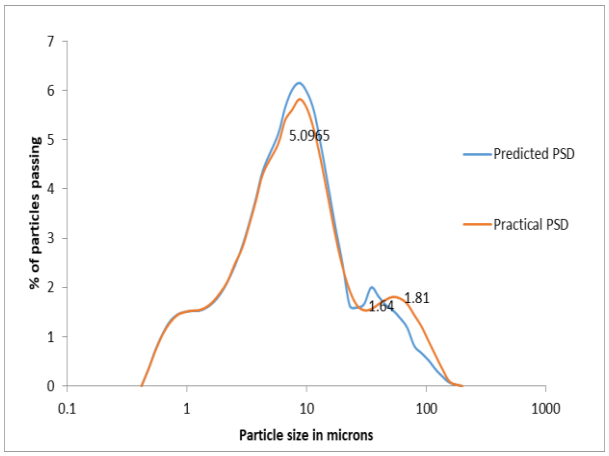
(d) S40



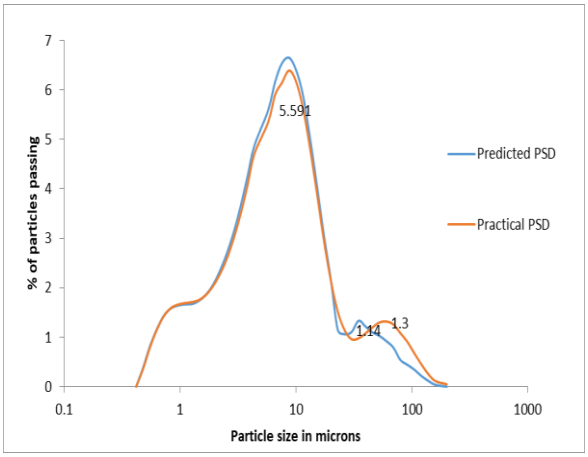
(e) S50



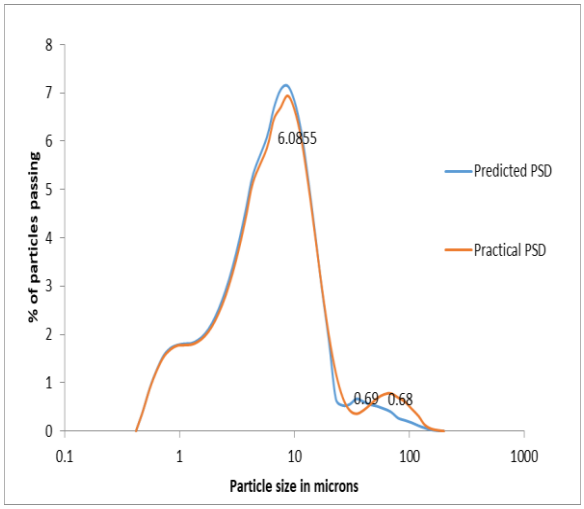
(f) S60



(g) S70



(h) S80



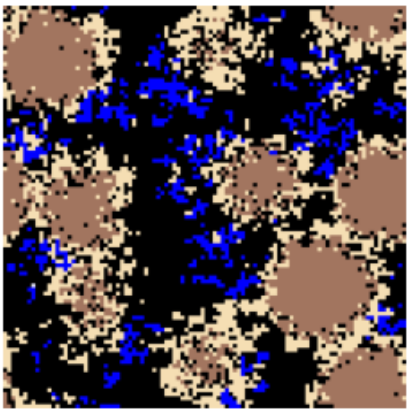
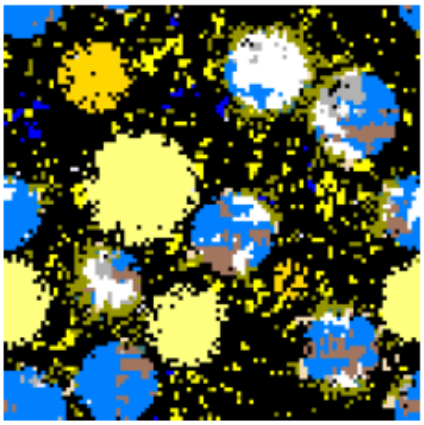
(i) S90

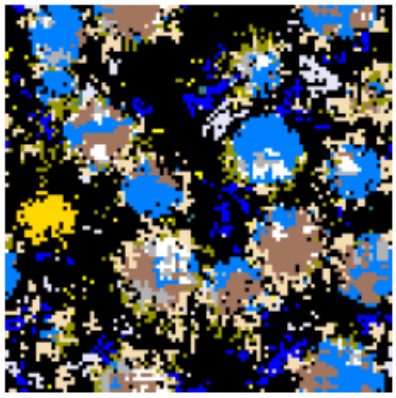
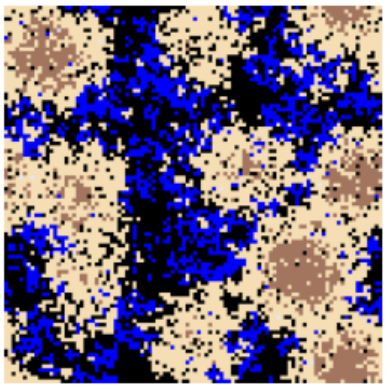
Figure 6-1: Comparison of Predicted PSD and experimental PSD

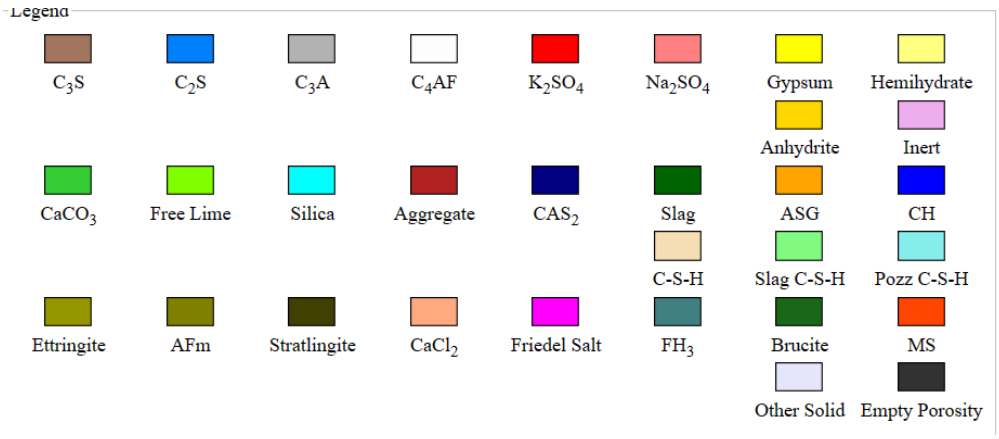
6.2 Simulation of Micro Structure using VCCTL for the data in article 6.1

VCCTL version 9.5 software developed by NIST- National Institute of Standards and Technology- United States. It provides a virtual testing laboratory environment that can be used by concrete scientists, engineers, and technologists for virtual testing of cement paste and concrete materials. It can create virtual materials, using carefully characterized cement powders, supplementary cementitous materials, fillers, and aggregates. It can simulate the curing of these materials under a wide range of conditions. It can also calculate their thermal, mechanical, and transport properties as a function of their processing. The present scope of the usage of VCCTL in the work is restricted to the comparison of microstructure images for the combinations refereed in Table 4.1. Referring to Table 6.1

Table 6-1: Simulation of microstructure using VCCTL

Nomenclature	VCCTL Microstructure Output image
OPC	
S40	

S50	
S100(Stimulator)	



Environmental suitability, practical applications, and ease of production ultimately cost related. A comparison of the cost of materials in terms of cement alone and concrete are estimated keeping the desired strength as a target. Chapter 7 presents the details of the same.

The above Chapter describes mathematical model and experimental validation and the next Chapter discusses sustainability analysis and cost analysis.

Chapter 7

Sustainability and Cost analysis

The importance of sustainability for human existence on Earth, societal growth, and human development is widely recognized. Concrete, being one of the most commonly used construction materials, is closely tied to economic development. However, the production of Portland cement, a vital component of concrete, contributes significantly to the emission of CO_2, a greenhouse gas. The cement industry is recognized as one of the most polluting industries in terms of CO_2 production. The carbon footprint of cement plays a crucial role in meeting the global CO_2 reduction targets set by the International Energy Agency (IEA). According to reports from the IEA, annual CO_2 emissions worldwide reached approximately 372 GT, with the cement sector accounting for around 8% of this total. In response to the stringent conditions outlined in the Kyoto Protocol, the cement industry has actively sought alternative production methods and process changes to address environmental concerns related to GHG emissions, as well as to address issues regarding natural resource usage. These efforts are essential for the sustainable development of the cement and concrete industry.

7.1 Environmental Aspect

The cement industry contributes to approximately 8% of CO_2 released by industrial activities on a global scale. (Sandra Nunes a, 2021). Table 7-1 below describes detailed calculations of the Carbon footprint of cement production in terms of conventional OPC and the proposed stimulator cement content on the basis of targeted strength.

Table 7-1: Amount of CO_2 released by the cement industry at the production level

Sr. No.	Details	Amount of CO_2 released (tonnes)
I	**1 tonne of OPC**	**0.8**
II	CO_2 released during "Stimulator" production	
b)	Electricity required for grinding OPC to Stimulator 30kWh of electricity	0.0285

	1 tonne stimulator	**0.8285**
III)	**1 tonne of modified cement S45**	**0.8128**
IV)	**0.75 tonnes of modified cement (S45)**	**0.6096**

7.2 Strength Efficiency of cement

Strength efficiency is calculated for cement samples using Eq. 7.1. It relates 28 days of compressive strength with the quantity of cement. Briefly, strength efficiency is strength achieved by the unit amount of cement (kg/m^3).

$$Strength\ Efficiency, SE\left(\frac{MPa}{kg}\right) = \frac{28\ days\ compressive\ strength, MPa}{Amount\ of\ cement\ used\ in\ mortar, \frac{kg}{m3}}\ x\ 100 \qquad \text{Eq. 7-1}$$

Sample calculations of data using equation 7.1 has been presented in appendix. With the increase in fineness, the efficiency has also increased since grinding increases the surface area available for hydration. The calculated strength efficiency values are depicted graphically in Figure 7-1. S40 and S50 are 11-20% more efficient than OPC. The improved efficiency can be pursued in 2 different ways viz. a) To achieve strength higher than OPC keeping cement quantity to be same. b) To achieve strength equivalent to that of OPC with reduced cement quantity.

The improvement in efficiency leads to a reduction in cement quantity requirement to achieve known strength which is also ascertained by concrete testing explained **subtopic 3.5**.

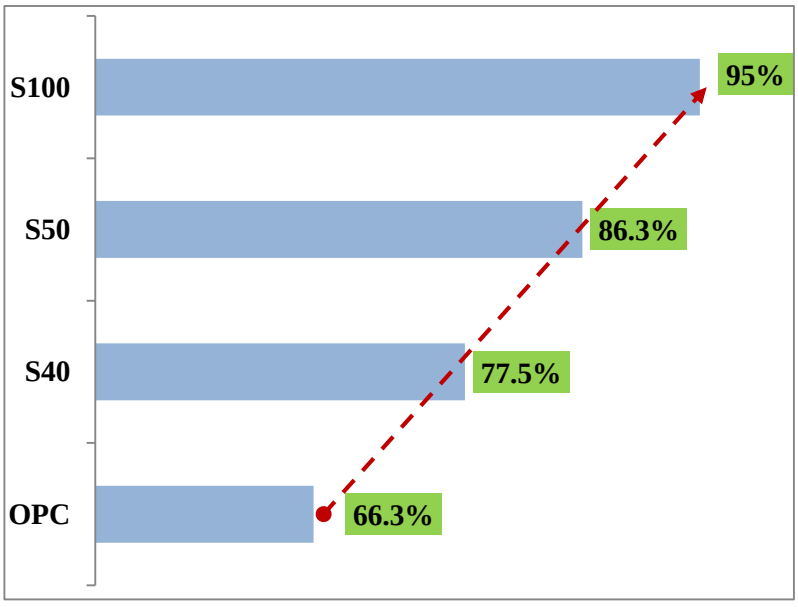


Figure 7-1: Strength efficiency values of cement mixes

7.3 Environmental Efficiency of cement

Environmental efficiency relates the calculated global warming potential with the compressive strength at a given cement composition found by using Eq. 7..2 stated below (Maciej Zajac, Gerd Bolte, Jan Skocek,2021.) Like strength efficiency increase in fineness leads to increase in environmental efficiency as depicted graphically in Figure 7-2 . S40-S50 are 10-19% efficient than OPC samples from environmental aspect.

.

$$Environmental\ Efficiency, EE\left(\frac{MPa}{kgCO2}\right) = \frac{28\ days\ compressive\ strength\ in\ MPa}{Global\ warming\ potential\ of\ cement,(kg)}\ x\ 100$$

.....Eq. 7-2

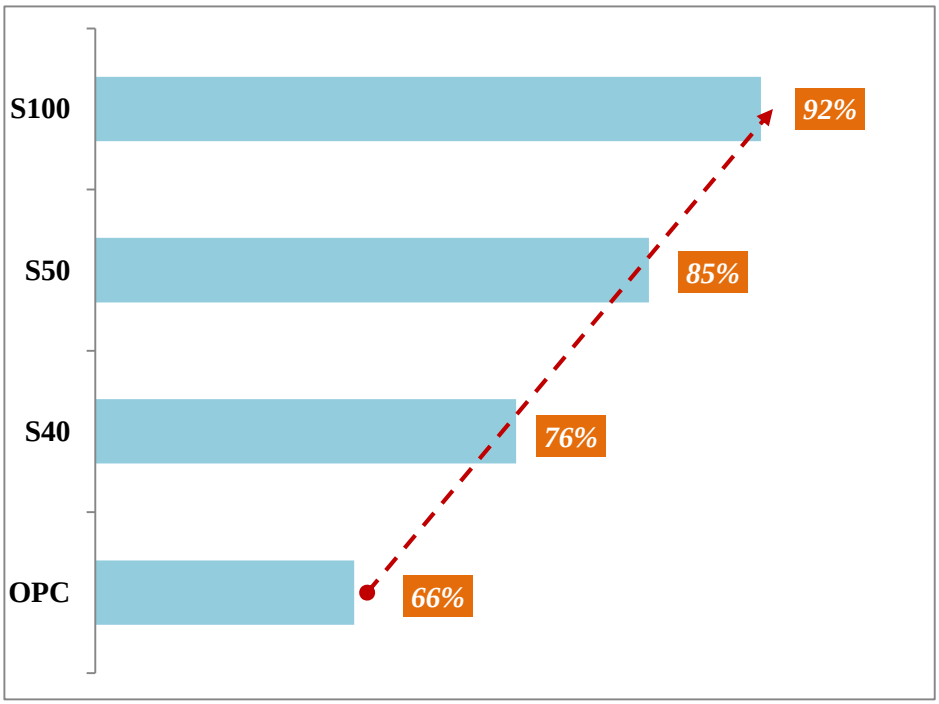


Figure 7-2: Environmental efficiency values of cement mixes

Sample calculations of data using equation 7.2 has been presented in appendix.

Though grinding of cement requires extra energy in the form of electricity it is compensated by the enhanced efficiency of cement samples leading to a reduced carbon footprint as witnessed in Table 7-1.

Percentage increase in water requirement and compressive strength for the mixes in study are calculated based on the results stated earlier. The fineness of cement is closely related to many parameters such as water requirement, compressive strength, setting times and overall performance of cement.

The precise relation between percentage increase in fines (<25µm) and percentage increase in a) Compressive strength (b) Water requirement and (c) Efficiency is established graphically in Figure 7-3. This precise relation can be used to estimate or predict vales for intermediate percentage of fines.

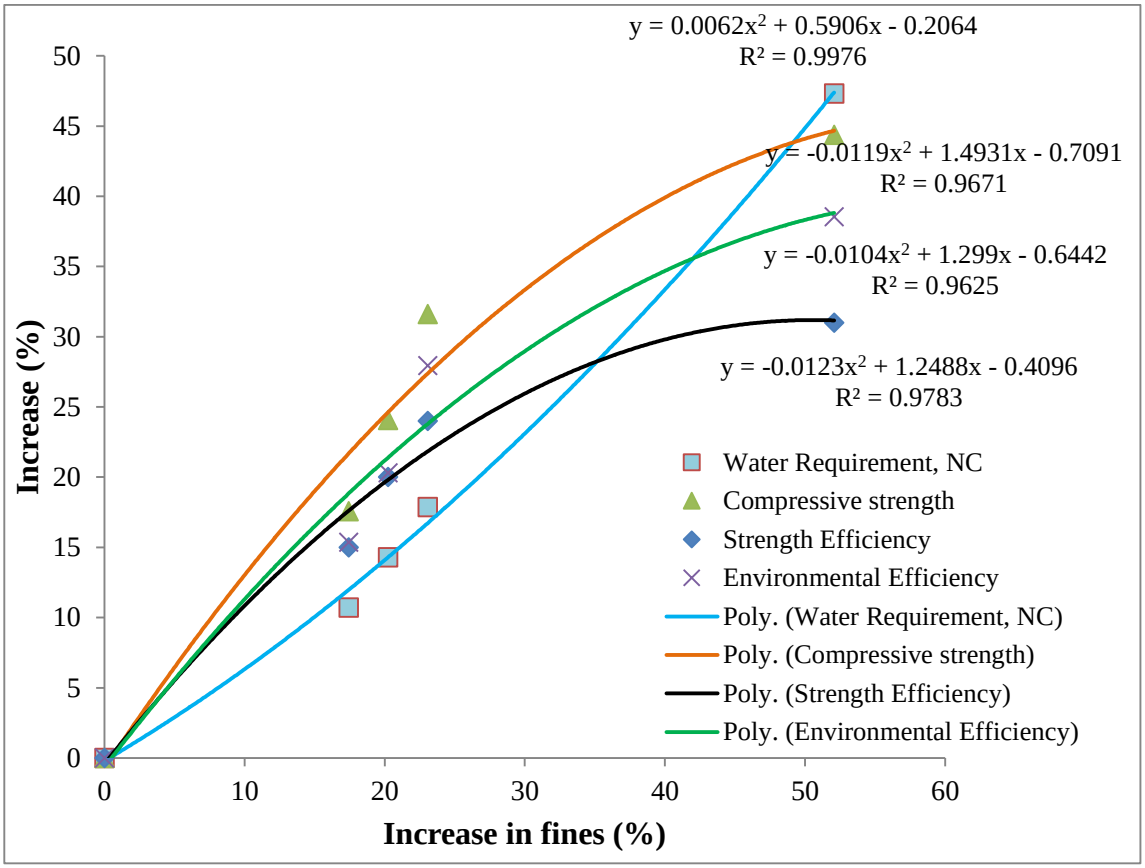


Figure 7-3: Relation between percentage increase of values of in particles < 25microns and water requirement, compressive strength, strength and environmental efficiency

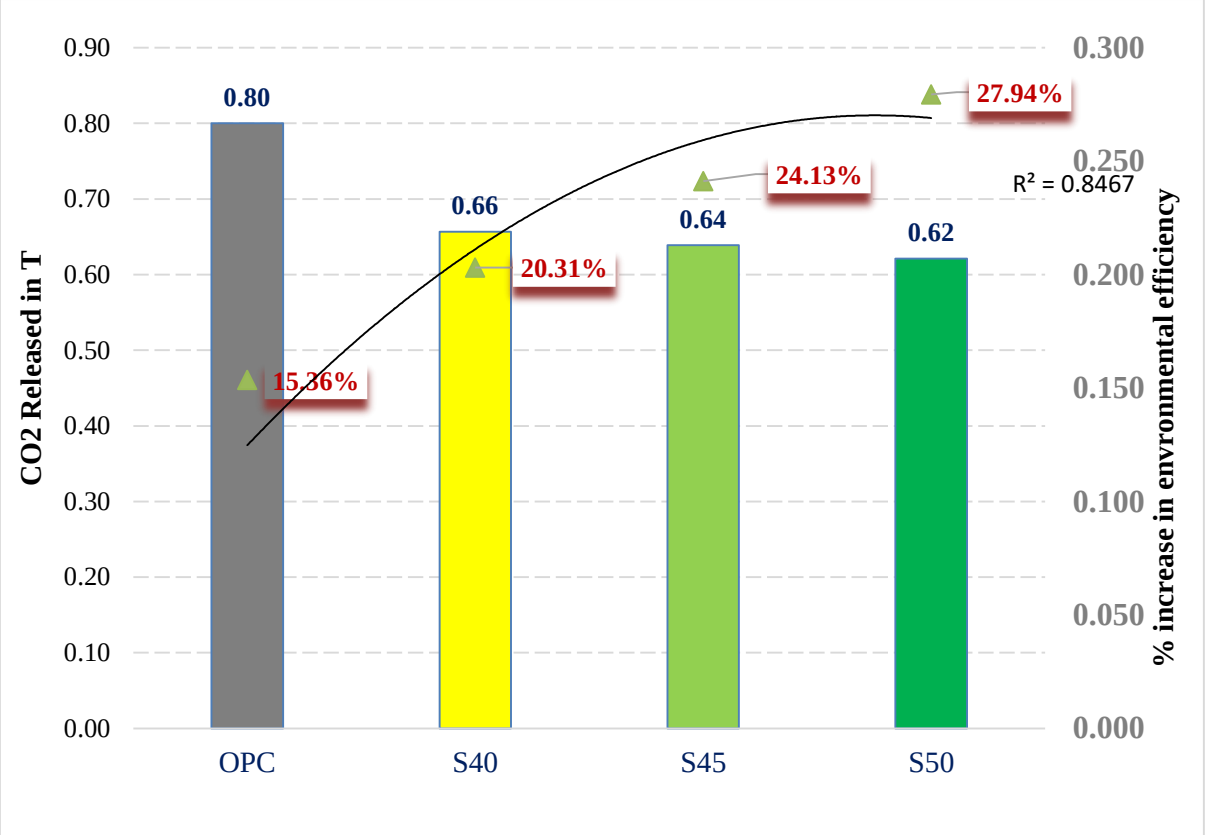


Figure 7-4: Comparison of CO_2 released and environmental efficiency

The Figure 7-3 above clearly depicts that if percentage fines (< 25µm) are wisely engineered performance of cement can be enhanced. Except water requirement the trend line of compressive strength, strength efficiency and environmental efficiency followed similar pattern.

Figure 7-4 presents a clear relation between carbon foot print and environmental efficiency of the cement mixes.

7.4 A. Cost Analysis (Production stage)

Table 7-2 and **Table 7-4** present detailed cost analysis of cement at production level and effect on cost analysis due to enhanced efficiency. **Table 7-4** presents the details of cost analysis of concrete produced using stimulated cement mix.

Table 7-2: Cost analysis of cement at production level

Sr.No	Description	Cost	Dimension
1	Price of OPC per tonnes	₹ 8,000.00	per tonne
2	Extra cost of production of Stimulator	₹ 2,800.00	per tonne
3	Price of OPC per kg	₹ 8.00	per kg
4	Price of Stimulator per kg	₹ 10.80	per kg
5	Price of Cement combination 40%S + 60% OPC	₹ 9.12	per kg
6	**Price of Cement combination 45% S + 55% OPC**	**₹ 9.26**	**per kg**
7	Price of Cement combination 50% S+ 50% OPC	₹ 9.40	per kg
8	Price of OPC per bag	₹ 400.00	per bag
9	Price of Stimulator per bag	₹ 540.00	per bag
10	Price of Cement combination 40%S + 60% OPC	₹ 456.00	per bag
11	Price of Cement combination 50% S + 50% OPC	₹ 470.00	per bag
12	**Price of Cement combination 45% S + 55% OPC**	**₹ 463.00**	**per bag**

Table 7-3. Cost reduction in cement due to enhanced efficiency

Sr. No.	Name of test /Parameter	OPC	40%MC	45%MC	50%MC	100%MC
1	**Compressive strength @ 28 days**	53	62.3	66.05	69.8	76.5
2	**% increase in compressive strength**	-	17.55	24.62	31.70	44.34
3	**Cost of cement for compressive strength 53N/mm^2**	1000	824.53	753.77	683.02	556.60
4	**Number of bags of cement required for achieving compressive strength 53 N/mm^2**	1	0.82	0.75	0.68	0.56
5	**% reduction in quantity**		17.5%	24.6%	31.7%	44.3%
6	**Cost of cement per kg**	₹ 8.00	₹ 9.12	₹ 9.26	₹ 9.40	₹ 10.80
7	**Cost of cement for reduced amount of cement**	₹ 8.00	₹ 7.52	₹ 6.97	₹ 6.42	₹ 6.01
8	**Cost of cement per bag**	₹ 400.00	₹ 375.98	348.50	₹ 321.02	₹ 300.57
9	**% reduction in cost**	0%	6.0%	15%	19.7%	24.9%

Table 7-4: Cost reduction in concrete due to enhanced efficiency

Concrete Grade With OPC 43	Cement proposed per m3 of concrete		% Quantity saving in cement	Cost of Cement		% Reduction in cement cost	Remarks
	Cement content for Existing plant Mix Design	**GEC Team design with developed cement**		**JK OPC 43 Gr**	**Cement developed by team GEC (@ 9.26/kg)**		
M 25	**360**	**300 (Minimum as per IS)**	**20%**	2880	1250+1320 =2570	**10.76%**	7.2 bags 6.0 bags
Strength achieved, MPa	1 day: 7-8 7 Day: 17-18	1 day- 12.25 7 day- 24.6					
M40	**430**	**330**	**23.25%**	3440	3055.8	**11.19%**	8.6 bags 6.6 bags
Strength achieved, MPa	1 day: 12-14 7 Day: 26-28	1 day- 18.3 7 day- 38					

The next chapter presents the specific conclusions, contribution of the work, outcomes of the study as conclusions.

Subsequent section mentions the conclusions drawn from the entire study.

Chapter 8

Summary and Conclusions

8.1 Summary

Cement has transformed into a modern construction material over a period of time. The grade 33 is outdated and higher grades are being used commercially as an efficient binder in the concrete matrix. Improvement in the properties of cement over a period of time, depending on the quality of the raw materials available in one hand and the chemical composition alternates at the factory on the other hand. Some properties of cement are influenced by the grain size and others by its composition. An effort has been made in the study by rearranging the PSD of the system to enhance its performance. Referring to the suggestions of Higginsons, a framework of the study was planned and executed to yield the results. The fineness of cement was so designed to yield the desired flow while considering its water of consistency such that there is no additional water required with the increased fineness. An experimental detailed study resulted in optimizing the fineness. An excel program further confirmed the optimized combination of the PSD. Microstructure was further validated using VCCTL. The cement grounded to 5600cm^2/g Blaine's when blended with commercially available cements in an optimized proportion results the material which will have a Blaine value of 4200 cm^2/g. This fine material brought a phenomenal change in the quality of cement is termed as Stimulator.

Mandatory checks for the properties further indicated its superiority over the existing cements. The performance in terms of strength, heat of hydration, durability were assessed and found to be far superior to OPC. The early strength achieved was really a commendable one. The tests on modified concrete using stimulated cements in comparison with the conventional one has resulted into the savings of the material to an extent of 25%. In terms of sustainability, resource material conservation it had outperformed with the conventional material. Several tests were also performed to understand the compatibility of curing compounds with the stimulator cement. Special application with high density materials this cement also performed without any negative

impacts. The cement so developed is also compatible with all types of admixtures without any adverse effects when compared with conventional concretes.

OPC is being the fundamental material in formulating double blended, triple blended and other possible combinations available with the cementitious materials. Hence, the scope of the work was restricted only with OPC in the present study. The scope further can be extended to the other blends also.

8.2 Conclusions

The proposed objectives of the study were achieved through a structured framework of study. Elaborative discussions on historical and current scenario of cement industry is presented in Chapter 1. Considering the anticipated increase in demand and the need of sustainability in cement and concrete production, a detailed review of literature was also carried out and presented in Chapter 2. The effect of cement fineness on various properties has been reviewed from the literature. From the comprehensive literature, the research gap is identified and objectives were framed for execution. It was proposed to engineer the PSD of cement to enhance the efficiency and reduce the cement quantity requirement to achieve the desired compressive strength. The applications to high density concrete using engineered PSD were compared with conventional concrete. Materials in the matrix of concrete were characterized and reported in Chapter 3. The methodology and the scheme of experimentation followed to investigate various physical, mechanical, durability characteristics of mortar and concrete specimens have been presented in Chapter 4. The methodology of production of stimulator is also added in Chapter 4 to give an insight better. Care was taken to satisfy the requirements of sampling, casting and interpreting the results as per the provisions in every experimentation carried out. Elaborative discussions on test results and analysis including the reasoning is narrated in Chapter 5. The Chapters 6 and 7 provides the economic feasibility and environmental sustainability of the material in terms of concrete production.

Conclusions drawn from the study

The following conclusions are drawn to highlight the findings of the work:

1. The fineness of conventional OPC, around 3200cm^2/g, does not meet the PSD requirements for full utilization. Consequently, following a study, a stimulator

was introduced, leading to significant changes in the strength and durability parameters. Generally due the belief/ assumption of increased demand in water due increase in fineness Cement manufacturing was restricted the production to 43 and 53 grades

2. The study concludes that engineering the PSD by averaging the amalgamation of commercial OPC and the stimulator to a Blaine's value of around 4200cm^2/g is vital for achieving the necessary fines for initial high early strength as well as strength with age. This novel concept, as referred to by Higginson, has been proven to be more suitable for introducing a greater amount of fines for a given water content.
3. The performance of cement is elucidated through the categorization of particle sizes into four groups: < 3, 3-9, 9-25, and > 25 µm. Particles < 9 µm demonstrate a direct and linear relationship with water requirement and compressive strength, unlike particles > 25 µm. Therefore, the overall efficiency of cement hinges on the percentage of particles < 9 µm.
4. The optimized combination S45 reduces the average particle size by 58% compared to OPC, with only a 10% increase in water content. This blend proves efficient in enhancing strength over time compared to normal OPC by incorporating engineered PSD graded particles.
5. The formation of a double peak PSD in the optimized cement mix (S40, S45, and S50) is a significant and specific outcome of this study. This phenomenon results in selective hydration of fine and coarse particles, unlike what is observed in OPC or using the stimulator alone. Engineered PSD (S45) completes the hydration process to an extent of over 90% by 10^{th} day.
6. The study contributes to the demarcation of active zone by engineered PSD (Reference to the Figure 5-13) PSD figure
7. The high early strength achieved, particularly the one-day strength of 32 MPa, represents an 80% increase compared to conventional 43 or 53 grade cements. The third day and Seventh day compressive strength of S45 combination is 40% and 20% higher than OPC.
8. Setting time typically decreases in a linear fashion with an increase in stimulator content; however, for the S45 condition, this trend does not hold true. Figure 5.20 illustrates a sudden drop, indicating little deviation from conventional OPC.

9. The engineered PSD controls the heat of hydration too. It is observed that a marginal or almost nil surge in comparison to OPC.
10. S45 is also compatible with all curing compounds and other admixtures at par with OPC.
11. The studies comparing concrete with OPC and the stimulator-assisted cement S45 have shown that the latter not only saves up to 25% of cement while achieving the desired strength, but also proves to be efficient in achieving a high aggregate-cement ratio. Irrespective of the density of the concrete materials, S45 outperforms OPC.
12. Casting of concrete with S45 did not pose any site specific problems like mixing, transporting, placing and vibration.
13. Engineered PSD of the combination S45 is environmental friendly by contributing the savings in carbon foot print. The studies and calculations thereby indicate a reduction of CO_2 production per tonnes of production from 0.8 to 0.621 while achieving a given target strength.
14. Tests on durability viz. Sorptivity, Acid resistance and permeability showed an improvement of 60% in comparison to the normal OPC.

8.3 Major contributions arising out of the study

1. The concept of PSD is redefined.
2. It has been proved that the fineness up to a certain level does not bring appreciable changes in water demand by the cement and there are no adverse effects on its properties.
3. High early strengths are achievable with a simple PSD technique
4. The novel concept of presenting the invention of participation of particles in a sequential manner in the form of double peaks is a result of engineered PSD.
5. The study contributes to the demarcation of active zone by engineered PSD (Reference to the Figure 5-13)
6. Given a targeted strength engineered PSD will always outperforms in all aspects of strength and durability while saving the cement by 25% and also contributes the material as the sustainable one in the environment.
7. Higher initial strengths results in early removal of formworks thus speedy construction can be achieved.

8.4 Future Scope for Study

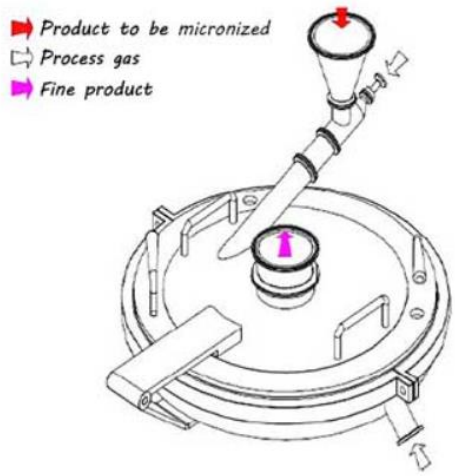
It is a fact that the cement is the main binding material in the matrix of concrete production. OPC being the mother for the generation of other blended cements. The performance of other cementitious materials in combination will add a better contribution with the extension of the studies cited in the above work. The present study has its limitation by introducing the concept for only OPC while characterizing the engineered PSD and thereby comparing its efficiency. Similar such studies on blended cements will add a rich content to the literature and the industry associated.

Annexure A

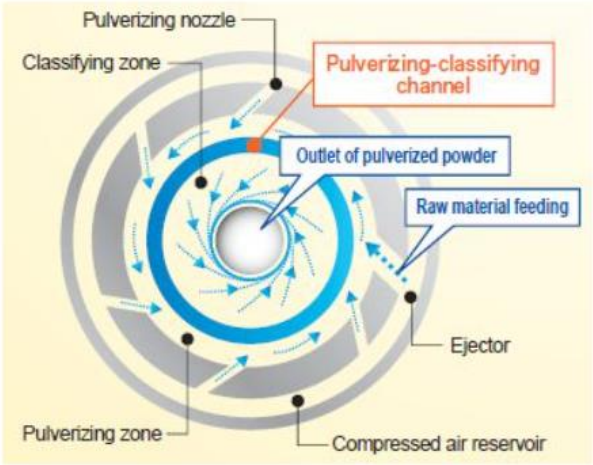
Jet Mill Working Principle

This text aims to explain the operational concept of a jet mill, also known as a fluid energy mill, which is utilized for micronizing solid materials. Micronization refers to the reduction of particle size to less than 10 microns, a process in which jet mills are employed for solid chemicals, pharmaceuticals, pigments, minerals, and other substances like heat-sensitive, corrosive, and abrasive materials.

Often viewed as a mysterious process, micronization through jet mills yields both fine powder and coarse particles. The operation of jet mills occurs in environments of compressed air, gas, or high-pressure superheated steam. These mills lack moving components, preventing contamination from external grinding media. Additionally, the absence of attritional heat generation makes jet milling ideal for grinding heat-sensitive and waxy materials.



Jet mill works on fluid energy. Micronizing and classification take place in a shallow, cylindrical chamber. High pressure air or steam is injected in this chamber through the specially designed nozzles placed at regular distance, on the peripheral wall. The axis of each jet is tangential to the circumference of a smaller, imaginary, concentric circle.



During operation, the material is introduced into a vortex by following an engineered tangent circle, which accelerates its movement. The strong velocity gradients near the jet cause the suspended particles of the material to collide, leading to attrition and reduction. Size reduction occurs primarily due to the high-velocity collisions between particles of the process material itself, without the involvement of grinding media. The jet fluid exits through a central outlet at the top of the chamber, carrying the micronized particles with it to the cyclone collection system. Heavier oversized particles are retained in the grinding chamber by centrifugal force until they are micronized to the desired size.

The feed particle size is a critical factor, limited by the size of the feed injector. For mills of 200-300 mm, the maximum feed size can be 1.5 mm, and for smaller-size mills, the feed size is correspondingly finer.

The fineness of the end product is influenced by several operational and physical factors, including feed rate, nozzle size, nozzle pressure, nozzle angle, airflow rate, feed particle size, chamber diameter and width, and product outlet diameter. While these variables can be adjusted during operation, it is more common for only the feed rate to be varied to achieve the required particle-size distribution.

It's important to note that the spiral jet milling process is not solely determined by the jet mill itself, but involves the integration and control of different machines and components. These include a source of fluid energy, a feeder, a cyclone separator, a dust collector, and other necessary accessories.

Annexure B

Sample calculations for the following equations

B.1. Law of mixture for PSD estimation

$$\mathbf{P(Blend) = P(Component\ 1)x\ mass\ fraction + P(Component\ 2)\ x\ mass\ fraction}\quad \mathbf{Eq.\ 1}$$

Where,

P – Property of interest (Differential volume)

$$\mathbf{P(S10) = 3.15x\ 0.1 + 0.78x\ 0.9 = 1.017}$$

Particle size in microns	MC	OPC	S10	S40	S50	S60	S90
2.512	3.15	0.78	0.1x[3.15]+0.9x[0.78]=1.017	1.728	1.965	2.202	2.913
5.754	6.58	1.635	2.1295	3.613	4.1075	4.602	6.0855
8.71	7.64	2.68	3.176	4.664	5.16	5.656	7.144
10	7.25	3.01	3.434	4.706	5.13	5.554	6.826
26.303	0	5.31	4.779	3.186	2.655	2.124	0.531
45.709	0	5.49	4.941	3.294	2.745	2.196	0.549

8.5 B.2. Strength Efficiency

Strength efficiency is calculated for cement samples using Eq. 7.1. It relates 28 days of compressive strength with the quantity of cement. Briefly, strength efficiency is strength achieved by the unit amount of cement (kg/m^3).

$$\boldsymbol{Strength\ Efficiency, SE\left(\frac{MPa}{kg}\right) = \frac{28\ days\ compressive\ strength, MPa}{Amount\ of\ cement\ used\ in\ mortar, \frac{kg}{m3}}\ x\ 100}\quad \mathbf{Eq.\ 2}$$

With the increase in fineness, the efficiency has also increased since grinding increases the surface area available for hydration.

Mortar Sample details

For compressive strength confirming to IS 4031. Part 6

Cement = 200g & Sand = 600g. Mortar Cube size = 50 x 50 x50 mm

Amount of cement used in mortar (1 cube) = 80g

Using above equation following calculations are presented in tabular format

Cement mix Name	**OPC**	**S40**	**S50**	**S100**
28 days Compressive strength, MPa	53	62.32	69.76	76.52
Amount of cement used in mortar, g	80	80	80	80
Strength efficiency (As per Eq 2)	53/80=66	77.5	86.3	95

8.6 B.3. Environmental Efficiency of cement

Environmental efficiency relates the calculated global warming potential with the compressive strength at a given cement composition found by using Eq. 7..2 stated below (Maciej Zajac, Gerd Bolte, Jan Skocek,2021.).

$$\boldsymbol{Environmental\ Efficiency, EE\left(\frac{MPa}{kgCO2}\right) = \frac{28\ days\ compressive\ strength\ in\ MPa}{Global\ warming\ potential\ of\ cement,(kg)}\ x\ 100} \quad \textbf{Eq. 3}$$

Cement mix Name	**OPC**	**S40**	**S50**	**S100**
28 days Compressive strength, MPa	53	62.32	69.76	76.52
Carbon dioxide released (Carbon foot print) per tonne of cement	0.8	0.811	0.814	0.828
Environmental efficiency (As per Eq 3)	53/0.8 =66	76	85	92

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