

USE OF CUPOLA SLAG AS FINE AGGREGATE IN CONCRETE

A thesis paper by

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Submitted in partial fulfilment of the requirements for the degree of

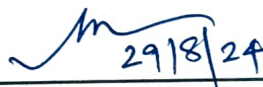
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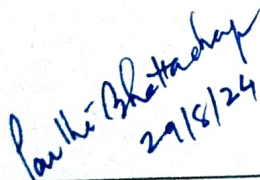
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Abstract:

The growing scarcity of natural sand and the increasing demand for sustainable construction have driven the search for alternative materials in concrete production. One promising option is cupola slag, an industrial waste by product generated during cast iron production in cupola furnaces operating at temperatures between 1400°C and 1600°C. The disposal of cupola slag is a matter of burden for the manufacturers of casting industry.

It may be mentioned that cupola slag contains only 10.7% CaO which is quite less than Granulated Blast Furnace Slag (GGBFS). Thus, it is not economically viable to replace the cement partially after grinding. Although it contains substantial amount of amorphous silica, the use of cupola slag as partial cement like fly ash as it requires fine grinding and will generate carbon footprint. Thus, recycling of cupola slag as a partial replacement for aggregate. This requires less energy compared to fine grinding. This will not only help reduce waste but also conserves natural resources. Few researchers have used cupola slag as a coarse aggregate in concrete.

Based on the above background, the present study focuses on the use of cupola slag as a fine aggregate replacing the natural sand to develop a practical way of solid waste management. The study is limited to normal grade concrete of grade M25. Cupola based fine aggregate has been prepared from chunks by hand grinding in the laboratory. The grading has been prepared as Zone II (as per IS 383-2017) which is similar to natural sand. It may be mentioned here that this can be made to Zone I and can be suitably used as manufactured sand. The cupola slag is used as fine aggregate (FA), partially replaced by 0–50% (by weight) in the step of 10% (by weight).

The experimental results indicate that the dry density of concrete mix increases with the increased weight percent of cupola slag based FA (CS) in concrete. The compressive strength and split tensile strength at 28 day specimens increased with the increase of replacement percentage of Cupola slag up to 40% replacement compared to the control specimen. In such cupola based concrete, the water absorption of concrete is less by about 1.23% for 40% replacement compared to the control specimen. The chloride ions

penetration in cupola based concrete decreases with the increase of Cupola slag as fine aggregate in concrete. Field Emission Scanning Electron Microscope (SEM) images revealed that use of Cupola slag modifies the concrete microstructure and fill the small pores. EDS and XRD based microstructure analysis indicates such improvement in microstructure with the development of new compound in the cupola slag based concrete. However more study is needed for other concrete grade and with the mechanically prepared cupola based fine aggregate.

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CHAPTER 1

INTRODUCTION:

1.1 Excessive Sand Mining and its Environmental Impacts:

Concrete, the single most widely used building material around the globe, is a heterogeneous composite that consists of combination of readily available basic building materials including cement, water, coarse aggregate, fine aggregate, and in some cases, admixtures, fibres or other additives, according to the need. When these ingredients are mixed together, they form a fluid mass that is easily moulded into any shape. Over time, when it is cured sufficiently, the cement forms a hard matrix which binds the rest of the ingredients together into a durable stone-like material, called concrete.

The reason behind the enormous use of concrete in the construction sector lies in its versatile, reliable and sustainable nature, because of its strength, rigidity, durability, mould-ability, efficiency and economy.

Humans have been using concrete in their pioneering architectural feats for millennia. Due to the ongoing boom in the housing sector and other developmental activities in the construction field, the demand of concrete is increasing with a very rapid pace all over the world. 4.1 billion tonne of cement are produced worldwide annually, approximately 27 billion tonnes consumption of concrete per year, as per a report published by United Nations Environment Program. Such volumes require vast amount of natural resources for aggregate and cement production.

The fine aggregate and coarse aggregates generally occupy 60% to 75% of the concrete volume and therefore, strongly influence the concrete freshly mixed and hardened properties, mixture proportions, and economy. Crushed stone and gravel are most commonly used as a coarse aggregate in concrete, while natural sand or river sand as a fine aggregate in concrete.

River sand is naturally occurring granular material composed of finely divided rock and mineral particles. River sand has the ability to replenish itself. The composition of

sand is highly variable, depending upon the local rock sources and conditions, but the most common constituent of sand is silica (silicon dioxide), usually in the form of quartz, which because of its chemical inertness and considerable hardness, is the most common mineral resistant to weathering.

Globally, between 47 to 59 billion tonnes of material mined every year, of which sand and gravel account for the largest share from 68% to 85%. The use of aggregates for concrete all over the world can be estimated at 25.9 billion to 29.6 billion tons a year for 2012 alone. This large quantity of material cannot be extracted and used without a significant impact on the environment.

Sand mining is an activity referring to the process of removal of sand from the foreshore including rivers, streams and lakes. Sand is also mined from beaches and inland dunes and dredged from large scale removal of riverbed materials and dredging below the streambed alters the channel form and shape, that, in turn, has several consequences such as erosion of the riverbed and banks, increase in channel bed slope and changes in channel morphology. Removed sand is a direct loss to the river system. It is also a threat to bridges, river banks and other nearby structures.

Physical disturbances due to human activities also lead to interruption in nesting/breeding activities. For example, in the National Chambal Sanctuary, mining of sand adversely affected ghariyals, who use sand banks for nesting and basking. They lay eggs under the sand beds, which were destroyed by sand mining related activities. The problem of environmental impacts associated with excessive sand mining has now become so serious that existence of river ecosystems is threatened in a number of locations, damage being more severe in small river catchment.

1.2 Alternatives for Natural Sand:

As the supplies of suitable natural sand near the point of consumption are becoming exhausted, the cost of this sand is increasing, which is ultimately increasing the cost of the construction. The demand of sustainable growth of infrastructure in modern times is to find an alternative material that should not only satisfy the technical specification of fine aggregate, but it should also be abundantly available. A lot of research has been done in the past to find alternate source of fine aggregate.

Use of industrial by-products in concrete has drawn a serious attention of researchers and investigators in recent years. There are many waste materials of some industries that have been successfully used as a partial as well as full replacement of natural fine aggregate. Siddique Rafat (2014) gave an overview about the utilization of waste foundry sand, coal bottom ash, cement kiln dust and wood ash as partial replacement of natural sand on fresh, mechanical and durability properties of concrete and also discussed physical, chemical and mineralogical properties of each waste product.

Recyclable industrial waste materials can be conveniently used in place of natural coarse or fine aggregates. One such source of fine aggregates is the slag, a by-product associated with foundry industries Slag being produced in large quantities; its disposal has become an environmental concern. Various industrial by-products like copper slag, electric arc furnace slag, **Cupola slag** have been utilized as an alternate to natural river sand.

1.3 Source of Cupola slag:

The cupola is used to melt pig iron and fluxes for producing grey cast iron. A secondary product is produced in this system is referred as cupola slag. 40–80 kg slag is produced per ton of cast iron manufacturing (Pribulova et al. 2019).

Cupola slag seems as a stony and amorphous material. It is a fused product that contains oxidized impurities of metals and silicon oxide. (Aderibigbe and Ojobo 1982). Cupola slag consists of Al_2O_3 , MnO , SiO_2 , MgO , TiO_2 , CaO , Fe_2O_3 , Cr_2O_3 , Na_2O_3 as mentioned by various academicians (Balaraman and Ligoria 2015).

There are 47,145 cast iron plants existing all over the world amongst them 21,532 are using cupola for melting. The amount of cast iron production is 47.795 million tons per year worldwide. There are 6000 cupola furnaces in India which produces 7 to 8 million tons of cast iron every year. 0.4–0.5 million tons cupola slag are produced each year in India (Chakravarty et al. 2021).



Fig -1.1: Lump sized of Cupola Slag aggregate



Fig -1.2: Cupola Slag Based Fine aggregate

The production of crushed fine aggregate starts with fragmentation of cupola slag lumped. The fragmented of cupola slag lumped is then crushed and screened through multiple stages. Crushing of lumped is generally carried out in multiple stages: primary crushing, secondary crushing and tertiary crushing. Fine aggregate is produced at the end of each stage and subsequently separated from coarse aggregate and dust below 75-micron portion via screening.

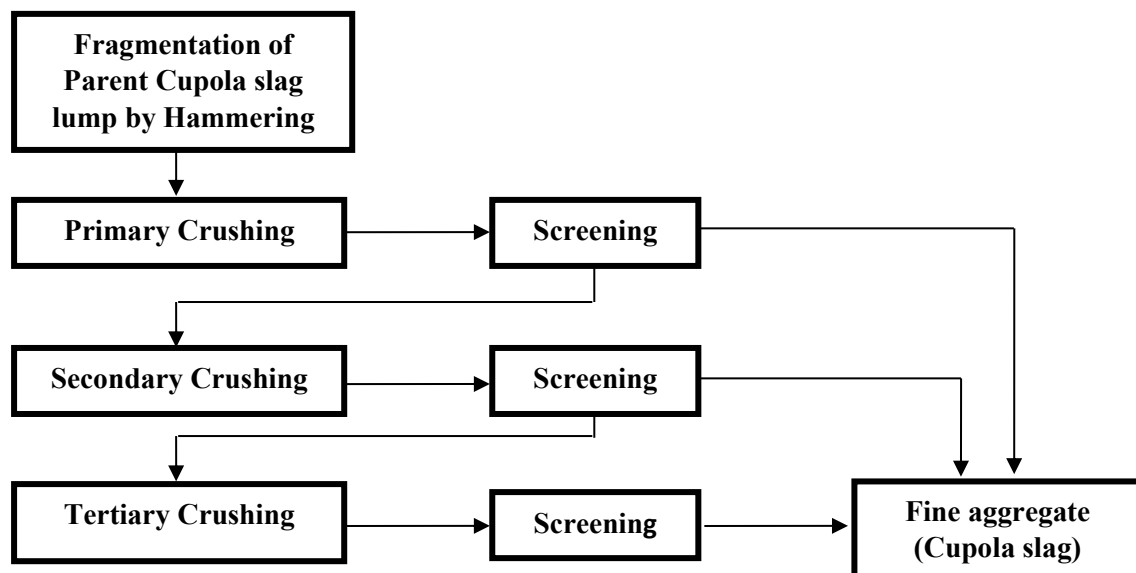


Fig. 1.3 : Schematic Diagram Showing the Production of Cupola Slag-based fine aggregate

1.4 Applications of cupola slag:

Cupola slag has a lot of applications in construction and infrastructure sector:

1. Cupola is used as a course and fine aggregate in bituminous mixes, i.e., Dense Bituminous Macadam (DBM), Bituminous Macadam (BM), Bituminous Concrete (BC), etc.
2. Cupola is used as a course and fine aggregate in concrete.
3. Cupola is used as an embankment construction, landfill capping, etc.

However, its use in concrete is not very popular. The main problem is using of cupola slag as fine aggregate that contain a large amount of dust. However, it can be used as a partial replacement of river sand in concrete so as to decrease the consumption of river sand by removing of dust. Proper design mix incorporating cupola slag as fine aggregate in concrete along with natural sand has a great potential to give concrete with identical or better properties as compared to conventional concrete.

1.5 Properties of Fine aggregate:

Cupola slag is different from natural sand in many aspects:

- i. **Mineralogy** – Natural sand is generally siliceous in nature, whereas the Cupola slag consists of Al_2O_3 , MnO , SiO_2 , MgO , TiO_2 , CaO , Fe_2O_3 , Cr_2O_3 , Na_2O_3 .
- ii. **Water absorption** – Fine aggregate from Cupola slag has lower water absorption as compared to natural sand.
- iii. **Particle Shape** – Natural sand particles are generally round in shape, while fine aggregate from cupola slag particles are angular in nature.
- iv. **Surface Texture** – Natural sand particles have smooth surface texture, while fine aggregate from cupola slag particles have rough surface texture.

1.6 Objectives of the Thesis:

The main objective of the thesis is to study the potential use of cupola slag as FA as a partial substitute of natural sand in concrete. In this study, concrete mixes are prepared with partial replacement of natural sand with FA from cupola slag at different substitution rates of 10%, 20%, 30%, 40% and 50%, of such concrete are compare with the control concrete mix containing fully natural sand. Different objectives of this study are given below:

- To compare the workability of concrete mixes incorporating FA from cupola slag as partial replacement of natural sand with control concrete mix.
- To compare the compressive strength, split tensile strength and density of concrete mixes incorporating cupola slag-based fine aggregate as partial replacement of natural sand with control concrete mix.
- To compare water absorption, sorptivity and chloride-ion permeability of concrete mixes incorporating cupola slag-based fine aggregate as partial replacement of natural sand with control concrete mix.
- To study various changes occurred in cement phases with inclusion of cupola slag-based fine aggregate as partial substitute of sand in concrete mixes by X-ray diffraction (XRD) analysis.
- To study the various changes occurred in microstructure of concrete with inclusion of cupola slag-based fine aggregate as partial substitute of sand in concrete mixes by Scanning Electron Microscope (SEM) analysis.
- To find out the optimum percent replacement of natural sand with cupola Slag based fine aggregate so as to give acceptable workability, hardened and durability properties.

1.7 Outline of the Thesis:

This thesis report includes five chapters:

Chapter 1 “Introduction” gives some quantitative data about excessive sand mining in the world and its environmental impacts, use of industrial by-products as fine aggregate in concrete, production process and properties of cupola slag dust.

Chapter 2 “Literature Review” gives an overview about the previously published

Chapter 3 “Experiential Program” gives the detail of scheme of experimentation, different raw materials used and procedures adopted for testing of raw materials, mix design, casting, curing and methodology of various tests on concrete.

Chapter 4 “Results and Discussion” gives results and properties of raw materials and mix proportioning of concrete mixes. It also includes results of workability of concrete, hardened properties of concrete, i.e. dry density, compressive strength and split tensile strength, durability properties, i.e., water absorption, sorptivity and rapid chloride-ion permeability, and microstructural analysis, i.e., XRD, SEM and EDX analysis of concrete mixes.

Chapter 5 Gives major conclusions drawn from the study.

CHAPTER 2

LITERATURE REVIEW

Joseph O. Afolayan et. al. (2013) they investigation on the Potentials of Cupola Furnace Slag in Concrete. The compressive strength of the concrete designed using cupola furnace slag and granulated cupola slag as a coarse aggregate and partial replacement for cement was investigated. A series of experimental studies were conducted involve concrete production in two stages. The first stage comprised of normal aggregate concrete (NAC) produced with normal aggregates and 100% ordinary Portland cement (OPC). Meanwhile, the second stage involved production of concrete comprising of cupola furnace slag an aggregates with 100% ordinary Portland cement (OPC) and subsequently with 2%, 4%, 6%, 8% and 10% cementitious replacement with granulated cupola furnace slag that had been grounded and milled to less than 75 μm diameter. The outcomes of compressive strength test conducted on the slag aggregate concrete with and without granulated slag cementitious replacement were satisfactory compared to normal aggregate concretes.

B.S. Thomas, A. Damare, R.C. Gupta (2014) They investigated the suitability of using copper tailings in cement concrete as a partial replacement for natural river sand. M25 grade concrete was designed according to IS:10262-2010, with water/cement ratios of 0.4, 0.45, and 0.5. Substitution of fine aggregates with copper tailings was done at levels ranging from 0% to 60%. The mix with 0% copper tailings was used as the control. Tests were conducted to determine the compressive strength, flexural strength, pull-off strength, abrasion resistance, drying shrinkage, air and water permeability, rapid chloride permeability, alkalinity, and resistance to sulfate attack in the concrete specimens. It was observed that copper tailings can be utilized for partial replacement of natural fine aggregates up to 60% with water/cement ratios of 0.4, 0.45, and 0.5. Since the copper tailing concrete (up to 60% substitution) exhibited good strength and durability characteristics, it may be recommended for all construction activities.

R.Balaraman and S. A. Ligorla (2015) they investigation on partially replaced fine aggregate and coarse aggregate by Cupola Slag. The design mix for M20 and M25 grade concretes were arrived and the target strength is found to be 26.960 N/mm² and 30.51 N/mm² respectively. Cupola slag is used in concrete as partial replacements for fine and coarse aggregates (5%, 10%, 15%, 20%, 25%, 50% and 100%) to ascertain applicability in concrete. The maximum compressive strength attained was 33.778 N/mm² and 38.222 N/mm² at 15% for both M20 and M25 grades of concrete respectively at 28 days. Similarly, the maximum split tensile strength attained was 3.206 N/mm² and 3.819 N/mm² for M20 and M25 grades at 15% and 5% respectively. The concrete with cupola slag as partial replacement for coarse aggregates gives less strength when compared to fine aggregates.

Ladomerský et al. (2016)) they investigation on One-year properties of concrete with partial substitution of natural aggregate by cupola foundry slag. Identical strength had been observed by partial replacement of 25.5% fine aggregates (0–4 mm) with cupola slag (0–4 mm). Fine aggregates are a very fundamental part of concrete. Fine aggregates substitution with the aid of cupola slag increases the compressive and tensile strength of concrete. Natural fine aggregate use can be reduce by reusing cupola slag as fine aggregates. Cupola slag substituted concrete found to be similar in terms of suction capability, penetration of water under pressure, elasticity module, strength and workability when compared to conventional concrete.

A.PRIBULOVÁ et.al. (2019) they investigated on Cupola Furnace Slag: Its Origin, Properties and Utilization. A cupola furnace is the most frequently used furnace aggregate for cast iron production. A by-product of the production of cast iron in cupola furnaces is cupola slag. Its amount is 40–80 kg per 1 tonne of the produced cast iron, and that is one of the reasons why this material is not as favoured as, for example, the blast-furnace slag. The purpose of this article is to provide the basic information on the formation of slag in a cupola furnace, and its chemical composition, structure and current potential applications.

The greatest potential for the use of cupola slag is in the building industry; therefore, a section of the present article deals with the property that plays an important role particularly with regard to the use of slag in the building industry, i.e. the slag hydraulicity. The achieved results indicate that the hydraulicity of the cupola slag is incomparable with the hydraulicity of the blast-furnace slag; this may be associated with the problems that arise when the slag of this type is used in the building industry. The authors used the air-cooled as well as granulated slag from cupola furnaces in the production of concrete is made from the slag alone. While the air-cooled slag may be use as a partial replacement for the blast-furnace slag in concrete mixtures, the use of granulated slag from cupola furnaces as a replacement for granulated blast-furnace slag in cement-free concrete has not proven to perform well.

At 28 days for 15% OPC replacement, which amounted to a **31.9%** increase above the compressive strength of the reference concrete. In addition, the porosity of concrete decreased as the replacement level of OPC by CFS increased. The chemical analysis of CFS also indicated that it has pozzolanic properties. The above results indicate the suitability of granulated cupola furnace slag for use in concrete for which reduced permeability is an essential performance requirement.

Waseem, N. Thakur et. al. (2021) they had partially replaced fine and aggregate coarse aggregate by Cupola Slag. Fine aggregate is replace by cupola slag at five different percentages (0%, 10%, 20%, 30% and 40%). In addition, the w/c ratios varied were 0.40, 0.45 and 0.50 at a fixed cement content of 380 kg/ m³. Across all the concrete mixes and at all curing ages, an increase in the compressive strength observed due to incorporation cupola slag **up to 30% replacement** level of fine aggregates after which a decrease in strength observed on further addition of cupola slag. This increment in compressive strength was higher at lower w/c ratios. Similar trends for split tensile strength. For all curing ages, a decrease in water penetration depth is with an increase in the percentage of

cupola replacement from 0% to 40% and this decrease was most prominent at the w/c ratio of 0.4. Correlations were developed between strength and durability properties to validate the experimentally obtained results. To examine the ‘effect’ and ‘size of effect’ of CR percentage and w/c ratio on the concrete properties, two-way analysis of variance is carried out and statistically significant results were obtained. At all curing ages, quadratic relationships is developed for establishing the dependence of concrete properties on CR and w/c ratio. The results of this investigation indicate that cupola slag is a sustainable material which can be effectively used as partial substitute to natural sand.

S. Bhat , H Sood et. al.(2022) they partially replaced fine aggregate by Cupola Slag.

In the current study an effort has been made to investigate the impact of cupola slag inclusion, on the mechanical and durability properties of concrete. Different percentages (10%, 20%, 30%, 50%, and 80%) of cupola slag were used to substitute fine aggregate and corresponding results have been observed. Also, different mechanical and durability tests were carried out to check the suitability of replacing natural fine aggregate by cupola slag. Compressive strength was found to be increased by cupola slag inclusion up to a percentage replacement of fine aggregates of 30%, after which further cupola slag addition resulted in a loss of strength. For flexural and tensile strength similar trends were observed. On the other hand, as the amount of cupola inclusion increased from 0% to 80%, a decreasing trend in both water penetration depth and water absorption was observed. Likewise, for all the aforementioned criteria a notable improvement in chloride penetration was observed due to the inclusion of cupola slag. The study basically focuses on present concern regarding industrial waste which poses a risk to the environment. Industrial waste has been utilized in a sustainable manner, as it has shown improvement in properties as compared to ordinary concrete mix. Such type of modified concrete can be employed in harsh exposure conditions as can be found from durability tests. The findings of this study show that cupola slag is a sustainable matter that works well as a partial replacement for natural sand.

R. Sikder et, al (2023) they partially replacement of coarse aggregate by cupola slag

The mix design for **M20** grade concrete ,w/c = 0.55. The coarse aggregate has been replaced with cupola slag by 0 to 50% in step of 10 weight percentage. It is observed that the workability has reduced with the increases in weight percent of copula slag. Maximum reduction of workability has been noted at the 50% replacement of coarse aggregate by cupola slag as compared to the controlled concrete sample. The criteria for M20 grade concrete are successfully attained by the samples up to 40% (by weight) replacement and with further addition of cupola slag i.e. the sample with 50% (by weight) of replacement fails to qualify as M20 grade concrete. It is observed that with increasing of cupola slag in concrete mix the compressive strength value decreases but 40% (by weight) of replacement can attain the target strength at 28 days as per mix design.

R. Sikder et, al (2024) they in this study focuses on assessing the durability, mechanical, and microstructure properties of **M20** concrete when incorporating CFS as a partial replacement for coarse aggregates. The replacement levels considered were 0%, 10%, 20%, 30%, 40%, and 50% (by weight).

The inclusion of CFS as replacement of coarse aggregates leads to a reduction in the dry density of the concrete. One of the major fact is there is a general downward trend in compressive strength with increase in the use of CFS in concrete, a 40% (by weight) partially replaced CFS concrete demonstrates the ability to achieve the target strength as per the design mix. The split tensile strength experiences a decline trend with the rising incorporation of CFS in concrete. The correlation study emphasizes the dependency of results of compressive strength, split tensile strength, and dry density in CFS-based concrete. XRD analysis identifies new compounds (Calcite, Muscovite, Al bite, and Quartz) resulting from the use of CFS in concrete, potentially achieve to target design strength up to a 40% substitution. SEM and EDS studies reveal that there is no significant increase in cement gel formation and oxide formation due to the inclusion of CFS in concrete which is up to the mark.

S. Chakravarty, et, al, (2023) They investigating the recycling and reutilization potential of industrial wastes is essential to achieving the vision of sustainable waste management. One such industrial waste is cupola slag, a by-product of grey cast iron. This slag often ends up in dump yards or landfills due to a lack of proper attention. This article aims to analyze existing literature to explore every possible avenue for reutilizing cupola slag to meet the goal of sustainable waste management. Primary emphasis is placed on the utilization of cupola slag in the building industry, where it can serve as a partial or full substitute for fine and coarse natural aggregates, as well as cement, in concrete production. The reusability of cupola slag has been assessed through extensive investigations into its microstructure, and chemical, and physical properties, starting from its origin. While limited research exists on the use of cupola slag in other sectors—such as in the production of glass ceramics, synthesis of zeolite and phosphorus-based fertilizers, manufacture of ceramic foams, road construction, and use as an artificial pozzolan—this comprehensive analysis highlights the significant potential for its reuse. By ensuring the reuse of industrial waste like cupola slag, we not only open up eco-friendly opportunities but also advance toward a sustainable future. More rigorous studies and the implementation of reuse methods for cupola slag are essential to realizing this potential.

Ashwini Patil, Harshada Kapure (2023) they compared to conventional concrete of **M30** grade, 30% replacement of fine aggregate and 0.05% GO give **57 N/mm²** compressive strength on 28 days of this newly modified concrete, which is the best result among different combinations. A split tensile strength test showed that almost 24% more tensile strength is gain by the addition of GO compared to conventional concrete. For this modified concrete, the Rapid Chloride Penetration Test (RCPT) yields good results when Cupola and 0.05% GO are used.

The pulse velocity of concrete with 30% cupola replacement without GO is slightly lower than that of other mixes. Overall, the concrete cast with good workmanship and precision. The number of voids and micro cracks seemed to be fewer, up to 10% reinforced with

graphene oxide in cupola-based concrete, according to SEM measurements, and the creation of extra **C-S-H** gel rose along with a rise in the percentage of **GO** replacement levels. Percentage increases in cupola result in a decrease in strength parameters, i.e., a combination having 40% or 50% replacement of cupola as fine aggregate results is decrease in results for this mix proportion.

Pribulova, et, al, (2017) they are investigating on Blast and cupola furnaces are used for producing pig iron and cast iron, respectively, and operate on similar principles, using coke as fuel and having comparable structures. While pig iron and cast iron differ slightly in carbon and silicon content, the slags produced by these furnaces are chemically similar. However, blast furnace slag is widely used in civil engineering, such as in road construction and cement production, whereas cupola furnace slag has limited applications. This analysis compares the properties of both slags, including cooling methods, chemical composition, and physical characteristics, to understand why their uses differ. GBFC has the highest melting temp. (1383°C) and GCFC has the lowest temp. (1242°C).

C. Arum, G.O, et. al. (2014) they investigation on the potentials of cupola slag as a partial replacement option for ordinary Portland cement (OPC) in applications requiring low permeable concrete. The chemical analysis of granulated cupola furnace slag (GCFS), its fineness, bulk density, specific gravity, and the standard consistency and setting times of binary OPC and GCFS pastes were conducted. Furthermore, concrete mixes of 0.55 water/cement ratio were produced using 1:2:4 ratio (volume basis) at 0%, 5%, 10% and 15% replacement levels of OPC by GCFS and the workability and permeability of the fresh concrete were determined. Thirty six (36) standard 150mm cubes were cast from the various concrete mixes, determine their compressive strengths. The results of the tests showed that within the OPC replacement range investigated, the compressive strength of concrete progressively increased at all curing ages as the replacement level of OPC increased and attained a maximum value of **29.8 N/mm²**.

I. Sosa, et. al. (2021) they are investing in the viability of cupola slag as an alternative eco-binder and filler in concrete and mortars. When the slag undergoes rapid cooling, verification is favoured, leaving silica in an amorphous structure, making it susceptible to reactions. Through these reactions, the slag can develop cementing properties, allowing for partial replacement of cement with this residue, leading to economic and environmental savings compared to traditional hydraulic binders. In this study, the physical and chemical properties of cupola slag and its recovery process are analyzed. Mortars incorporating traditional admixtures (fly ash and limestone filler) were manufactured, and their consistency and mechanical properties were compared with those of mortars incorporating cupola slag admixture. Additionally, mortars were produced using normalized sand with Portland cement replacements (0%, 10%, 20%, and 30% by weight) with cupola slag, and both the consistency and mechanical properties were compared at 7, 28, 60, and 90 days. The results demonstrate the suitability of cupola slag as a binder and admixture, comparable to traditional ones, with the mechanical properties tending to converge across all replacement levels around 90 days.

S.N. Chinnu, et. al. (2021), they investigating on use of artificial aggregates, comprehensive reviews on the performance of alternative aggregates in concrete are highly limited. Therefore, this review focuses on reusing waste materials from the agricultural and industrial sectors to produce alternative coarse aggregates. The effects of replacing coarse aggregates with agricultural and industrial wastes, such as furnace slag, oil palm shell, coconut shell, waste ceramic tiles, glass, expanded polystyrene, lightweight expanded clay aggregate, and recycled concrete aggregate, on the fresh, hardened, and durability properties of concrete are reported. Moreover, a critical evaluation of sintered and cold-bonded artificial aggregate production processes and their influence on concrete properties is also presented. Recycled concrete aggregates, oil palm shell, and expanded polystyrene increased workability, whereas the addition of ceramic waste and steel slag as coarse aggregates reduced the workability of concrete. The characteristics of artificial coarse aggregates depend on the binder type and binder dosage adopted in the production process, while the properties of sintered aggregates are governed by sintering temperature. The lower crushing strength of alternative coarse aggregates results in a subsequent reduction in compressive strength with an increase in the replacement level. A reduction in the density of concrete is observed when sintered and cold-bonded aggregates are used.

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1 General:

The chapter describes the details of experimental program and methodology for the evaluation. In first section, results of physical testing of cement, coarse aggregate, natural sand and cupola slag are given. For coarse aggregates, sieve analysis, bulk density, specific gravity and water absorption of both 20mm and 10mm nominal size of aggregate were determined. For fine aggregate, different tests conducted were sieve analysis, bulk density, specific gravity and water absorption. For cupola slag, along with other physical testing also done. In next section, mix design of **M25** grade of concrete is given and mix proportioning of different concrete mixes is fixed. All-in aggregate grading of combined aggregate is presented. Fresh properties (workability), hardened properties (density, compressive strength and split tensile strength), durability properties (chloride-ion permeability, sorptivity and water absorption) and mineralogical & microstructural characteristics (X-Ray diffraction, i.e., XRD and Scanning Electron Microscopic, i.e., SEM) of concrete mixes made with varying percentages of cupola slag based fine aggregate as partial replacement of natural sand. In this chapter, result of testing of constituent materials, i.e., cement, coarse aggregate, natural sand and cupola slag based fine aggregate used for making concrete.

This chapter also includes procedure adopted for mix design of concrete, details of test specimens to carry out different tests, procedure of casting as well as the test procedures adopted, age of specimen at testing, are also discussed in this chapter.

3.2 Testing of Constituent Materials:

3.2.1. Cement:

Ordinary Portland Cement of Grade 53 (OPC 53), manufactured by The Ramco Cements Limited, was used for making all concrete mixes. The cement was free from any hard lumps and uniform in colour. Chemical properties of cement are given in Table 3.1.

TABLE 3.1: Chemical composition of Ordinary Portland Cement of 53 Grade

Components	OPC 53 (Wt.%)	Limit
CaO	61.9	60 – 67%
SiO ₂	22.1	17 – 25%
Fe ₂ O ₃	3.71	0.5 – 6%
Al ₂ O ₃	4.91	3 – 8%
Na ₂ O	0.31	0.4 – 3%
K ₂ O	0.61	0.4 – 3%
SO ₃	2.21	1 – 3%
MgO	2.8	0.1 – 4%

3.2.2. Coarse Aggregate:

A combination of 20mm and 10mm nominal size aggregate is used as coarse aggregate. Both types of coarse aggregate were locally procured and conformed to Indian Standard Specifications given in IS 383:2016. Different physical properties of both types of coarse aggregate are given in Table 3.2

TABLE 3.2 : Physical Properties of Coarse Aggregate

Physical Property	Test Result	
	20mm Nominal Size Coarse Aggregate	10mm Nominal Size Coarse Aggregate
Specific Gravity	2.79	2.64
Water Absorption (%)	0.7	0.64
Bulk Density (kg/m ³)	1640	1590

Sieve analysis results of 20mm nominal size coarse aggregate and 10mm nominal size coarse aggregate are given in Table 3.3 and Table 3.4, respectively.

TABLE 3.3: Sieve Analysis of 20mm Coarse Aggregate

Sieve Size	% Finer
40	100
20	95.83
10	8.82
4.75	0.52
pan	0

TABLE 3.4: Sieve Analysis of 10mm Coarse Aggregate

Sieve Size	% Finer
10	95.82
4.75	15.86
2.36	1.86
pan	0

3.2.3. Fine Aggregate:**3.2.3.1 Natural sand**

Locally procured natural sand is use in the experimental program and it is conformed to Indian Standard Specifications given in IS 383:2016. Different physical properties of natural sand is given in Table 3.5

TABLE 3.5: Physical Properties of Natural sand

Physical Property	Natural sand
Particle Size	Finer than 4.75mm
Specific Gravity	2.66
Bulk Density	1539 kg/m ³
Water Absorption	1.05
Fineness Modulus	2.62

TABLE 3.6 : Sieve Analysis of Natural Sand

Sieve Size	% Finer
10 mm	100
4.75 mm	98.5
2.36 mm	94.5
1.18 mm	79
600 μ	47
300 μ	24
150 μ	5
pan	0

3.2.3.2 Cupola Slag based Fine Aggregate (CS)

Cupola slag was collected from Foundry in Howrah, West Bengal. Different physical properties of Cupola slag presented in Table 3.7

TABLE 3.7 : Physical Properties of cupola Slag based fine aggregate

Physical Property	cupola Slag based fine aggregate
Particle Size	> 4.75mm
Specific Gravity	2.68
Bulk Density	1542 kg/m ³
Water Absorption	1.01
Fineness Modulus	2.875

TABLE 3.8 : Chemical composition of cupola slag based fine aggregate

Chemical composition	Cupola slag (Wt.%)
Lime, CaO	10.7
Silica, SiO ₂	53.1
Alumina, Al ₂ O ₃	11.1
Iron oxide, Fe ₂ O ₃	16.1
Magnesia, MgO	1.94
Manganese, MnO	3.33
Sodium Oxide, Na ₂ O	0.16
Potassium oxide, K ₂ O	1.05

TABLE 3.9 : Sieve Analysis of cupola slag-based fine aggregate

Sieve Size	% Finer
10 mm	100
4.75 mm	100
2.36 mm	99.75
1.18 mm	75.25
600 µ	47.5
300 µ	9.5
150 µ	0.5
pan	0

Comparing the properties of cupola slag with natural sand, it can be observed that although specific gravity of cupola Slag based fine aggregate is slightly higher than that of sand. Natural Sand and cupola slag both are confirm to grading **Zone-II** specifications given in Indian Standard **IS 383:2016**.

3.2.4 All-In Aggregate Grading:

Based on the individual sieve analysis of coarse aggregate and fine aggregate, all-in aggregate grading was done as per the specifications of IS 383:2016, so as to fix their proportions during the mix design of concrete. All-In aggregate grading, as per the proportions taken in mix design, is given in Table 3.10.

TABLE 3.10 : All-In Aggregate Grading

Sieve Size	Individual % Passing			Combined Aggregate % Passing			All-In Aggregate % Passing	Limits as per IS 383:2016
				CA= 62%		FA (38%)		
				20mm (80 %)	10mm (20 %)			
	20mm	10mm	Sand					
40 mm	100	100	100	49.6	12.4	38	100	100
20 mm	95.83	100	100	47.532	12.40	38	97.93	95 to100
4.75 mm	0.515	15.9	98.5	0.255	1.967	37.43	39.65	30 to 50
600 μ	-	-	47.0	-	-	17.86	17.86	10 to 35
150 μ	-	-	5.0	-	-	1.9	1.90	0 to 6

3.3 Mix Proportioning of Concrete:

3.3.1 Mix Design of Concrete:

Mix design of concrete used in the experimental program is as per Indian Standard Specifications given in IS 10262:2016. Grade of the concrete selected **M25**.

Based upon the quantities of different ingredients in control concrete, mix proportions of concrete mixes with FA from cupola slag were calculated. Concrete mixes with replacement of natural sand with 10%, 20%, 30%, 40% and 50% (by weight) FA from cupola slag were designated as D10, D20, D30, D40 and D50 respectively.

TABLE 3.11: Mix Proportion of Concrete Mixes with Aggregate in SSD Condition

Mix Designation	W/C ratio	Cement (kg/m ³)	Water Content (kg/m ³)	Coarse Aggregate (kg/m ³)		Natural Sand (kg/m ³)	(FA) Cupola slag (kg/m ³)
				20mm	10mm		
CM	0.5	383	192	938	234	687	0
D10	0.5	383	192	938	234	618	69
D20	0.5	383	192	938	234	550	137
D30	0.5	383	192	938	234	481	206
D40	0.5	383	192	938	234	412	275
D50	0.5	383	192	938	234	344	344

3.4 Mix Proportioning of Concrete Ingredients:

All-In aggregate grading for 20mm nominal size aggregate as the requirements given in IS 383:2016 was used to fix the proportion of aggregate in concrete. Trial and error method was used to fix the proportion of 20mm nominal size aggregate, 10mm nominal size aggregate and natural sand in concrete based on their individual gradations. Based upon this all-in aggregate grading, percentage of 20mm nominal size aggregate, 10mm nominal size aggregate and natural sand was fixed, which was to be used in the calculation of quantities of coarse aggregate and fine aggregate at the time of mix design.

Mix design of concrete was done as per IS 10262:2016. Grade of control concrete was chosen as **M25** and target slump was **75mm**. Conditions for exposure were taken as moderate. As per the procedure of IS 10262:2016, first of all, target strength was calculated assuming suitable value of standard deviation. Estimated water content was calculated for the desired workability and free w/c ratio was chosen 0.5. From estimated water content and free w/c ratio, cement content was calculated. Based on the volume of aggregate in concrete, quantity of coarse as well as fine aggregate was calculated as per their specific gravity and proportion fixed as per all-in aggregate grading. Coarse aggregate and fine aggregate quantities calculated were based upon the SSD (Saturated Surface Dry) condition. Thus, necessary water corrections must be applied based upon the moisture content of coarse aggregate and fine aggregate at the time of casting.

3.5 Mixing of Ingredients and Casting of Samples:

3.5.1 Mixing of Ingredients:

Laboratory drum mixer was used for the preparation and mixing of all concrete mixtures. A drum mixer is a mechanical device, which uses a revolving drum to combine cement, coarse aggregate, fine aggregate and water to form a homogenous mass. Both, coarse aggregate as well as fine aggregate, were in dry conditions. So, necessary water corrections were applied before the mixing operation. All the ingredients, i.e., cement, coarse aggregate, fine aggregate and water, were weighted with an accuracy of 1.0 gram. Drum mixer was started and firstly, coarse aggregate and fine aggregate were dry mixed thoroughly. After that, cement was added in the drum mixer and it was rotated till a uniform mass was obtained. In the end, water was added very carefully, so as to prevent any loss of water during the mixing operations. The drum mixer was rotated till we got a concrete mass

with uniform colour and consistency. Care was taken during the whole operation so as to ensure the proper mixing of all ingredients. Workability of all concrete mixtures was checked immediately after the finishing of mixing operation.

3.5.2 Sample Preparation:

All the concrete specimens were casted in steel moulds. All the moulds were cleaned and oiled properly before the mixing of concrete ingredients. They were properly tightened to correct dimensions before casting operations. Care was taken to ensure that there must not be any gap left so as to prevent the leakage of slurry. Concrete specimens were compacted in two layers using vibrating table. After the casting operations, concrete specimens were left in the casting room for approximately 24 hours, after which they were de-moulded and placed in the curing tank. Use cube size 100mm x 100mm x 100 mm and Cylinder size 100 dia and 200 height due to shortage of cupola slag. The detail of the specimens casted to perform various tests is given below:

1. Compressive Strength: Cubical specimens of dimensions 100mm×100mm×100mm were casted for testing of compressive strength of concrete.
2. Split Tensile Strength: Cylindrical specimens of diameter 100mm and height 200mm were casted for testing of split tensile strength of concrete.
3. Water Absorption: Cubical specimens 100mm×100mm×100mm were casted for testing of water absorption of concrete.
4. Sorptivity: Cylindrical specimens of diameter 100mm and height 30mm were casted for testing of sorptivity of concrete
5. Rapid Chloride-Ion Permeability: Cylindrical specimens of diameter 100mm and height 50mm were casted for testing of rapid chloride-ion permeability of concrete.

TABLE 3.12 : Number of cube/Cylinder required for testing

Mix Designation		Number of cube/Cylinder				
		Compressive strength test (Cube)	Split tensile test (Cylinder)	RCPT (Cylinder)	Sorptivity test (Cube)	Water Absorption test (Cube)
DM	7 days	3	3	-	-	-
	28 days	3	3	3	3	3
D10	7 days	3	3	-	-	-
	28 days	3	3	3	3	3
D20	7 days	3	3	-	-	-
	28 days	3	3	3	3	3
D30	7 days	3	3	-	-	-
	28 days	3	3	3	3	3
D40	7 days	3	3	-	-	-
	28 days	3	3	3	3	3
D50	7 days	3	3	-	-	-
	28 days	3	3	3	3	3

3.6 Test Procedures:

3.6.1 Workability:

Workability of concrete is the ease with which concrete can be properly mixed, transported, compacted and finished, with minimum loss in homogeneity. Slump test is the most extensively used test to measure workability of concrete all around the world in construction sector. Workability of the concrete was evaluated by slump test as per Indian Standard Specifications given in **IS 1199:2018**. A mould in the form of frustum of a cone with bottom diameter 200mm, top diameter 100mm and height 300mm was filled with three approximately equal layers, tempering each layer with a standard tempering rod with 25 strokes.

**Fig 3.1 : Slump Test of Concrete**

After filling and levelling the surface, mould was removed by lifting it in vertical direction, allowing concrete to subside. Results of the workability testing were reported as slump in mm, which is the difference between height of the mould and that of highest point of subsided concrete mass.

3.6.2 Compressive Strength:

Compressive strength is regarded as the most important property of hardened concrete. Compressive strength test was done as per Indian Standard Specifications, according to the procedure given in IS 516:1959. Compressive strength of concrete was evaluated at age of 7 days and 28 days using standard cube specimens of 100mm×100mm×100mm. Concrete specimen were demoulded 24 hours after the casting and placed in the curing tank to ensure sufficient curing.



Fig 3.2 : Compressive Strength Test on Compression Testing Machine

The compressive strength was calculated according to the following formula:

$$\sigma = P/A$$

where,

σ = Compressive Strength (N/m²)

P = Maximum load sustained by the cube (N)

A = Area of cross section of cube (mm²)

Results of the compressive strength testing were reported as average of compressive strength of 3 specimens at 7 days, and 28 days for each concrete mix in N/mm²..

3.6.3 Density of Concrete:

Density of concrete is an important aspect, as it plays a major role in the calculation of dead weight of a structure. At the time of de moulding of cubical specimens of 100mm×100mm×100mm used for testing of compressive strength, mass of 3 random cubes was taken using a weighting balance of 10 kg capacity with an accuracy of 1.0g and density of concrete was calculated from the following formula:

$$\rho = M / V$$

where,

ρ = Density of concrete in kg/m³

M = Mass of 100mm×100mm×100mm cube in kg

V = Volume of cube in m³

3.6.4 Split Tensile Strength:

As concrete is strong in compression, but very weak in tension, so it is necessary to determine the tensile strength of the concrete so as to prevent cracking in tension zones. Split tensile strength is an indirect method to determine tensile strength of concrete. Split tensile strength test was done as per Indian Standard Specifications, according to the procedure given in IS 5816. Split tensile strength of concrete was evaluated at age of 7 days, and 28 days using standard cylindrical specimens of 100mm diameter and 200mm height. For the evaluation of split tensile strength, each specimen is place centrally between the bearing plates of CTM with suitable packing strips at top and bottom to ensure proper distribution of load as shown in **Fig. 3.4**. Load is apply continuously and uniformly at specified loading rate of 1.2 N/mm²/min to 2.4 N/mm²/min. The split tensile strength is calculate according to the following formula:

$$\sigma_{st} = 2P/\pi DL$$

Results of the split tensile strength testing were reported as average of split tensile of 3 specimens at 7 days, and 28 days for each concrete mix in N/mm².



Fig. 3.3 : Split Tensile Strength Test of Concrete

3.6.5 Water Absorption:

Pore structure of concrete plays a very important role to have an idea about the durability aspects of concrete. Water absorption of concrete is an indicator of how dense the microstructure of concrete is. Water absorption of concrete was evaluated at various specified ages as per the procedure given in ASTM C 642-13.

Water absorption test was performed at 28 days, after initial curing of 28 days on cubical specimens of 100mm×100mm×100mm. Oven dry mass and saturated mass of the concrete specimens were determined as per the standard procedures given in ASTM C 642-13.

Water absorption of concrete was calculated using the following formula:

$$\text{Absorption after Immersion (\%)} = [(B - A)/A] \times 100$$

where,

B = Oven Dried mass of specimen in air (g)

A = Mass of surface-dry specimen after immersion in air (g)

3.6.6 Sorptivity:

Movement of liquids through interconnecting pores plays a very important role to determine the durability of concrete. Sorptivity of concrete is rate of absorption of water by one dimensional capillary action. Sorptivity of concrete was evaluated as per the procedure given in ASTM C 1585 – 04. Rate of absorption of water test as performed at 28 days, after initial curing of 28 days on standard cylindrical specimens of 100mm diameter and 50mm height. Each specimen was prepared as per the procedure give in ASTM C 1585 - 04. Sides of the specimen were sealed with epoxy coating and adhesive tape was wrapped over the outer curved surface. Mass of each specimen was taken and it was recorded as initial mass. The schematic diagram of the experimental procedure is given in Fig. 3.5. As soon as the specimen was placed in water, a stop watch had been started and mass of the specimen was taken after 1, 5, 10, 20, 30, 60, 120, 180, 240 and 360 minutes. At each specified time slot, specimen was lifted and its surface in contact with water was surface dried with the help of a towel and its mass was recorded.

For calculation of Sorptivity of concrete specimen, first of all, rate of absorption of water, I , was calculated using change in mass of the specimen divided by the product of the cross-sectional area of the test specimen and density of water. For this purpose, density of water is adopted as 0.001 g/mm^3 and the unit of I comes out to be mm^3/mm^2 or mm .

Sorptivity (S) is a material property which characterizes the tendency of a porous material to absorb and transmit water by capillarity. The cumulative water absorption (per unit area of the inflow surface) increases as the square root of elapsed time (t)

$$I = S\sqrt{t} \text{ Therefore } S = I/\sqrt{t}$$

Where S = sorptivity in $\text{mm}/\sqrt{\text{sec}}$.

t = elapsed time in seconds.

$I = \Delta W/Ad = \text{Absorption in mm.}$

$\Delta W = \text{change in weight} = W_2 - W_1$

$W_1 = \text{oven dry weight of specimen in grams.}$

$W_2 = \text{weight of specimen after different time intervals like 60 seconds, 5, 10, 20, 30 and 60 minutes etc.in grams.}$

$A = \text{surface area of the specimen through which water penetrated in mm}^2.$

$d = \text{density of water in gm/mm}^3 \text{ater (g/mm}^3)$

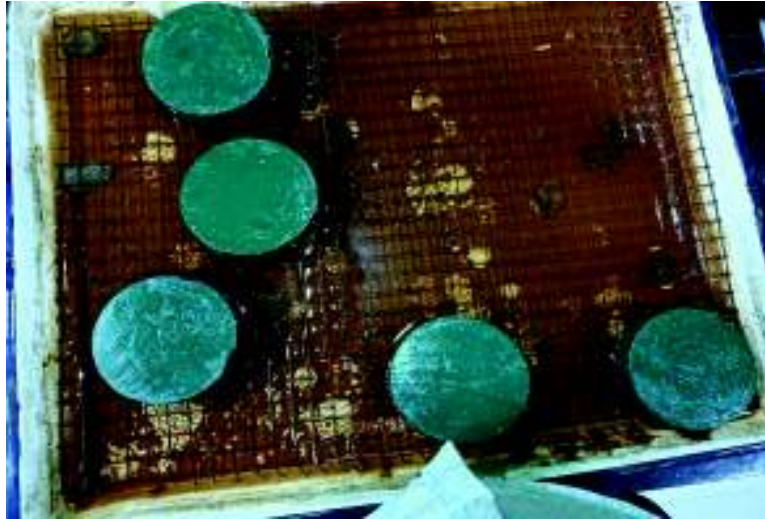


Fig. 3.4 : Sorptivity Test of Concrete Samples

3.6.7 Rapid Chloride-ion Permeability Test:

Rapid chloride-ion permeability test is a very fast method to determine durability of concrete. In this test, durability of concrete specimen is determined in terms of their electrical conductance. Rapid chloride-ion permeability test was evaluated as per the procedure given in ASTM C 1202 – 97. Rapid chloride-ion permeability test was performed at 28 days on standard cylindrical specimens of 100mm diameter and 50mm height. Each specimen was prepared as per the procedure give in ASTM C 1202 – 97. Each specimen was placed in between two solutions in a standard set up, with 3% NaCl solution on one side and 0.3N NaOH solution on other side and a potential difference of 60V is applied between the two terminals. The test set up is shown in Figure 3.6. Results of rapid chloride-ion permeability test were reported as average charge passed specimens at 28 days in Coulombs.



Fig. 3.5: Test setup of rapid chloride penetration test

TABLE 3.13: Chloride-ion Permeability Based on Charge Passed (ASTM 1202)

Charge Passed (Coulombs)	Chloride-ion Permeability
> 4000	High
2000 – 4000	Moderate
1000- 2000	Low
100 – 1000	Very Low
< 100	Negligible

3.6.8 Microstructural Analysis

Microstructure analysis of concrete involves examining the internal structure and composition of concrete at a microscopic level to understand how its properties and performance are influenced by its composition and curing conditions. This analysis is crucial for assessing the quality, durability, and overall behaviour of concrete.

3.6.8.1 Method for Preparation of Specimen

Preparation of specimens has long been recognized as an art in optical microscopy, particularly in metallurgy and mineralogy. Ideally, the specimens prepared are representative of the structure of interest and are free from damage and contamination. Fig. 3.7 illustrates the general procedure for preparing the specimens.

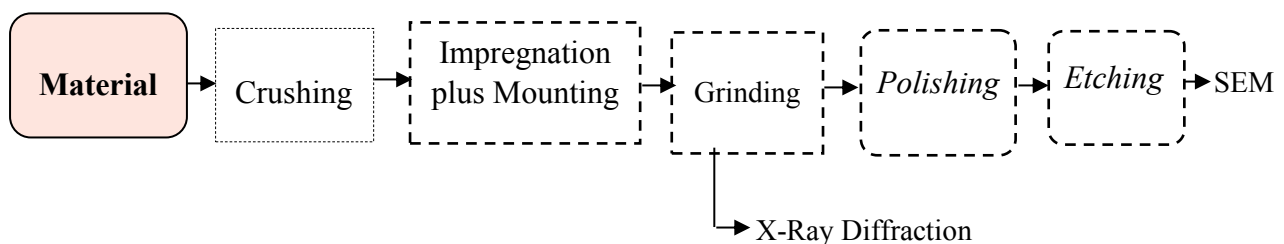


Fig. 3.6 : Method for preparation of specimen for SEM and XRD

The first step is the preparation of a fresh surface which will positively comprise the characteristics of interest. Once the preferred plane has been nominated, a section of the material constrained on one side by this plane is detached using hacksaw or automatic abrasive cut-off machine.

The following step in the specimen preparation procedure is impregnation and mounting. This step is presented in a dotted box in Figure 3.7 to designate that it may not be essential for every sample. If a material is porous, it normally needs some form of impregnation to lessen pickup of grinding and polishing abrasives which end in scratching and disproportionate relief. In this procedure, an organic polymer is embedded into the porosity of the specimen by vacuum impregnation. This process may be augmented by succeeding over pressuring to additional force the polymer into the specimen.

The next step in the preparation of the sample, as shown in Figure I, is grinding. The sample of each grinding step is to take away the broken surface layer causing from the instantaneously earlier step.

The subsequent step in the specimen preparation system is that of polishing; this is usually done by mechanical means.

However, chemical or electrolytic polishing either independently or in grouping with mechanical polishing is finding increased application.

The last step in this preparation system is etching. The various conventional methods existing are chemical, electrolytic and thermal etching. The details of XRD and SEM analysis are described below.

3.6.8.2 Techniques of Microstructure Analysis

Techniques commonly used for microstructure analysis include:

1.0 X-ray Diffraction (XRD)

2.0 Scanning Electron Microscopy (SEM)

3.0 Energy Dispersive X-ray (EDX)

1.0 X-ray Diffraction (XRD):

The X-ray diffraction (XRD) method comprises of a suitable method to find the mineralogical investigation of crystalline solids. If a crystal-like mineral is brought in contact to X-rays of a specific wavelength then covers of atoms deflect the rays and create a array of peaks, which is representation of the mineral. The horizontal scale (diffraction angle) of a usual XRD pattern provides the crystal lattice spacing, and the vertical scale (peak height) provides the intensity of the diffracted ray.

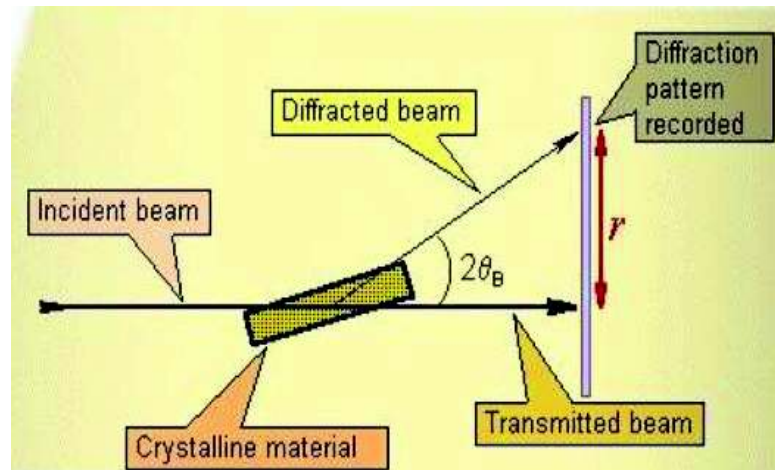


Fig. 3.7: Schematic Diagram of X-Ray Diffraction

When the sample being X-rayed comprises of more than single mineral, the concentration of specific peaks from the specific minerals are proportionate to their amount. The XRD analysis is normally carried out at room temperature by means of powder X-Ray diffraction with filtered 0.154 nm Cu, K α radiation. Samples are scanned in a continuous mode from 10 θ -80 θ with a scanning rate of 2 θ per minute.

The X-Ray powder diffraction provides the graph between the X – Ray light intensity, which is distribute on the sample and angle variance of the deflected X – Rays.

The XRD method can be adopted to recognize the single crystals, and to make known the single crystal structure. It can be also adopted to recognize various crystals which are available in a mix, e.g. minerals in a stone. For minerals having inconstant formulations and structures (clays), XRD is the best technique for recognising the formulations and finding their percentage in a sample.

2.0 Scanning Electron Microscope (SEM):

In this current study, the hydrated cement paste obtained from the seven powder samples are subjected to SEM analysis. The range of scale used in SEM analysis was 5 μ m with the resolution of x 5000. The detailed process of sample preparations for SEM analysis is described below.

After Compressive testing was finished, the cube samples are crushed and the hydrated cement was collected from the innermost core of the concrete cube sample. The collected samples are sieved through 300 μ sieve.

The sample preparation was done by cone and quartering method for reducing the sample size. The sample was dispensed on flat surface so that it takes on a conical shape. The top

of the conical shape was flattened. The cone is divided into quarters. Two opposite quarters was discarded; the other two are combined. The process was repeated until the suitable sample size was reached the sample preparation process for micro structural analysis is pictured below Fig.3.8



Fig.3.8 Cone and Quartering Method for SEM Sample Preparation

A scanning electron microscope is that kind of electron microscope which prepares images of a sample by scanning the top surface with a focused beam of electrons.

A scanning electron microscope (SEM) scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition.

This is influential method, mostly when the microscope is fitted with a microprobe analyser. It comprises techniques similar to X-ray fluorescence to identify the chemical composition of hydrates. The higher resolution of the SEM allows the microstructure of the hydrated cement paste in concrete or mortar to be identified and studied. But, attention must be implemented when understanding the images as sample preparation and the vacuum needed by most of the microscopes can produce specifications, which are not available in the wet paste.

Working of SEM:

- The SEM uses electrons instead of light to form an image.
- A beam of electrons is produced at the top of the microscope by heating of a metallic filament.
- The electron beam follows a vertical path through the column of the microscope. It makes its way through electromagnetic lenses which focus and direct the beam down towards the sample.
- Once it hits the sample, other electrons (backscattered or secondary) are expelled from the sample.
- Detectors collect the secondary or backscattered electrons, and convert them to a signal that is sent to a viewing screen similar to the one in an ordinary television, producing an image.

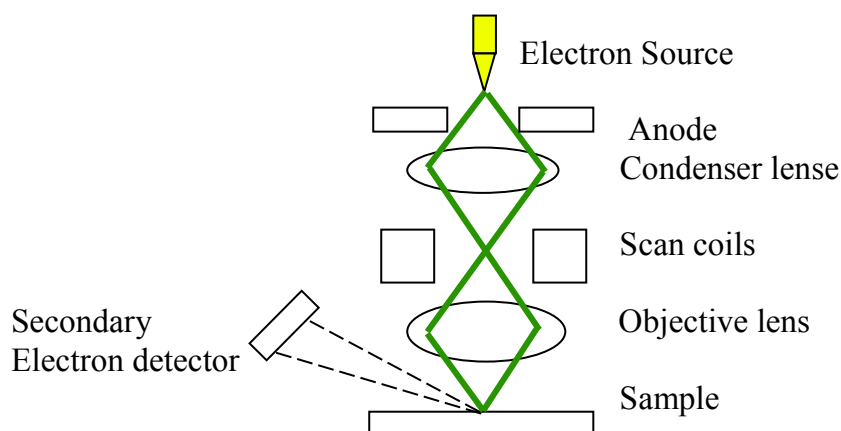


Fig. 3.9: Schematic Representation of SEM components

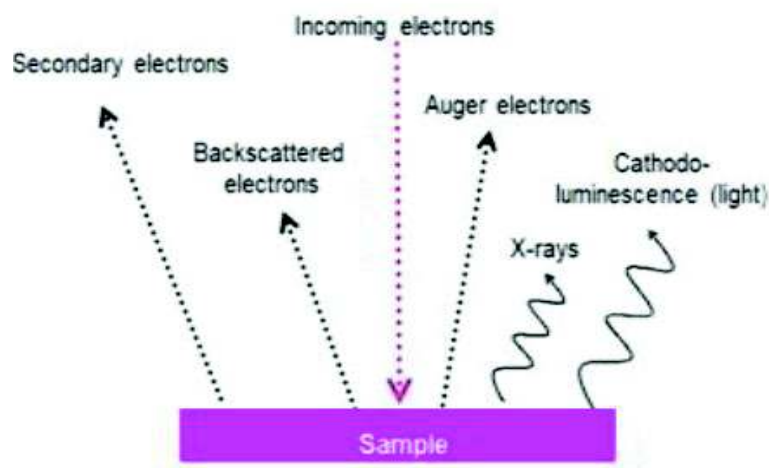


Fig. 3.10: Working of Electrons

The advantages of a scanning electron microscope include its wide-array of applications, the detailed three-dimensional and topographical imaging and the versatile information garnered from different detectors. SEMs are also easy to operate with the proper training

and advances in computer technology and associated software make operation user-friendly.

This instrument works fast, often completing SEI, BSE and EDS analyses in less than five minutes. In addition, the technological advances in modern SEMs allow for the generation of data in digital form. Although all samples must be prepared before placed in the vacuum chamber, most SEM samples require minimal preparation actions.

3.0 Energy-dispersive X-ray spectroscopy

In order to study elemental composition and distribution (mapping) in nanomaterials, energy-dispersive X-ray spectroscopy (EDS) is commonly used. The EDS detector is incorporated in both SEM and TEM systems and the measurements are carried out at high energy. The material surface is bombarded by high-energy electrons, where an electron will be knocked out from the inner shell and will thus leave a vacancy. When the high energy level is transferred to lower levels, an X-ray photon is released as shown in Fig. 3.11A and detected by the EDS detector. The emitted X-rays can be allocated according to each element present in the sample and represented in a form of a spectrum or mapping. Fig. 3.11C and D shows a typical EDS spectrum and mapping of ZnO doped with Au. EDS is a qualitative technique for elemental composition; for quantitative analysis, X-ray photoelectron spectroscopy (XPS) should be used.

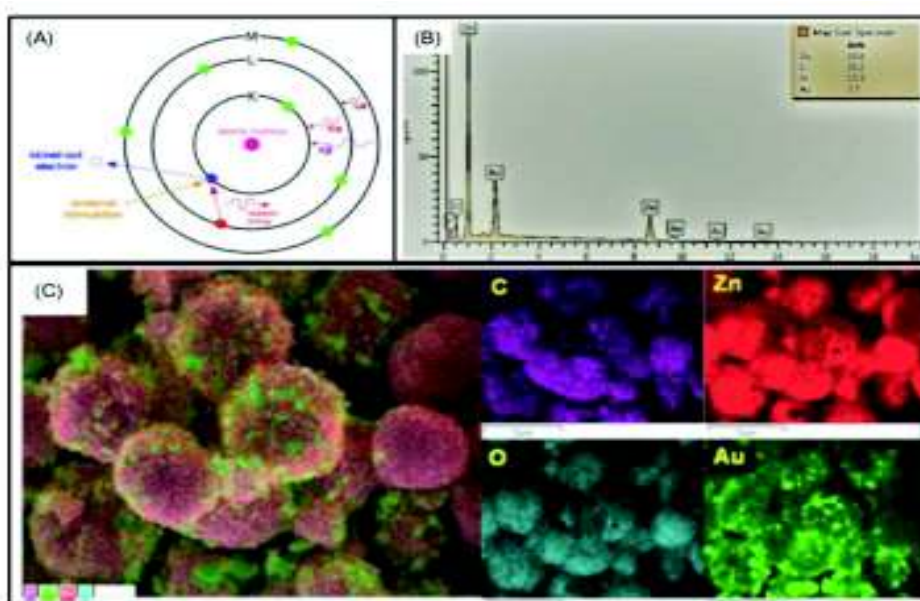


Fig. 3.11: Typical EDS spectrum and mapping

CHAPTER 4

RESULTS AND DISCUSSION

4.1 General:

In this chapter, the results of various experimental investigations on the use of cupola slag as fine aggregate are reported and critically discussed. Natural sand was partially replaced by cupola slag-based fine aggregate at 10%, 20%, 30%, 40%, and 50% (by weight). Various tests were conducted to evaluate the effects of these replacements on workability, density, compressive strength, split tensile strength, water absorption, sorptivity, and chloride-ion permeability of normal grade concrete of M25. X-ray diffraction (XRD), Scanning electron microscope (SEM) and X-ray spectroscopy (EDS) analyses were also performed to study the mineralogical and microstructural changes caused by the with cupola slag-based fine aggregate in the concrete matrix.

4.2 Comparison of natural sand and Cupola slag based fine aggregate

The properties of natural sand and cupola slag-based fine aggregate are presented in Tables 4.1 and 4.2. Fig 4.1 shows the particle size distribution of both the two different fine aggregates. Both the fine aggregates (natural sand and cupola based fine aggregate) are in zone – II as per IS:383- 2016. It is also noted that natural sand is an inert material and the cupola slag based fine aggregate is reactive material as it contains Cao and reactive SiO₂.

Table. 4.1: Physical Properties of Natural Sand and Fine Aggregate from Cupola slag

Physical Property	Natural Sand	FA from CS
Particle Size	> 4.75mm	> 4.75mm
Specific Gravity	2.66	2.68
Bulk Density (kg/m ³)	1539	1542
Water Absorption (%)	1.05	1.01
Fineness Modulus	2.62	2.875
Grain Shape	Generally rounded to sub-angular, providing good workability in concrete	Generally angular and rough, which can improve the mechanical interlocking in concrete.

Table. 4.2: Chemical Properties of Natural Sand and Fine Aggregate from Cupola slag

	Natural Sand	Fine Aggregate from Cupola slag
Chemical Property	Generally siliceous in nature (Chemically inert)	Cupola slag consists of Al_2O_3 , MnO , SiO_2 , MgO , TiO_2 , CaO , Fe_2O_3 , Cr_2O_3 , Na_2O_3 . (Ref. Table 3.8)

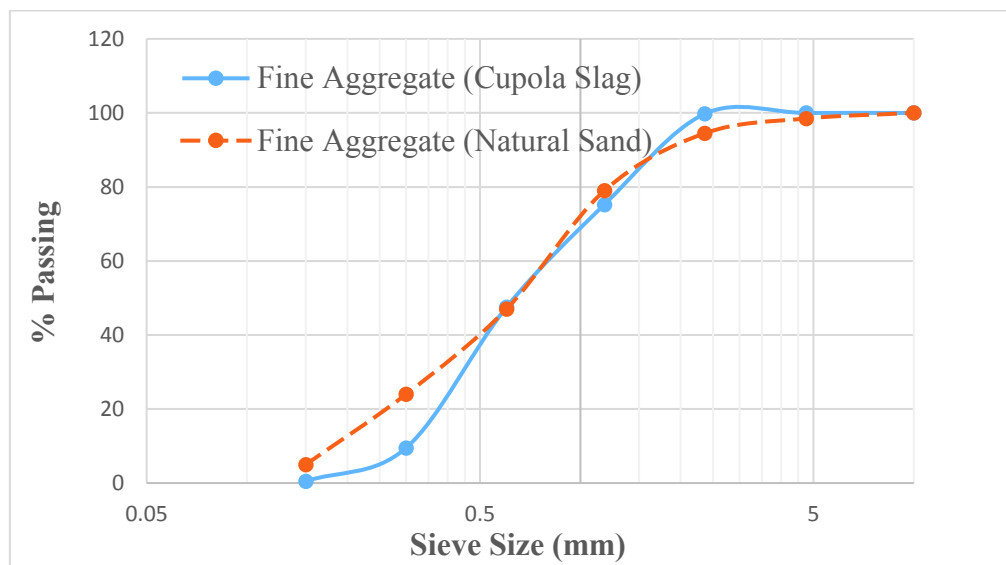


Fig. 4.1: Comparison of Particle Size Distribution of Natural Sand and Cupola Slag-based fine aggregate

4.3 Fresh Properties of Concrete

4.3.1 Workability:

The workability of all the six concrete mixes with varying cupola based fine aggregate replacing the natural sand was evaluated as slump in mm are presented in Table 4.3.

TABLE 4.3 : Slump Test Results of Concrete Mixes (M25)

Mix Designation	% replacement of natural sand by Cupola slag based Fine aggregate	Slump(mm)
CM	0 %	70
D10	10 %	67
D20	20 %	65
D30	30%	63
D40	40 %	60
D50	50%	55

It is noted that with the replacement of cupola slag-based fine aggregate as a partial substitute for natural sand, the workability of concrete mixes decreases. This decrement is more pronounced at higher replacement levels of 40% and 50% (by weight). The decrease in workability is mainly due to the difference in shape between natural sand and cupola slag-based fine aggregate particles. Moreover, the fine aggregate from cupola slag has angular particles with a rough surface texture, whereas natural sand generally has rounded particles with a smooth surface texture. Therefore, to maintain the same slump, the water content may be increased slightly or chemical admixture may be employed.

4.4 Hardened Properties of Concrete:

4.4.1 Dry Density of Concrete:

The dry density of all the concrete mixes, based on the weight of 100mm×100mm×100mm cubes after 28 days of curing, was determined after drying in an oven at 110°C for 24 hours. The results of concrete density with increasing substitution rates of natural sand with cupola slag-based fine aggregate are presented in Table 4.4 and Fig 4.2.

TABLE 4.4 : Dry Density Results of Concrete Mixes

Mix Designation	Weight(kg/m ³)
CM	2580
D10	2585
D20	2588
D30	2590
D40	2593
D50	2595

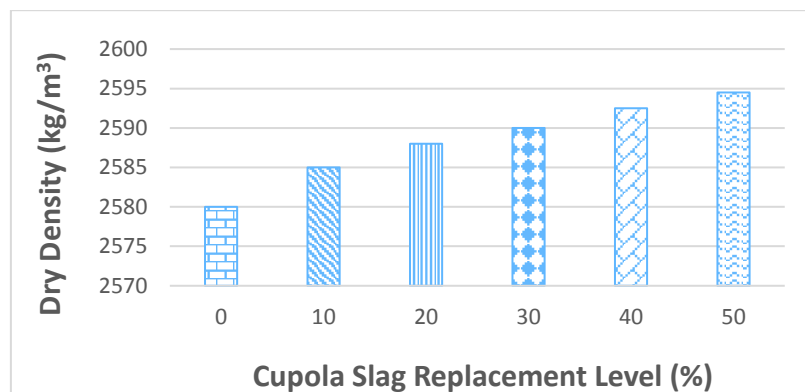


Fig. 4.2 : Effect of Replacing Natural Sand with Fine Aggregate from Cupola Slag on the 28-Day dry Density of Concrete

It is noted that with the addition of cupola slag based fine aggregate as a partial substitute of natural sand, the dry density of the concrete is slightly enhanced. This enhancement is mainly due to higher specific gravity of cupola slag based fine aggregate than that of natural sand.

4.4.2 Compressive Strength:

The compressive strength of all the six concrete mixes was evaluated at 7 days and 28 days to study the effect of partially replaces natural sand with cupola slag-based fine aggregate. The results are presented in Table 4.5 and Fig. 4.3.

TABLE 4.5 : Compressive Strength Test Results of Concrete Mixes

Mix Designation	Compressive Strength (MPa) at			
	7 days		28 days	
	Strength	% increase	Strength	% increase
CM	21.9 ± 1.3	-	31.2 ± 0.7	-
D10	22.8 ± 1.6	3.8	32.9 ± 1.4	5.42
D20	23.4 ± 1.2	6.9	33.1 ± 1.73	6.32
D30	22.9 ± 0.81	4.2	34.3 ± 0.75	10.1
D40	23.1 ± 1.2	5.3	34.4 ± 2.8	10.4
D50	17.6 ± 0.86	-19.5	29.5 ± 1.12	-5.3

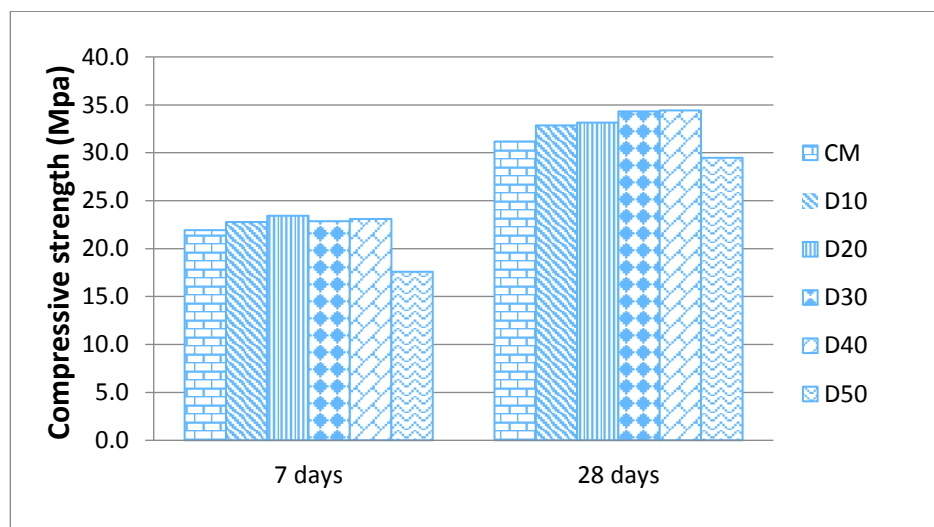


Fig. 4.3 : Effect of Replacement of Natural Sand with Cupola Slag based FA on Compressive Strength of Concrete

It is noted that the inclusion of cupola slag-based fine aggregate as a replacement for natural sand leads to a slight enhancement in the compressive strength of concrete at all ages compared to the control mix up to 40% replacement. At 7 days, the compressive strength of the control mix (with Natural sand only) was **21.9** MPa, while the compressive strength

of D10, D20, D30, and D40 concrete mixes increased by 3.8%, 6.9%, 4.2%, and 5.3%, respectively. However, at 50% replacement with cupola slag-based fine aggregate, the strength decreased by 19.5% with respect to control mix. Similarly, at 28 days, the compressive strength of the control mix was **31.2 MPa**, whereas the compressive strength of D10, D20, D30, and D40 concrete mixes increased by 5.42%, 6.32%, 10.1%, and 10.4%, respectively. At 50% replacement with cupola slag-based fine aggregate, the strength decreased by 5.3% with respect to control mix. Therefore, based on the present study, it is suggested to limit the use of cupola slag-based fine aggregate up to 40% replacement. More details study is also needed to specify the limit of replacement for other grades concrete. The improvement of strength of concrete within 40% replacement seem to be better bond between the cupola based fine aggregate and cement paste due to the refinement of the interfacial transition zone with new component as per microstructure analysis (discussed later). The rough surface texture of cupola slag fine particles may have also contributed to a better bonding between the fine aggregate particles and cement paste.

4.4.3 Split Tensile Strength:

Table 4.6 and Fig. 4.4 show the results of the split tensile strength of different concrete mixes with/without cupola based fine aggregate replacement at 7 and 28 days.

TABLE 4.6 : Split Tensile Strength Test Results of Concrete Mixes

Mix Designation	Split Tensile Strength (MPa)			
	7 days		28 days	
	Strength	% increase	Strength	% increase
CM	1.98 ± 0.02	-	2.67 ± 0.15	-
D10	2.12 ± 0.03	7.1	2.72 ± 0.09	1.9
D20	2.16 ± 0.04	9.1	2.77 ± 0.15	3.8
D30	2.19 ± 0.04	10.6	2.80 ± 0.08	4.9
D40	2.28 ± 0.09	15.2	2.81 ± 0.02	5.2
D50	2.06 ± 0.15	4.0	2.54 ± 0.22	-5.9

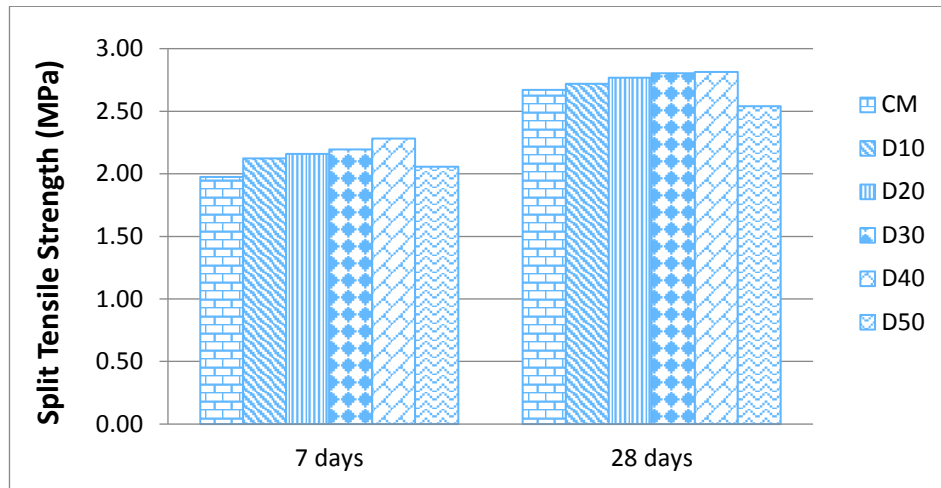


Fig. 4.4 : Effect of Replacement of Natural Sand with FA from Cupola slag on Split Tensile Strength of Concrete

It can be observed that the inclusion of cupola slag-based fine aggregate as a replacement for natural sand leads to a slight enhancement in the split tensile strength of concrete at all ages compared to the control mix, except for mix D50. At 7 days, the split tensile strength of the control mix was 1.98 MPa, while the split tensile strength of D10, D20, D30, D40, and D50 concrete mixes increased by 7.1%, 9.1%, 10.6%, 15.2%, and 4.0%, respectively. Similarly, at 28 days, the split tensile strength of the control mix was 2.67 MPa, whereas the split tensile strength of D10, D20, D30, and D40 concrete mixes increased by 1.9%, 3.8%, 4.9%, and 5.2%, respectively. However, at 50% replacement with cupola slag-based fine aggregate, the strength decreased by 5.9%. This indicates that the split tensile strength of concrete increases with the level of natural sand replacement, reaching a maximum at 40% replacement before decreasing. The increase in split tensile strength, except for D50, follows a similar pattern similar to that observed in compressive strength.

4.5 Durability Properties of Concrete:

4.5.1 Water Absorption:

The water absorption (%) of the six concrete mixes, with or without cupola slag-based fine aggregate, was evaluated at the age of 28 days and are presented in Table 4.7 and Fig. 4.5.

TABLE 4.7 : Water Absorption of Concrete Mixes at D28 days Ages

Mix Designation	Water Absorption (%)
	At 28 Days
CM	3.4
D10	2.3
D20	2.4
D30	2.3
D40	2.2
D50	1.7

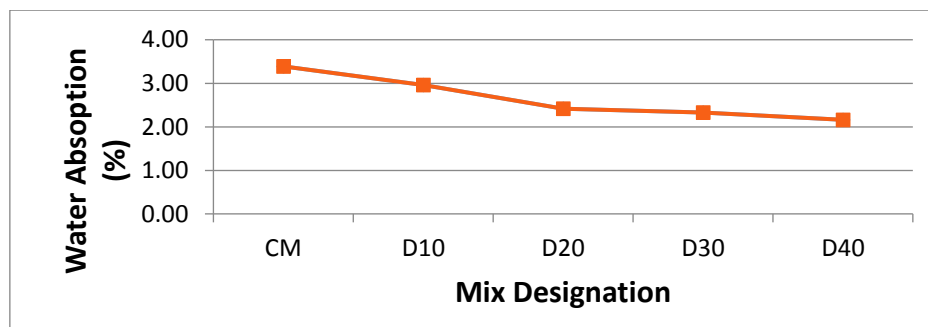


Fig. 4.5 : Effect of Replacement of Cupola slag-based fine aggregate on Water Absorption of Concrete

It is noted that the partial substitution of natural sand with cupola slag-based fine aggregate has a significant effect on the water absorption of concrete. The water absorption of the concrete mixtures decreases as the sand replacement level increases. The water absorption of the control concrete mix was noted as 3.4%, while the water absorption of concrete mixes with 10%, 20%, 30%, 40%, and 50% sand replacement was 3.0%, 2.4%, 2.3%, 2.2%, and 1.7%, respectively. This lower water absorption may be due to fewer voids present in the concrete with cupola based fine aggregate, resulting in a denser microstructure.

4.5.2 Sorptivity:

Fig. 4.6 shows the water absorption with time different concrete mixes after initial curing period of 28 days to study the effect of partially substituting natural sand with cupola slag-based fine aggregate. Further, the sorptivity of the above concrete mixes were calculated and presented in Fig 4.7.

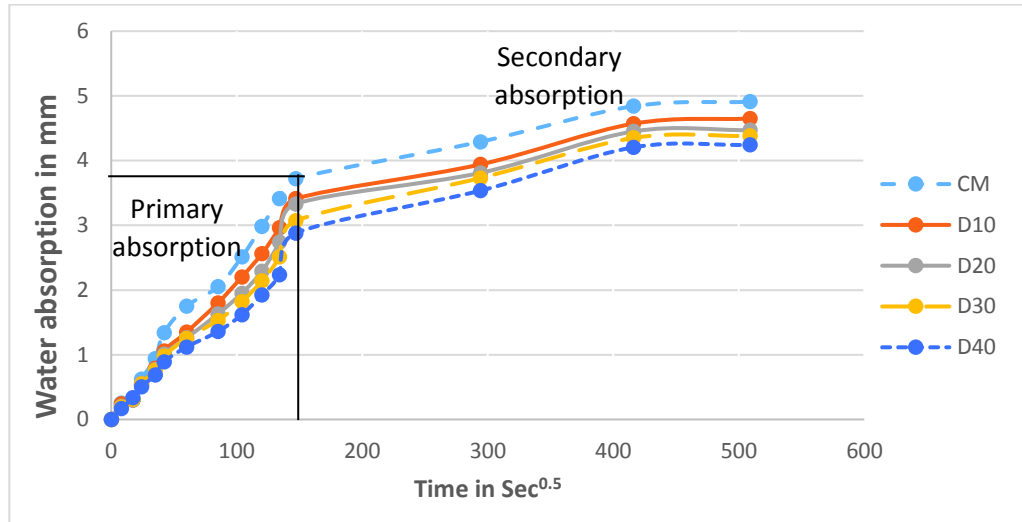


Fig. 4.6 Water absorption rate of control and others mixes

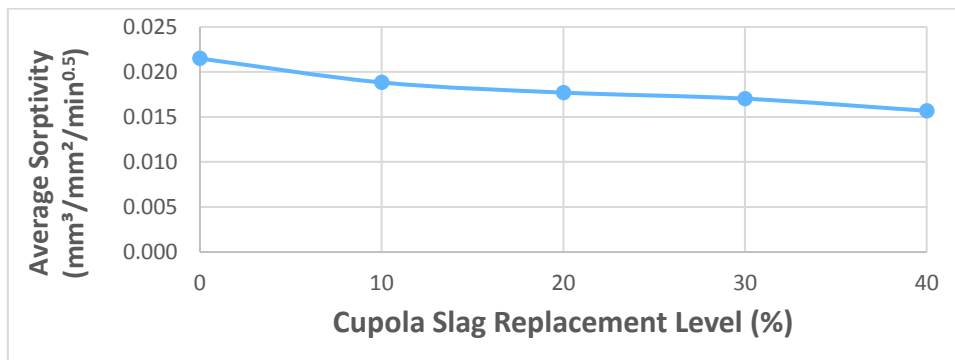


Fig. 4.7 : Effect of Replacement of Cupola slag on sorptivity of Concrete (Primary absorption)

It is noted that the replacement of natural sand with cupola slag-based fine aggregate has a significant effect on the sorptivity of concrete. Moreover, the sorptivity of concrete mixtures decreases as the sand replacement level increases. At 28 days, the average sorptivity of the control concrete (in $\text{mm}^3/\text{mm}^2/\text{min}^{0.5}$) was 0.021, whereas the sorptivity of concrete mixes with 10%, 20%, 30%, and 40% sand replacement was 0.019, 0.018, 0.017, and 0.016, respectively. This observation is consistent with the findings for water

absorption rate with time. It seems that the cupola slag-based fine aggregate not only reduces the size of the voids but also modifies the internal capillary pore structure by blocking interconnecting capillary pores.

4.5.3 Rapid Chloride-ion Permeability:

A rapid chloride-ion permeability test was conducted, and the charge passed in coulombs was recorded for all concrete mixes at the age of 28 days to study the effect of partially substituting natural sand with cupola slag-based fine aggregate as shown in Fig. 4.8 and Table 4.8.

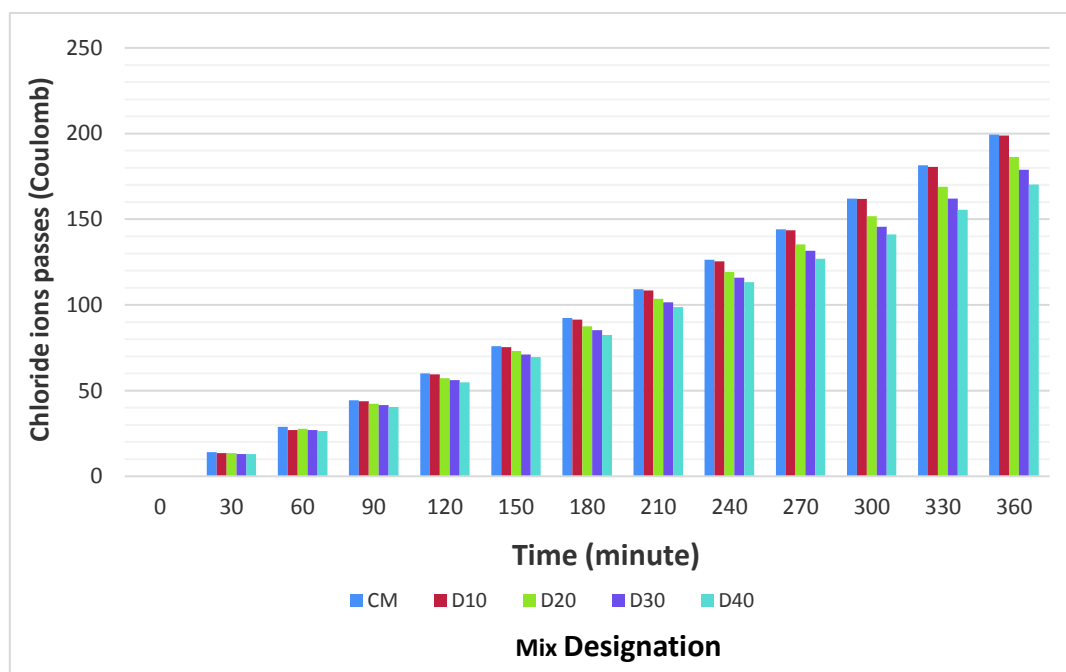


Fig. 4.8 : Rapid Chloride-ion Permeability Test Results for Concrete Mixes at 28 Days

TABLE 4.8 RCPT result for different mixes

Mix Designation	Charge Passed at 28 days (Coulombs)	Chloride-Ion Permeability as per ASTM C-1202
CM	2049	Moderate
D10	2034	Moderate
D20	1932	Low
D30	1872	Low
D40	1814	Low

It is noted that with the increase in the replacements of cupola slag based fine aggregate the chloride ion permeability decreases. This decrement in permeability can be attributed to the increase in density and compressive strength of concrete with the partial substitution of natural sand with cupola slag based fine aggregate. As explained earlier, with a higher sand substitution rate, cupola slag-based fine aggregate may tend to fill the voids in the concrete, resulting in a denser matrix compared to the control concrete. It is noteworthy that the concrete mix with 40% cupola slag-based fine aggregate replacement has the lowest chloride-ion permeability, which corresponds to the mix with the highest compressive strength at 28 days.

4.6 Mineralogical Characteristics and Microstructural Analysis:

4.6.1 X-ray Diffraction Analysis

X-ray diffraction analysis was performed to identify various cement phases in concrete and to detect any qualitative changes in these phases due to the partial substitution of natural sand with cupola slag-based fine aggregate. The X-ray diffraction pattern was recorded using an X-ray diffractometer at a diffraction angle (2θ) ranging from 10° to 80° with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$) in steps of $2\theta = 0.013^\circ$. The X-ray diffractogram of the control mix and mix D40 at 28 days are presented in Fig. 4.9. Since the maximum strength was obtained for the D40 mix, XRD analysis focused on mix D40 and control mix only.

It can be observed that at 28 days of age, the control concrete mix (CM) contained usual phases as in the normal concrete such as quartz, calcium silicate, portlandite, calcium silicate hydrate, calcium aluminium silicate hydrate and calcite. In the D40 concrete mix sample, the presence of some other characteristic peaks of new compounds along with the usual phase as in the normal concrete was also noted.

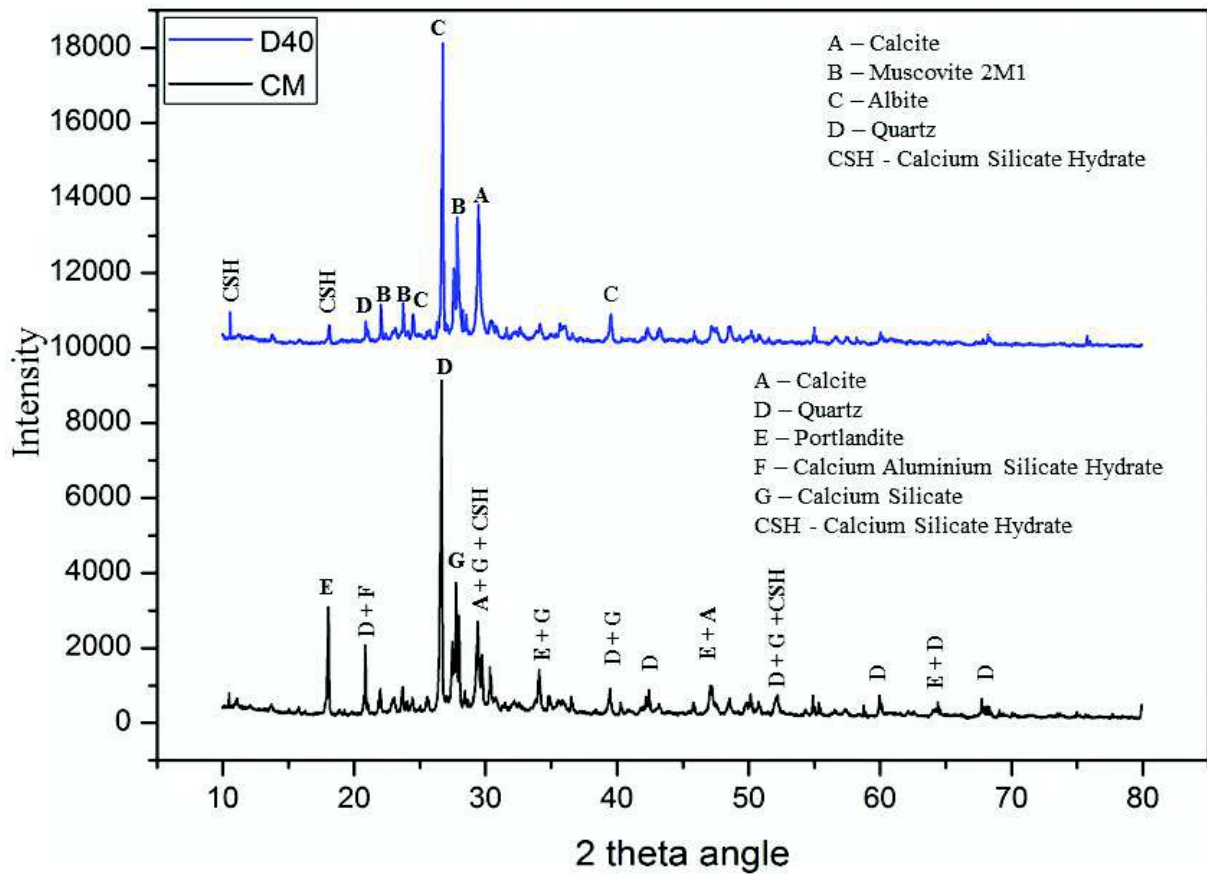


Fig. 4.9: XRD Result of Control Concrete (CM) and Concrete Mix D40 at 28 Days

These new compounds like Calcite (CaCO_3 , COD-9009668), Muscovite 2M1 ($\text{Al}_3\text{H}_2\text{KO}_{12}\text{Si}_3$, COD-1011049), Albite ($\text{AlNaO}_8\text{Si}_3$, COD-9002200) and Quartz (O_2Si , COD-5000035) were formed due to the incorporation of cupola slag in the concrete mix. The lattice structures of these new compounds are hexagonal (Calcite), monoclinic (Muscovite 2M1), triclinic (Albite), and hexagonal (Quartz). The significant peaks at 2θ for the new compounds appeared at the following angles: Calcite (29.443° , 36.0638° , 39.5141° , 43.1976° , 47.5571° , and 48.5362°), Muscovite 2M1 (22.0295° , 23.7314° , and 27.8344°), Albite (24.4774° , 26.7387° , 29.443° , 34.1289° , 36.0638° , 39.5141° , 47.5571° , and 50.2147°), and Quartz (20.9105° , 51.5203° , 60.0294° , and 68.2122°). X-ray diffraction analysis further indicates that cupola slag has a reactive material, contributing to these enhancements. It is also noted that cupola slag has limited CaO content (10.7%) and substantial SiO_2 content (53.1%). These two components have a positive effect in the improvement as reported in the microstructure analysis.

4.6.2 Scanning Electron Microscope (SEM) Analysis:

Fig. 4.10 show the micrograph of SEM analysis of control mix and D40 mix at the same magnification scale.

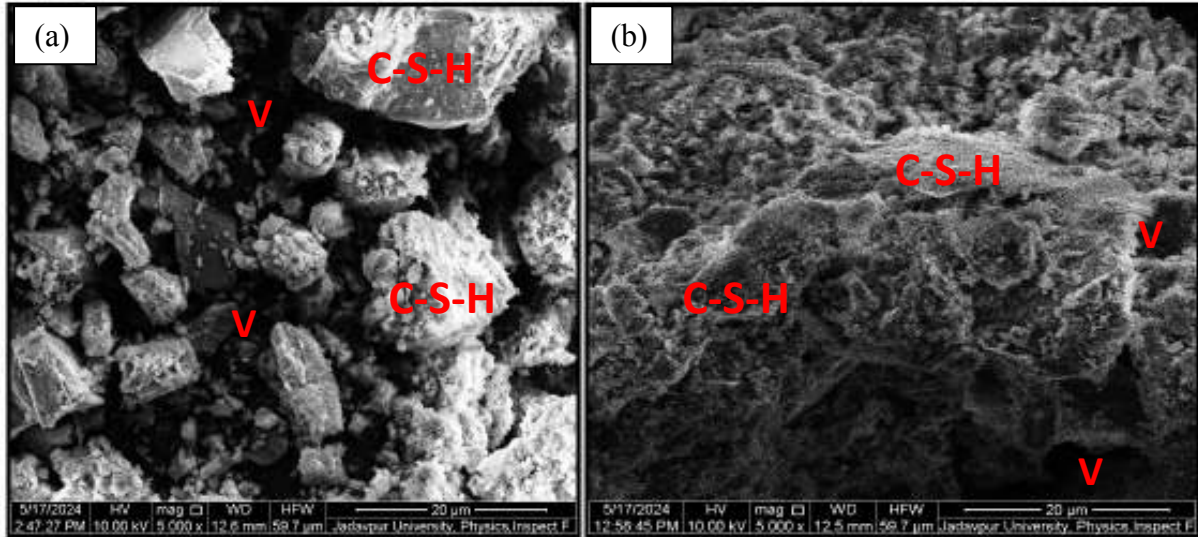


Fig. 4.10 SEM images of (a) control mix and (b) cupola slag (40%) based fine aggregate concrete mixes at 28 days. (Magnification- 5000:1).

It reveals that the inclusion of cupola slag-based fine aggregate has a profound effect on the microstructure of concrete. The control concrete exhibits more voids compared to the concrete mixes with a 40% replacement of natural sand by cupola slag-based fine aggregate. This improvement in the concrete's microstructure can be attributed to the filling effect of the cupola slag-based fine aggregate, which reduces voids and makes the microstructure denser. In conclusion, the improvement in strength and durability properties of concrete, with the addition of cupola slag-based fine aggregate as a partial substitute for natural sand, can be attributed to the denser microstructure of these concrete mixes.

4.6.3 Energy Dispersive X-ray (EDX) Analysis:

The results of EDX analysis of the control mix (CM) and the mix with 40% replacement of natural sand with cupola slag (D40) are shown in Fig. 4.11.

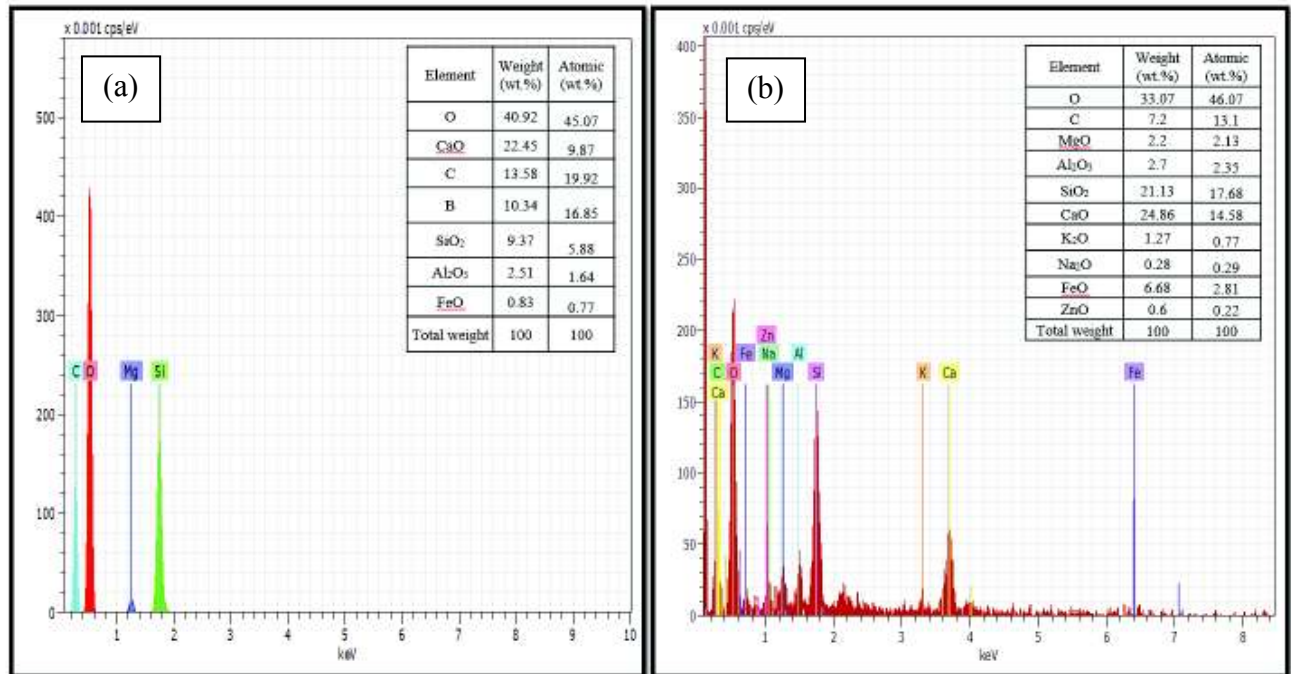


Fig. 4.11 EDX images of (a) control mix and (b) cupola slag (40%) based fine aggregate concrete mixes at 28 days.

It is noted that iron and silicon levels increased in D40 mix compared to the control concrete mix. Iron and silicon, in particular, play critical roles in enhancing the properties of concrete. Iron contributes to the strength, while silicon is essential in forming calcium silicate hydrate (C-S-H), which is the primary binder in concrete.

CHAPTER 5

CONCLUSIONS

5.1 General

The present experimental investigation was conducted to evaluate the suitability of cupola slag-based fine aggregate as a partial replacement for natural sand in concrete. The workability, compressive strength, split tensile strength, water absorption, sorptivity, and rapid chloride-ion permeability of concrete were tested by replacing natural sand with cupola slag-based fine aggregate at varying percentages. XRD, SEM, and EDX analyses were also performed on the control mix (CM) and on concrete mixes with 40% replacement of natural sand by cupola slag-based fine aggregate (D40) to study changes in cement phases and the microstructure of the concrete. Based on the limited experimental study the following conclusion can be made:

- I. **Workability:** The workability of concrete decreases as the percentage of natural sand replaced by cupola slag-based fine aggregate increases. This is due to the rough surface texture of the cupola slag-based fine aggregate.
- II. **Dry Density:** The inclusion of cupola slag as a replacement for fine aggregate results in an increase in the dry density of concrete. This is because the specific gravity of cupola slag-based fine aggregate is higher than that of natural sand.
- III. **Compressive and Split Tensile Strength:** The compressive and split tensile strengths of concrete increase as the percentage of cupola slag-based fine aggregate increases, up to a 40% replacement level. Beyond 40%, the compressive strength begins to decrease.
- IV. **Water Absorption and Sorptivity:** Water absorption and sorptivity decrease with the increased use of cupola slag-based fine aggregate. The mix with 40% sand replacement shows the lowest water absorption and sorptivity, due to the slag's filling effect, which reduces voids and blocks capillary pores in the concrete.

- V. Chloride-Ion Penetration Resistance: The resistance to chloride-ion penetration improves as cupola slag-based fine aggregate replaces natural sand. The 40% replacement mix exhibits the lowest charge passed, indicating the highest resistance to chloride-ion penetration.
- VI. Microstructure Analysis: XRD analysis identified new compounds such as calcite, muscovite, albite, and quartz in cupola based concrete mix (D40) . SEM analysis revealed that the control mix had the more void content, which decreased with increasing levels of cupola slag replacement. The 40% replacement mix had the less voids and the densest microstructure. EDX analysis showed increased levels of iron and silicon, contributing to improved strength through the formation of calcium silicate hydrate (C-S-H).
- VII. Sustainability and Environmental Impact: The use of cupola slag as a fine aggregate promotes sustainable resource use and reduces the environmental impact by minimizing the demand for natural aggregates. Additionally, reusing cupola slag provides economic benefits by reducing the consumption of natural aggregates.

5.2 Suggestion for Future Scope of Research

The future scope of research on the use of cupola slag-based fine aggregate in concrete may be focussed on the following areas:

- i. Long-Term Durability Studies: The long-term durability of concrete made with cupola slag-based fine aggregate, particularly in aggressive environments to be an important area of research for practical use. This may include resistance to chemical attacks, freeze-thaw cycles, and the rate of carbonation.
- ii. Thermal and Acoustic Properties: The thermal insulation and acoustic properties of concrete containing cupola slag needs attention particularly for use in building construction where these properties are crucial.
- iii. More study is needed using different other type of cupola slag from other source to make a guideline for practical use.

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