

Annex 19

Interim report for the research project on
Study of durability properties of composite cement (a type of hydraulic cement) currently being manufactured by the cement industry in India (CED 0237)

Submitted to

**Sectional Committee for Cement and Concrete (CED 02)
Bureau of Indian Standards (BIS), New Delhi**

by

Principal Investigator (PI) : **Dr. Jayachandran K.**
Assistant Professor, Department of Civil Engineering
National Institute of Technology Calicut (NITC), Kerala

CO-PI's : **Dr. Sharan Kumar Goudar**
Assistant Professor, Department of Civil Engineering
National Institute of Technology Calicut (NITC), Kerala

Dr. Praveen Nagarajan
Professor, Department of Civil Engineering
National Institute of Technology Calicut (NITC), Kerala

November 18, 2024

TABLE OF CONTENTS

1	Introduction to Composite Cement.....	4
2	Literature Review	4
2.1	Status of cement requirement in India.....	4
2.2	Need for Composite cement (CC) in India.....	4
2.3	Environmental benefits of composite cement	5
2.4	List of Composite Cement manufacturers in India	5
2.5	Issues with the raw materials required for the composite cement.....	5
2.6	Influence of using the fly ash and slag in the concrete	5
2.7	Comparison of performance of fly ash and slag in the fresh and harden concrete	6
3	Comparison of international standards for composite cement	6
4	Fresh properties of the blended cement concrete	7
4.1	Initial Setting Time of the blended cement	8
4.2	Final Setting Time of the blended cement	8
5	Harden properties of the blended cement concrete.....	9
5.1	Compressive strength of concrete with ternary blending of varying SCMs content (fly ash and slag).....	10
5.2	Drying shrinkage behaviour of the blended cement concrete	12
6	Review on the durability properties of the blended cement concrete.....	12
6.1	Influence of SCMs on the Water penetration depth.....	12
6.2	Influence of SCMs on the Rapid Chloride Penetration Test.....	13
6.3	Review on carbonation data	14
7	Conclusion of the literature review on Composite Cement	14
8	Aim and Significance of the Project.....	14
9	Objectives of the study and final deliverables as per the project description.....	16
9.1	Scope of the study	16
9.2	Methodology as per the project description	16
10	Road Map	18
11	Plan of work, methods, and techniques to be used.	18

11.1	Determination of chemical and physical properties according to Indian standards..	18
11.1.1	XRF test	19
11.1.2	XRD tests	19
11.1.3	TGA tests	19
11.1.4	Particle size analysis	19
11.2	Mechanical and durability tests on various concrete mixes	20
11.2.1	Workability and Compressive strength tests.....	20
11.2.2	Accelerated carbonation test.	20
11.2.3	Rapid Chloride Penetration Test (RCPT)	21
11.2.4	Rapid Chloride Migration Test (RCMT)	22
11.3	Evaluation of corrosion in concrete beams	23
11.3.1	Study on corrosion induced by carbonation.....	23
12	Deliverables	25
13	References.....	26

1 Introduction to Composite Cement

Cement industry plays a major role in the economic development of a nation, provides the vital raw material for the basic building blocks of nation's infrastructure and housing development. India is the world's second largest cement producer with an installed capacity of 390 million tonnes per annum. By 2025, the total cement production could reach 500-650 million tonnes.

The Bureau of Indian Standards previously had approved only blends of either fly ash based cement (Portland Pozzolana Cement-PPC) or slag-based cement (Portland Slag Cement-PSC). Portland Composite Cement (PCC) is a blend of both fly ash and slag together with clinker. BIS has released the standards (IS: 16415-2015) for composite cement also. Production of composite cement would be helpful in enhancing the sustainability of cement industry. Various lab trails conducted for composite cement in the country shows fruitful result for composite cement. Composite cement production in India will help in utilizing waste materials such as fly ash and slag together in one cement when both are available in sufficient quantity at the same place.

2 Literature Review

2.1 Status of cement requirement in India

Cement production and consumption is directly linked to economic development. The availability of cement is vital for infrastructure expansion, modern housing and urbanization. India has a low per-capita cement consumption at about 190 kg as of 2015 against the world average of 350 kg. The cement demand is projected to grow 2.5 to 2.7 times the current volume, reaching 500-650 million tonnes per annum (mtpa) by 2025, mainly on account of rising infrastructure and housing needs propelled by rapid urbanisation. Meeting the rising demand for cement will require considerable capacity addition along with a sharp rise in available resources, which could present many challenges. (CII, 2016)

Cement production is also an energy intensive process and one of the major sources of CO₂ emissions. The manufacture of cements with additives has been pursued as an alternative to reduce CO₂ emissions. The environment-friendly blended cement is more cost-effective to produce, as it requires lesser input of clinker and energy. The availability of fly-ash (from thermal power plants), blast furnace slag (from steel plants) and use of advance technology has increased the production of blended cement in the recent years.

As per the standards, in the composite cement, clinker can be substituted up to 65% by additives. The Composite cement has considerably lower cost of production as well as lower CO₂ intensity. (CII, 2016)

2.2 Need for Composite cement (CC) in India

For 1 million tonne cement plant producing OPC, PPC, and PSC requires nearly 1.5-1.6, 1.0-1.1 and 0.8-0.9 million tonnes of lime stone respectively, whereas production of same quantity of composite cement requires 0.6-0.7 million tonnes thus resulting in reduction of limestone consumption. CO₂ emission intensity reduces by 56% for Composite cement production as compare to OPC, and for PPC and PSC cement it reduces by 27% and 39% respectively. (CII, 2016)

2.3 Environmental benefits of composite cement

Based on the study by various cement manufacturing companies, the production of Composite Cement in a 1 MTPA cement plant, requires 57% less raw material, 52% less thermal energy and 34% less electrical energy in comparison to Ordinary Portland Cement (OPC) production. The CO₂ emission intensity of Composite cement is 0.36 tCO₂/ton of cement which is 56% lower than OPC. (CII, 2016).

2.4 List of Composite Cement manufacturers in India

The following are the some of the composite cement manufacturers on India, listed is based on the reports from the various association relevant to the cement sectors:

- Ambuja Cement - Compocem
- Anjani Cement - Anjani Super Gold Cement
- Chettinad Cement Composite
- Dalmia Cement - PCC Cement
- JK Lakshmi – Composite Cement
- Maha Cement - Maha Solid Concrete Special Cement
- Maha Cement - Maha Solid HD+ Cement
- Maha Cement – Portland Composite Cement
- MP Birla - Durga Hitech Cement - Composite Cement
- Shree Cement- Bangur Cement- Composite Cement
- Greencem - Composite Cement Additive - GB – 50 (not a cement)

The above list may not be complete, as some of the cement manufacturer have listed the cement brand /model name of their choice and not as composite cement, in the websites and the front side of the cement bags given in the online trade links. Further, the compliance statement for each of the cement is not clearly mentioned in their websites.

2.5 Issues with the raw materials required for the composite cement

The chemical composition of fly ash or slag is determined by the raw materials used and the conditions under which they are processed. These factors can vary, not only from one plant to another, but also within the same plant. It is natural to expect large variations in chemical composition between different batches of fly ash and slag. It is, however, generally accepted that the pozzolanic and cementitious properties of these materials are governed less by the chemistry and more by mineralogy and particle size. (Hegde S B, 2014)

2.6 Influence of using the fly ash and slag in the concrete

The use of flay ash or slag by replacing or reducing the cement have been executed in the field and showcased many positive influences on the fresh and hardened state of the concrete as shown in the Table 4. In India, numerous trials have been carried out on composite cement wherein different combination of fly ash and granulated blast furnace slag was used. The study of these trials indicates the physical properties of composite cement prepared using 40-60% clinker and 20-30% each of fly

Interim Report - Study on durability properties of composite cement currently being manufactured by the cement industry in India
ash and granulated blast furnace slag (GBFS) were in the range specified for Composite cement by Bureau of Indian standard (IS 16415:2015) provided lower early days strength development.

2.7 Comparison of performance of fly ash and slag in the fresh and harden concrete

Table 1 Influence of Pozzolans on concrete performance

State of concrete	Variables	Grounded Granulated Blast Furnace Slag (GGBS) addition in concrete	Fly ash (FA) addition in concrete
Fresh Concrete	Setting Time	Slight retardation	Slight retardation
	Cohesiveness		Ultra fine FA increases cohesiveness
	Workability	Slight improvement with some aggregates	Improves: lower water requirement for given slump
Hardened Concrete	Rate of early-age strength	Reduces especially at lower temperature	Slight reduction, especially at lower temperature
	Response to steam curing	Improves	Improves
	Strength gain after 28 days	Improves	Improves
	Rate of heat generation	Reduces	Reduces
	Pore structure	Improves	Improves
	Density of aggregate paste interfacial zones	Improvement	Improvement, especially with ultra fine FA
	Impermeability of concrete	Improves	Improves
	Sulphate resistance		Improves
	Rate of chloride diffusion	Reduces: improves protection of embedded steel against corrosion	Reduces: improves protection of embedded steel against corrosion
	Alkali aggregate reaction	Prevents or retard if content is sufficient	Prevents or retard if content is sufficient

3 Comparison of international standards for composite cement

The fly ash-slag-based composite cement is a relatively newly standardized type of cement and its specifications have only been formulated by the Bureau of Indian Standards in 2015. However, there are also a few international standards for blended or composite cement. The European standard EN 197-1-2011 (BDS EN 197-1: 2003 n.d.) categorises composite cement as CEM V/A and CEM V/B comprising blast furnace slag, pozzolans and/or siliceous fly ash in different proportions. The latest version, EN 197-5-2021 (EN 197-5: 2021 2021) also specifies a type of composite cement CEM VI which contains limestone apart from blast furnace slag, pozzolans and/or siliceous fly ash. ASTM C595/C595M-14 (ASTM C595/C595M-14 2014)

prescribes ternary blended cement which is produced by intimate blending of Portland cement clinker or Portland cement with 1) two different pozzolans, 2) slag and a pozzolan, 3) a pozzolan and a limestone, or 4) a slag and a limestone. The Australian standard AS 3972-2010 (AS 3972-2010 n.d.) specifies requirements of blended cement comprising Portland cement and fly ash or slag. Canadian standards CSA A3001-03-2004 (CSA A3001-03-2004 n.d.) and CAN/CSA-A362-98 (CAN/CSA-A362-98 n.d.) deal with blended hydraulic cement which contains Portland cement with up to three supplementary cementing materials like fly ash, silica fume etc. Similar specifications are published by the American Association of State Highway and Transportation Officials (AASHTO) such as M240 for blended cement (AASHTO M240 n.d.). Table 2 shows the permissible limits for partial replacement of cement clinker with other substituent materials according to the relevant Indian and International Standards.

Apart from the quantity of materials used in the manufacture of composite cement, the chemical composition and physical properties of the materials are also variable according to different standards followed by different countries and hence lack uniformity. Therefore, the optimum content of clinker in composite cement manufacture needs to be studied to achieve the desired strength and durability properties.

Table 2. Permissible limits of ingredients in composite cement from different standards

Standards	Cement type	Permissible limits (% by weight)			
		Portland cement clinker	Pozzolan/Fly ash	Slag	Limestone
IS 16415:2015	Composite cement	35 - 65	15 - 35	20 - 50	
IS 16415:2015 R2023, amd 01	Composite cement	45-50	10-25	25-40	
ASTM C595-10	Ternary blended cement		≤ 40	≤ 70	≤ 15
BS EN 197-1:2011	CEM V/A	40 - 64	18 – 30 – Limited to any 2 constituents		
BS EN 197-1:2011	CEM V/B	20 - 38	31 – 49 – Limited to any 2 constituents		
BS EN 197-5:2021	CEM VI (S-V)	35 - 49	31 - 59	6 - 20	
BS EN 197-5:2021	CEM VI (S-L)	35 - 49	31 - 59		6 - 20
BS EN 197-5:2021	CEM II /C-M	50-64	36-50 – Limited to any 2 constituents		

4 Fresh properties of the blended cement concrete

An important note for this literature review section. As research articles based on the composite cement that complies with the IS 16415:2015 is very limited. For this review, the results from the studies where the OPC blended or replaced with the secondary cementitious materials (SCMs) especially with fly ash and slag is considered. As the report will be reviewed by the committee of experts, this report summarizes the major and final results obtained from the literature survey relevant to the standard and the project. In particular, the sources and the typical chemical and physical properties of the binders are considered as generic.

4.1 Initial Setting Time of the blended cement

As per IS 16415:2015, the minimum initial setting time of the composite cement should be more than 30 minutes. Figure 1 shows the initial setting time determined from the blended cement with varying proportions of fly ash, slag, and the remaining with the Ordinary Portland Cement (OPC). Composite cement blends have shown significant variation in the initial setting time values. As the sources of the raw materials and OPC will be varying with different researcher. Thus, a definite trend was not observed with respect to increase in fly ash or slag contents. However, all the reported values are more than the minimum requirement of 30 minutes. Most of the values falls in the range of 90 to 150 minutes. A huge scatter was observed from the data range from 120 to 200 mins. Thus, having the given range of fly ash and slag will not reduce the initial setting time of the composite cement. However, there may a variation in the normal consistency with the increasing fineness of the fly ash content in the composite cement blends due its ball bearing effect of spherical fly ash particles. However, a thorough investigation on the consistency or the water demand for making concrete may be required.

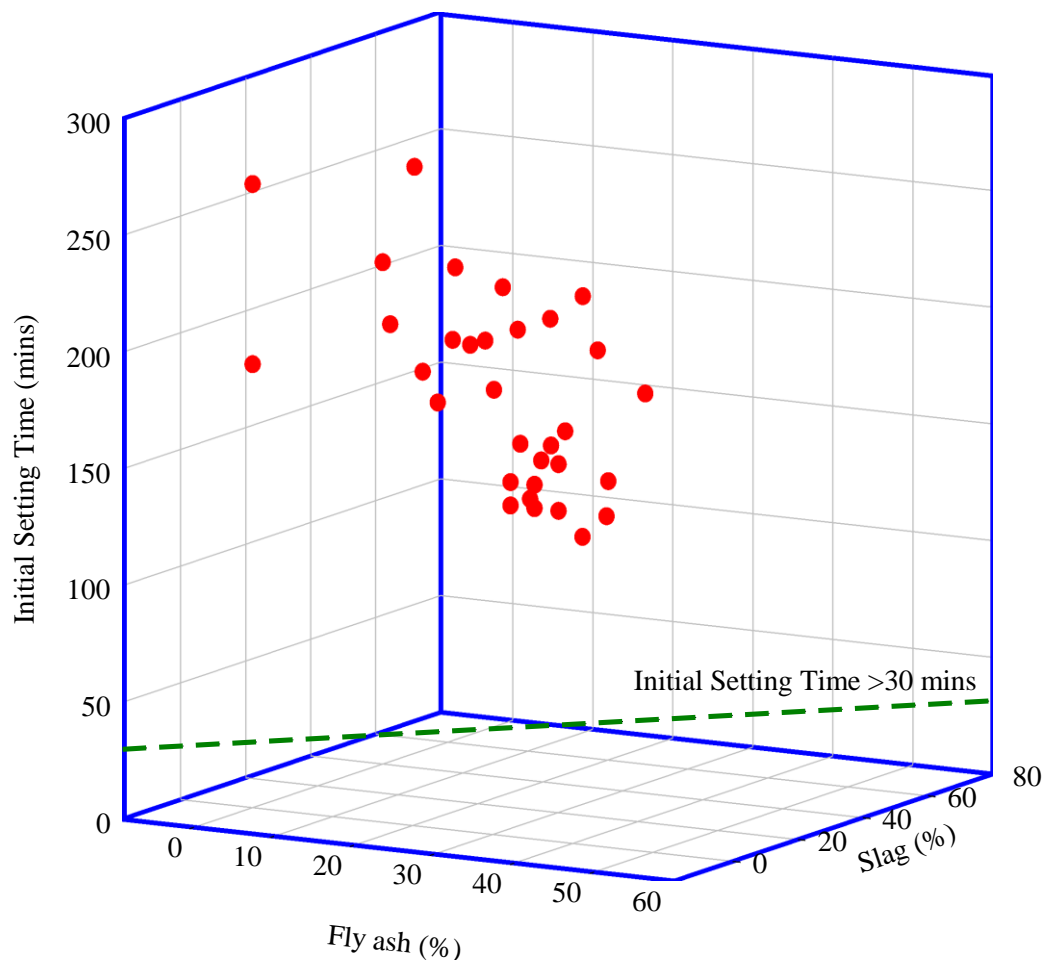


Figure 1 Influence of varying SCMs on the initial setting time of composite cement

4.2 Final Setting Time of the blended cement

As per IS 16415:2015, the maximum final setting time of the composite cement should be less than 600 minutes. Figure 2 shows the final setting time determined from the blended cement with varying

proportions of fly ash, slag, and the remaining with the OPC. Composite cement blends have shown significant variation in the final setting time values. As the sources of the raw materials and OPC will be varying with different researcher. Thus, a definite trend was not observed with respect to increase in fly ash or slag contents. However, most of the reported values are within the requirement of 600 minutes. Most of the values falls in the range of 180 to 300 minutes. A huge scatter was observed from the data range from 300 to 260 minutes. Thus, having the given range of fly ash and slag will not reduce the final setting time of the composite cement. The values above 600 minutes may be an outlier due to the change the test method say by using penetrometer instead of using vicat's apparatus (Anurag Gupta et al, 2019).

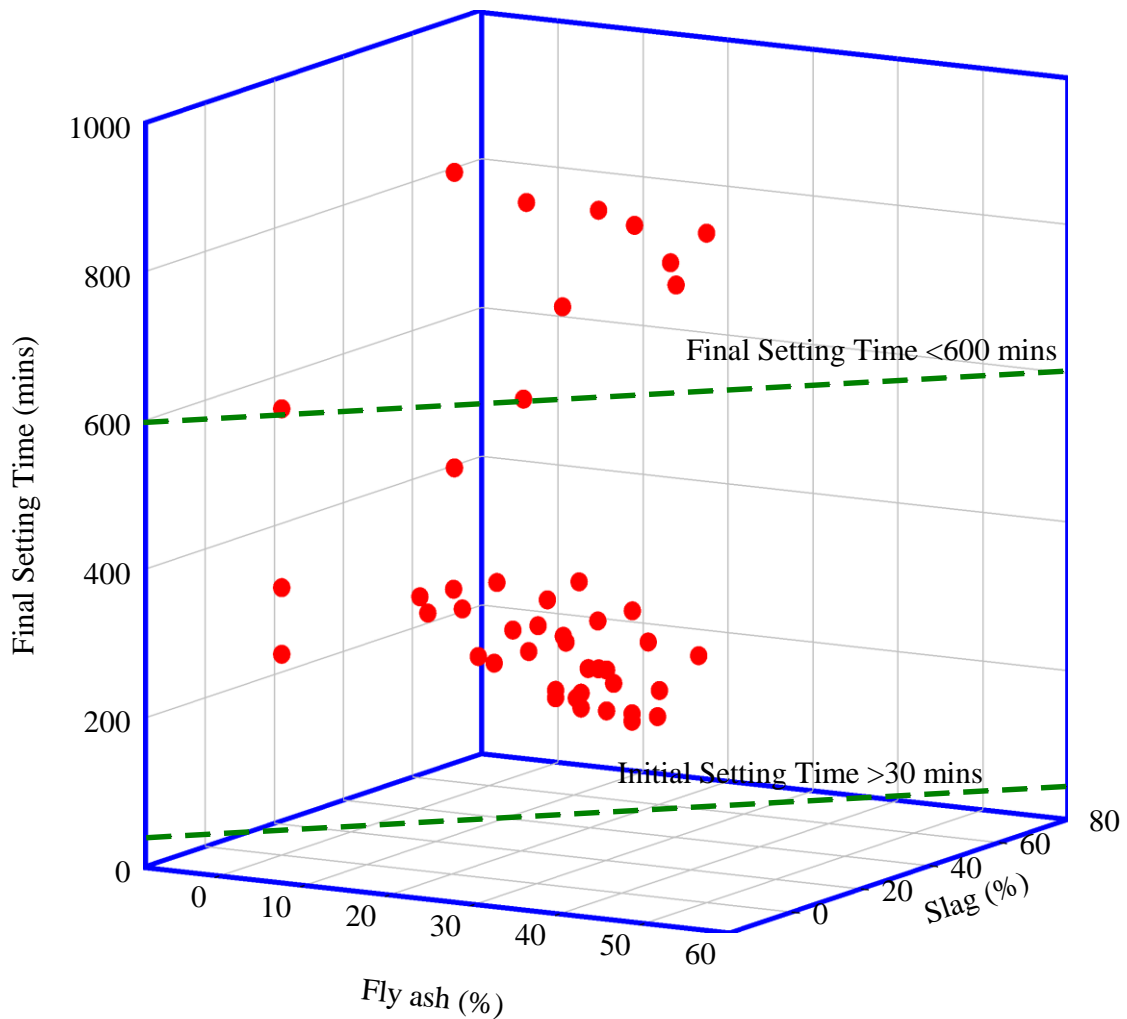


Figure 2 Influence of varying SCMs on the final setting time of composite cement

5 Harden properties of the blended cement concrete

The concrete made of blended cement containing the total binder content from 300 to 350 kg/m³ and the water to binder ratio between 0.4 to 0.45 have been considered for analysis. Though the scope of the project is to assess the compressive strength of composite cement, a very less literature available on it. Thus, the concrete with blended cement which will have the target compressive strength of around 45 MPa in 28 days is considered as the replicate for the mortar strength with OPC 43 grade as mentioned in Table 03 of both IS 269:2015 and IS 16415:2015. Further, IS 269:2015 specifies that the maximum compressive strength can be 58 MPa after 28 days for OPC 43 cement.

5.1 Compressive strength of concrete with ternary blending of varying SCMs content (fly ash and slag)

As per IS 16415:2015 (Amendment No.1, 2023), the average compressive strength is expected to be more than 23, 33, 43 MPa on 3 days, 7 days, and 28 days respectively. Figure 3 shows the compressive strength of concrete on 3rd day with varying the fly ash and slag contents. The expected target compressive strength as per the 16415:2015 on 3 days is 23 MPa. Most the results are more than 16 MPa and less than the minimum requirement of 23 MPa specified in the amendment 01 of IS 16415:2015. The concrete with high fly ash content (>30%) shows low early compressive strength as low as 16 MPa. Similarly, the increase in slag content shows significant increase in the strength gain with 3 days which is more than 25 MPa. Thus, the composite cement with the combination with higher slag and lower fly ash will be much suitable for early strength gain.

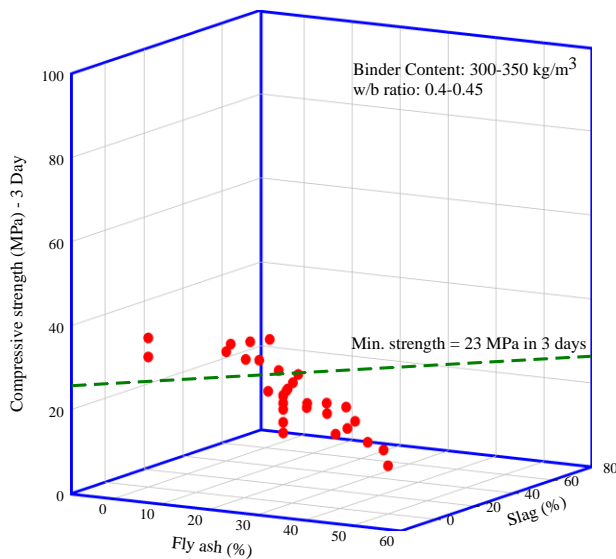


Figure 3 Compressive strength of blended cement concrete - 3 days

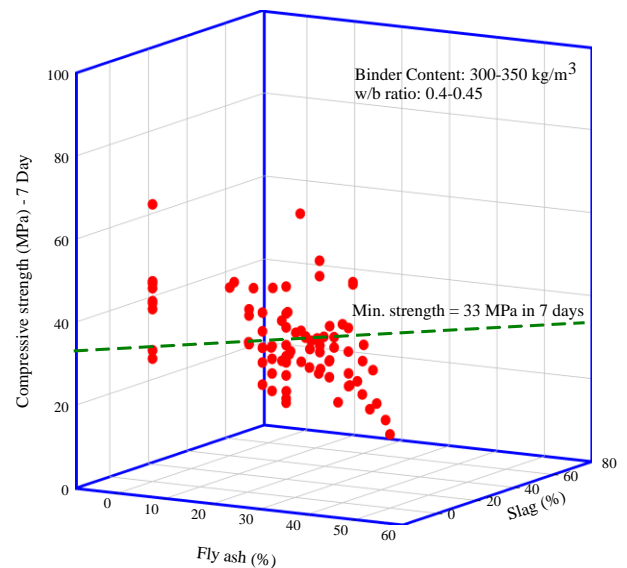


Figure 4 Compressive strength of blended cement concrete - 7 days

Figure 4 shows the compressive strength of concrete on 7th day with varying the fly ash and slag contents. The expected target compressive strength as per the 16415:2015 on 7 days is 33 MPa. Most the results fall at around 33 MPa. The concrete with high fly ash content (>30%) shows a very clear falling trend with compressive strength lower than 25 MPa with the increase of the fly ash content. Similarly, the increase in slag content shows significant increase in the strength gain with 7 days which is more than 35 MPa. They are few evidence that the concrete may gain strength even more than 40 MPa in 7 days with the higher slag content. Thus, the composite cement with the combination with higher slag and lower fly ash will be much suitable for the strength gain.

Figure 5 shows the compressive strength of concrete on 28th day with varying the fly ash and slag contents. The expected target compressive strength as per the 16415:2015 on 28 days is 43 MPa. Most the results within 43 MPa and with the 10% range as specified in IS 4031. There is no specific trend based on the increase in the particular SCM contents. Thus, the strength gain with any proportion of the fly ash and slag is expected to have a better strength gain by 28 days. This information makes it very clear that, the addition of more slag which gained higher early strength in 3rd and 7th day. However, considering the 28th day, the fly ash have hydrated in parallel to achieve the required target strength. The data on the left end that shows the steady increase in the strength for around 40 to 60 MPa without slag and fly ash is the OPC concrete

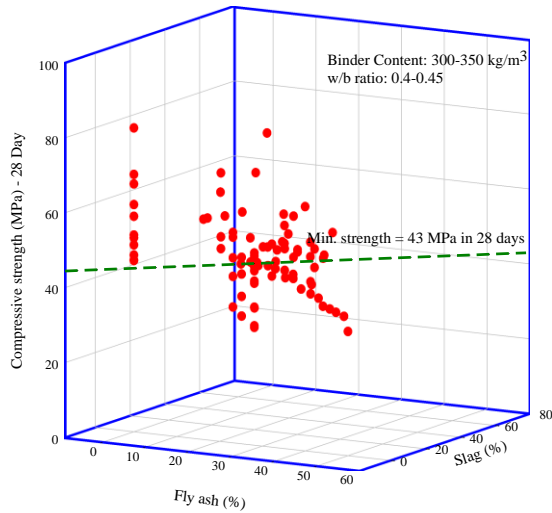


Figure 5 Compressive strength of blended cement concrete - 28 days

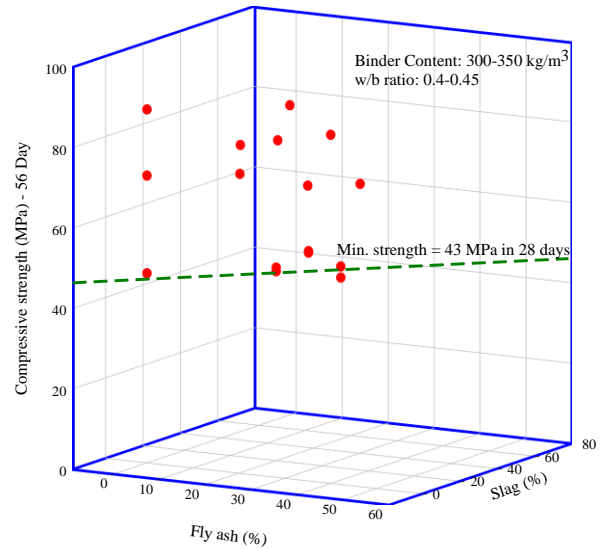


Figure 6 Compressive strength of blended cement concrete - 56 days

Figure 6 shows the compressive strength of concrete on 56th day with varying the fly ash and slag contents. Most the results are above 43 MPa and scatters up to 80 MPa. There is specific trend based on the increase in the particular SCM contents. Thus, the strength gain with any proportion of the fly ash and slag is expected to have a better strength gain after 28 days. However, considering the 28th day, the fly ash has hydrated in parallel to achieve the required target strength. Thus, the composite cement with the any combination with slag and fly ash will be suitable for the strength gain after 56 days.

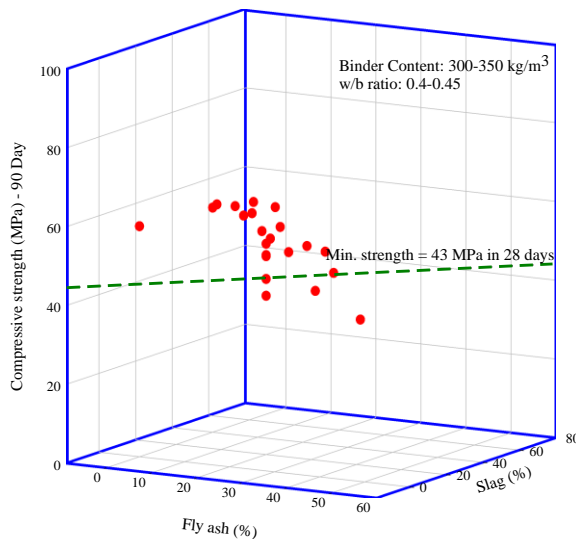


Figure 7 Compressive strength of blended cement concrete - 90 days

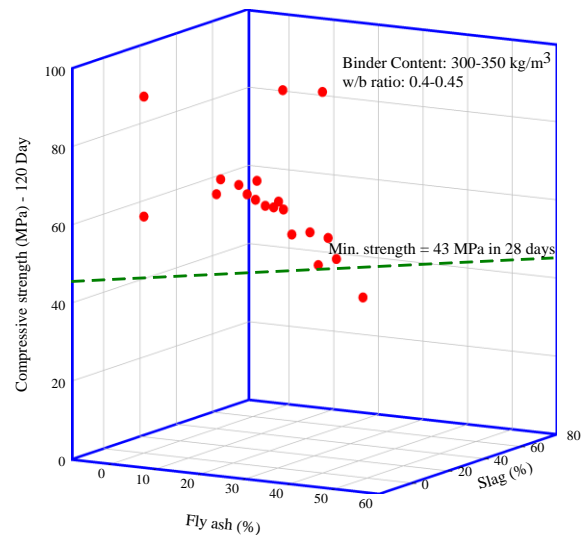


Figure 8 Compressive strength of blended cement concrete - 120 days

Figure 7 and Figure 8 shows the compressive strength of concrete on 90th day and 120th day with varying the fly ash and slag contents. In both the cases, the compressive strength of most of the specimens falls between 50 to 60 MPa. This shows, blended cement have the compliance with the maximum strength limit of 58 MPa provided in IS 269. The strength gain is not significant after 56 days.

5.2 Drying shrinkage behaviour of the blended cement concrete

Based on the outcomes from many of the studies, the drying shrinkage reduced linearly as the fly ash continuously increases (Jian et al., 2024). The variation of cement and slag slightly influences the drying shrinkage of cement blended with fly ash more than 35%. Fly ash dominates the total drying shrinkage when it the replacement is over 35 % in the blended cementitious system. A observations were identified in many similar studies. (Hu et al., 2017; Nath and Sarker, 2013).

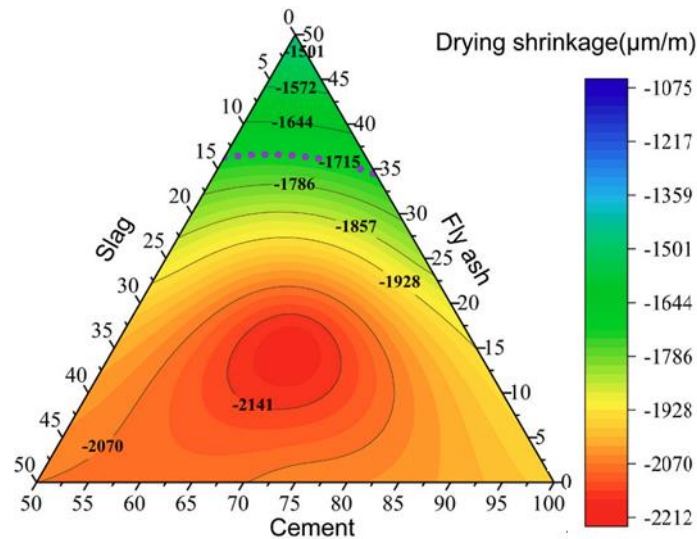


Figure 9 Drying shrinkage contours on blended cement concrete - 28 days

6 Review on the durability properties of the blended cement concrete

6.1 Influence of SCMs on the Water penetration depth

The water penetration depth of concrete with varying SCM content after 28 days of curing was shown in Figure 10. A clear trend was observed with the increase in the fly ash content. The depth of penetration was reducing linearly with the increase in the fly ash content and the slag content together. (Venkatachalapathy V et al. 2023, Mithun et al. 2018). The performance against the water penetration was low in the mixes with only fly ash or only slag. The depth of penetration is low in the ternary blended cement.

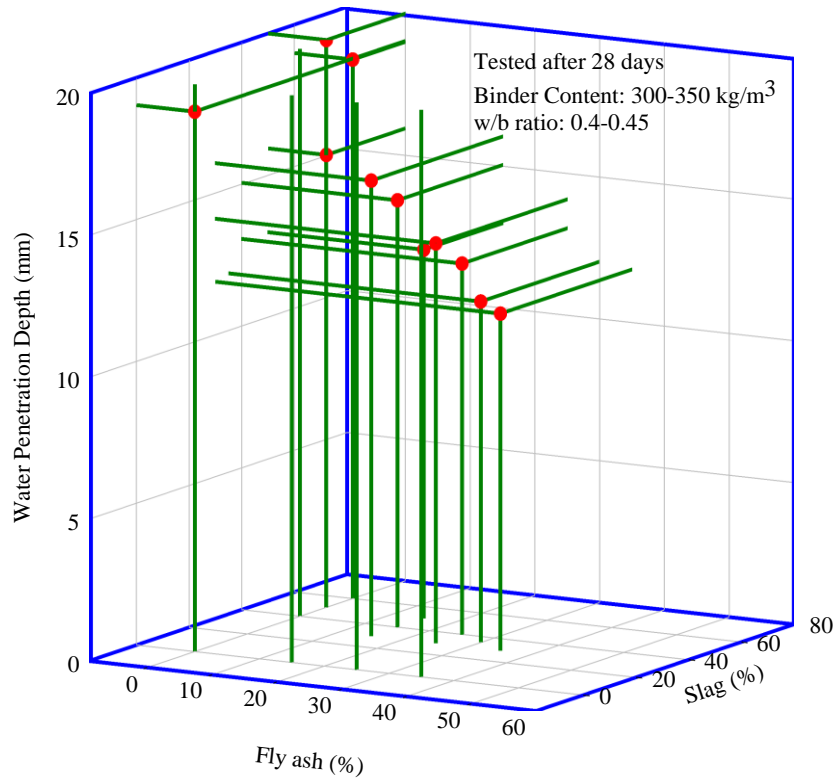


Figure 10 Depth of water penetration on blended cement concrete - 28 days

6.2 Influence of SCMs on the Rapid Chloride Penetration Test

To check the effect of SCMs on the durability of concrete, the Rapid Chloride Penetration Test (RCPT) was conducted as per ASTM C1202 guidelines at 56 days.

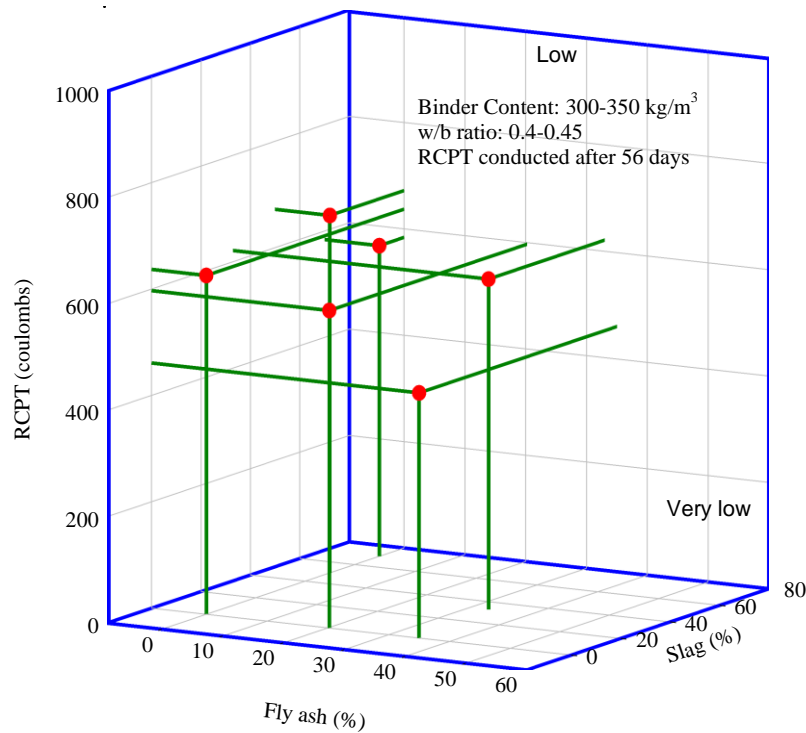


Figure 11 RCPT values tested on blended cement concrete - 56 days

The RCPT is one among the most common test conducted to evaluate the resistance of concrete to chloride ions ingress through electrical conductivity measurements. Figure 11 shows the RCPT

values of the blended cement concrete obtained after 56 days of curing. All the data falls in the classification as very low as the data range less than 800 coulombs. Thus, the RCPT or the electrical conductivity is very less than a normal concrete.

6.3 Review on carbonation data

The colourless appearance due to phenolphthalein test was measured from the edge of the specimen was noted as carbonation depth after exposing to an accelerated carbonation as per IS 516 (Part 2/Sec 4):2021. The depths of carbonation measured periodically for fly ash and slag blended concrete exposed to 3 % CO₂ and depths measured at 88 days and 146 days are shown in Figure 12. (Venkatachalapathy V et al. 2023).

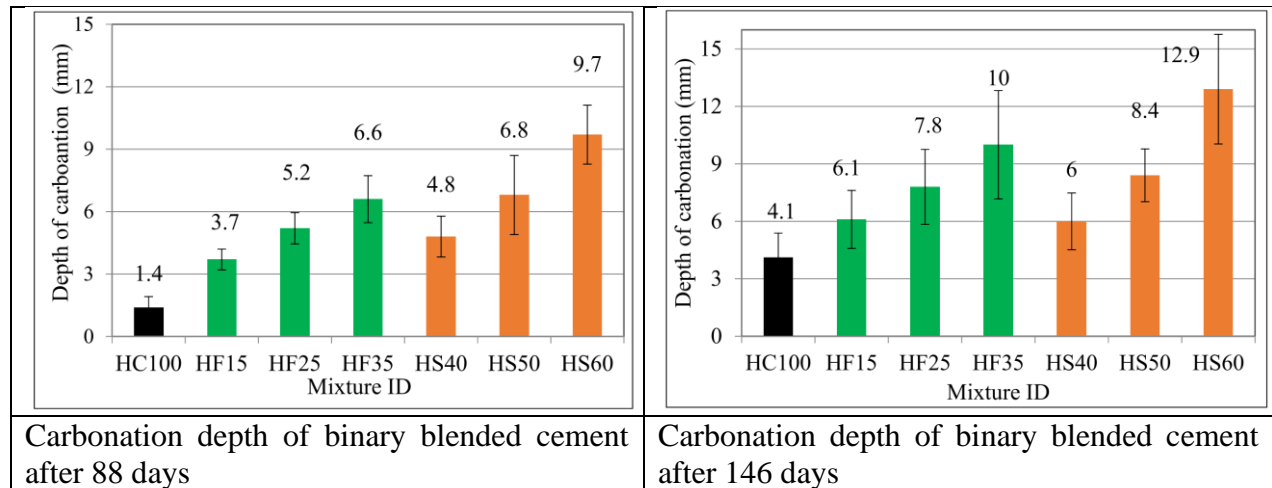


Figure 12 Carbonation depth of binary blended cement concrete – 88 and 146 days

When the substitution rate increased from 40% to 60%, concrete containing slag-blended cement showed greater carbonation depth compared to mixtures with 35% fly ash. The pattern of increasing carbonation depths for OP) and other SCM blended mixtures remained consistent during the extended 146-day accelerated carbonation test. Concrete mixtures incorporating 15% fly ash and 40% slag exhibited similar carbonation depths, which were comparatively lower than those observed in concrete containing higher proportions of fly ash (25% and 35%) and slag (50% and 60%). In summary, the accelerated carbonation test revealed that the carbonation resistance of high-workability concrete blended mixtures decreased in the following order: 100% OPC, 15% fly ash, 40% slag, 25% fly ash, 50% slag, and finally 35% fly ash and 60% slag replacements.

7 Conclusion of the literature review on Composite Cement

Based on the literature review, it is evident that the composite cement are better than the PPC, PSC, not only in terms of cost and sustainability, and also better in the performance of strength, durability, etc. Thus, a well-defined standard on the use of composite cement would bring value addition to the construction industry and saves the natural resources by utilizing the abundant industrial wastes.

8 Aim and Significance of the Project

Composite cement is formulated by blending Portland clinker with other finely ground materials such as pozzolana and granulated blast furnace slag. It can be used in making concrete, mortar, and plaster. The production of the elementary cement component, Portland clinker, is characterized by high carbon dioxide emission levels along with high energy and material consumption (Salas Montoya et al. 2023). This stimulates the search for solutions aiming at the use of cement and concrete components with reduced Portland clinker content. Therefore, fly ash which is a residue

obtained from the combustion of pulverized coal, and granulated slag obtained by processing molten slag by rapidly chilling or quenching it with water or steam and air are used as partial replacements for Portland clinker (Giergiczny 2019). The composite cement (conforming to IS: 16415-2015 (IS 16415: 2015 (Reaffirmed 2020) n.d.)) can be produced either by inter-grinding of Portland cement clinker (conforming to IS: 16353-2015 (IS 16353: 2015 (Reaffirmed 2020) n.d.)), granulated slag (conforming to IS: 12089-1987 (IS 12089: 1987 (Reaffirmed 2018) n.d.)) and fly ash (conforming to IS: 3812(I)2013 (IS 3812 : Part 1 : 2013 (Reaffirmed 2022) n.d.)) with the addition of natural/chemical gypsum or by an intimate and uniform blending of ordinary Portland cement (conforming to IS: 269-2015 (IS 269: 2015 (Reaffirmed 2020) n.d.)), finely ground granulated slag and fly ash with the addition of ground gypsum. The composite cement is manufactured using 35-65% Portland cement clinker/ ordinary Portland cement along with 15-35% fly ash and 20-50% granulated blast furnace slag together as the blending component (IS 16415: 2015 (Reaffirmed 2020) n.d.). The partial replacement of clinker, which is not only the more expensive component of cement but also the most resource, energy, and emission intensive, with mineral additives improves the sustainability of the material.

The durability of concrete depends majorly upon its resistance to the ingress of deleterious agents including moisture, carbon dioxide (CO₂) and chlorides. These agents that enter the concrete can lead to corrosion of steel reinforcement and reduce the life of the structures drastically. As such, the durability of concrete depends largely on its permeability, defined as the ease with which it allows the fluids to pass through it. It is also dependent on several other factors such as w/b ratio, use of different binder materials etc. The use of granulated blast furnace slag and fly ash apart from Portland clinker in composite cement manufacturing could affect the strength and durability characteristics of the concrete owing to the changes they would bring in pore structure, permeability, particle packing, and microstructure (Konieczna et al. 2021). Therefore, this study is necessary to ascertain the ideal proportion of clinker to use in composite cement to prevent adverse impacts on the performance, durability, and service life of reinforced cement concrete buildings constructed using this type of cement.

The following part of the document contains the modified proposal as per the test requirements.

9 Objectives of the study and final deliverables as per the project description

9.1 Scope of the study

- a) Undertake study and comparative analysis of the available literature on the subject which includes International Standards and Journals, Research Papers published on the subject, guidelines/regulations issued by the concerned Ministry/Government agency and any other study conducted by other industry/organization.
- b) Carry out factory visits to the manufacturers of composite cement, and identify/collect the information regarding the manufacturing base of the product and the testing facilities available in the country. During the visit, the researcher needs to observe the following activities: 1) Variety of the products manufactured, 2) Manufacturing process, 3) In-process quality and safety checks, 4) Marking and labelling, 5) Packaging requirements, 6) Testing facilities and equipment used at the factory location, 7) Sustainability practices adopted by the manufacturer such as energy consumption, use of renewable energy resources, waste management and disposal mechanisms and reduction of carbon footprint.
- c) Visit Government/NABL accredited testing laboratories (one government and one private) to identify the testing infrastructure available in the country.
- d) Collect requisite samples of composite cement from the manufacturers and conduct various tests to obtain data on various physical, chemical and durability requirements such as RCPT, RCMT, chloride-induced corrosion and carbonation-induced corrosion of composite cement.

9.2 Methodology as per the project description

1. To study the chemical and physical characteristics of cement samples from two different manufacturers (with 45%, 40% and 35% clinker), and Portland Pozzolana Cement (PPC).
 - To assess the following chemical characteristics: insoluble residue, magnesia, total sulphur content, sulphide sulphur, loss on ignition, chloride, alkali content.
 - To determine the following physical properties: fineness, soundness, setting time, compressive strength drying shrinkage
 - 1.1 To determine the chemical composition, the major and trace elements in the cement samples by carrying out X-ray fluorescence (XRF) method
 - 1.2 To study the phase composition of cement by X-ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) methods
 - 1.3 To analyse the particle size of the cement samples by Laser Diffraction (LAS) method
2. To prepare concrete mixes for each cement sample with a water/binder ratio of 0.6, 0.5, 0.45 and 0.4 adhering to M10, M20, M25 and M30 concrete mix proportions respectively.
3. To identify the dosage of superplasticizer for each concrete mix to achieve a target slump of 100-140 mm.
4. To determine the compressive strength of concrete mixes at 7,14, 28 and 90 days of curing
5. To test the concrete for carbonation resistance by measuring carbonation depth and chloride resistance by Rapid Chloride Penetration test (RCPT) and Rapid Chloride Migration test (RCMT)
6. To evaluate corrosion in reinforced concrete elements by the following methods:
7. Accelerated carbonation test.
8. Instantaneous corrosion current density measurements

Table 3. Different clinker content, w/b, specimens for various durability tests on concrete

Supplier	Test Duration (days)		7,14,28,90	28, 56, 90	28, 56, 90	28, 56, 90	From casting to corrosion rate
	Clinker (%)	w/b	Compressive strength (10 cm cubes)	RCPT (ø10 cm)	RCMT (ø10 cm)	Carbonation depth (4x4x16 cm)	Carbonation & Corrosion (lollipop)
A	35	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	40	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	45	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	PPC	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
B	35	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	40	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	45	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
	PPC	0.4	12	3	3	3	5
		0.45	12	3	3	3	5
		0.5	12	3	3	3	5
		0.6	12	3	3	3	5
Sub-total			384	96	96	96	160
Replicate mix			3	3	3	3	3
Total specimens to cast			1152	288	288	288	480

10 Road Map

Tasks	Dec 24	Jan 25	Feb 25	Mar 25	Apr 25	May 25
Factory visit to collect samples						
Interim report on literature review						
Casting and curing						
Compressive strength						
First progress report						
Durability studies						
Corrosion studies						
Final Project Report						

11 Plan of work, methods, and techniques to be used.

To obtain six samples of composite cement from different manufacturers based on clinker percentage (two with 45% clinker, two with 40% clinker and two with 35% clinker). Also, samples of PPC will be taken from the same manufacturers for comparison purposes.

11.1 Determination of chemical and physical properties according to Indian standards

The chemical and physical requirements of the composite cement samples as mentioned in IS 16415:2015 (IS 16415: 2015 (Reaffirmed 2020) n.d.) are determined. The samples are tested as per the methods given in IS 4032:1985 (IS 4032:1985 (Reaffirmed 2005) 1985) to determine the chemical characteristics including the per cent by mass of insoluble residue, magnesia, total sulphur content, sulphide sulphur, loss on ignition, chloride, and alkali content. The physical characteristics such as fineness, soundness, setting time, compressive strength and drying shrinkage are tested according to the methods given in IS 4031. The permissible limits for each of these parameters as prescribed in IS 16415:2015 (IS 16415: 2015 (Reaffirmed 2020) n.d.) are listed in Table 4.

Table 4. Permissible limits for chemical and physical characteristics according to IS 16415:2015 Reaffirmed in 2023

Sl. no.	Parameter to test	Permissible limits	Standard
Chemical Characteristics			
1	Insoluble mass (% by mass)		
	Maximum	$x + \frac{4.0(100 - x)}{100}$	IS 4032:1985
	Minimum	0.6x where x is % of pozzolana in composite cement	
2	Magnesia (% by mass), Max	8.0	IS 4032:1985
3	Total sulphur content calculated as sulphuric anhydrite (% by mass), Max	3.5	IS 4032:1985
4	Sulphide sulphur (S), Max	0.75	IS 4032:1985
5	Loss on ignition (% by mass), Max	5.0	IS 4032:1985
6	Chloride content (% by mass), Max	0.1	IS 4032:1985
7	Alkali content	0.05	IS 4032:1985
Physical Characteristics			

1	Fineness, m ² /kg, Min	300	IS 4031 (Part 2):1999
2	Soundness		IS 4031 (Part 3): 1988
	By Le-Chatelier method, mm, Max	10	
	By Autoclave test, %, Max	0.8	
3	Setting time		IS 4031 (Part 5): 1988
	Initial, min, Min	30	
	Final, min, Max	600	
4	Compressive strength, MPa		IS 4031 (Part 6): 1988
	72±1 h, Min	23	
	168±2 h, Min	33	
	674±4 h, Min	43	
5	Drying shrinkage, %, Max	0.15	IS 4031(Part 10): 1988

11.1.1 XRF test

XRF test is conducted to quantify the elemental composition of the clinker. XRF entails exposing a sample of cement to X-ray light, which excites the elements present in the sample. The elements emit light as they return to their ground state. The light emitted as the elements relax is distinctive to the elements present in the sample, and measuring the fluorescence makes it possible to calculate the exact chemical composition of the sample. Pressed powder pellet specimens are prepared by grinding the cement sample to a fine powder and then mixing the resulting powder with a binding agent. The binding agent typically used is boric acid or a cellulose mixture. The powders are put in a mould (called a 'die') with a 32 mm or 40 mm cylindrical bore and compressed under a load of around 20 tons using a hydraulic press. The final pellet is then sent for analysis on the XRF spectrometer (Khelifi et al. 2017).

11.1.2 XRD tests

XRD is applied to identify and measure the mass fractions of various crystalline phases in Portland clinker and cement. Powder diffractometry is by far the most used technique to obtain x-ray diffraction data, and is how cement and clinker are analysed, provided they are prepared and ground to a suitable particle size. The specimens are homogeneously mixed and ground to about 45 µm for qualitative analysis. Powders are pressed into a sample holder and a flat surface created will be exposed to x-rays. XRD allows one to determine directly what phases are present and their relative proportions (Sim et al. 2023).

11.1.3 TGA tests

TGA is an analytical technique used to determine a material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a sample is heated at a constant rate. As different compounds tend to leave within a specified temperature interval, TGA can be used to quantify the amounts of different phases in a sample. TGA measures H₂O, OH⁻, CO₂, SO₄²⁻ released during the heating of the sample under N₂ environment (Sim et al. 2023).

11.1.4 Particle size analysis

One of the most common methods for the characterization of the particle size distribution in cement is the Laser diffraction technique. The LAS method involves the detection and analysis of the

angular distribution of light produced by a LASER beam passing through a dilute dispersion of particles. The method requires that the particles be in a dispersed state, either in liquid (suspension) or in air (aerosol). The wet LAS method is commonly adopted for cement and due to the reactive nature of cement in water, alcohols, such as isopropanol, methanol, and ethanol, are commonly used in its place (Cepuritis et al. 2017).

11.2 Mechanical and durability tests on various concrete mixes

Concrete specimens as required for each of the following tests are prepared for each of the composite cement samples and PPC concrete with different w/b ratios, 0.6, 0.5, 0.45 and 0.4. Refer to Table 2 in Section 10 for the details of the experimental design.

11.2.1 Workability and Compressive strength tests

The fresh concrete is tested for workability using the slump cone test as prescribed in IS 1199:2018 (Part 5) and the amount of superplasticizer is adjusted to attain a target slump in the range of 100 – 140 mm. Thereafter, the concrete mixes will be cast and cured under normal curing conditions i.e. at $27\pm 2^\circ\text{C}$ under 90% RH for 28 days. Three replicates of each concrete sample are prepared for testing.

The compressive strength of the concrete mixes is tested on 7, 14, 28 and 90 days of curing on 150 x 150 x 150 mm cube specimens following the method given in IS 516 (Part-1 Sec-1):2021. A gradual loading of 14 N/mm²/minute is applied using a servo-controlled compression testing machine until the specimen fails. The average of the three replicate specimens is reported.

11.2.2 Accelerated carbonation test.

Concrete containing fly ash and slag is prone to early carbonation attack due to the additional secondary pozzolanic reactions that consume the calcium hydroxide and hence carry the risk of carbonation-induced corrosion of rebars when used in reinforced concrete elements (Divsholi et al. 2014). Therefore, to measure the carbonation depth, concrete prism specimens of dimensions 100 x 100 x 500 mm are subjected to 3% CO₂ at $27\pm 2^\circ\text{C}$ and 40-60% RH in a carbonation chamber as per IS 516: Part 2: Sec 4: 2021 and IS 516: Part 5: Sec 3: 2021. The specimens are coated with an anti-carbonating coating at the cross-sectional surface to ensure carbonation only through the side surfaces. The testing is done on 28, 56, 90, and 120 days of carbon dioxide exposure. On the specified days of testing, the specimens are taken out and dry cut at 100 mm from one end and 1% phenolphthalein indicator is sprayed on the freshly cut surface. The non-carbonated concrete area will turn pink, and the carbonated area will remain colourless. The maximum depth at which the concrete has turned pink from the edges is considered the carbonation depth of the concrete specimen. The average of the three replicate specimens is calculated and reported. After every test, the remaining portion of the prism specimen is placed back in the chamber after applying an anti-carbonation coating on the previously tested surface. Figure 13 shows the schematic of the carbonation depth measurement and Figure 14 shows the tested concrete specimens.

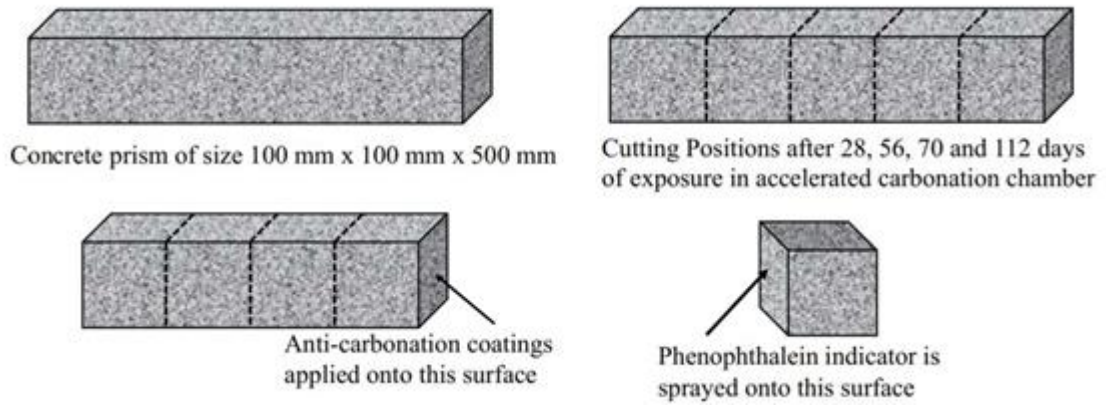


Figure 13 Specimens to test to find the pH of concrete

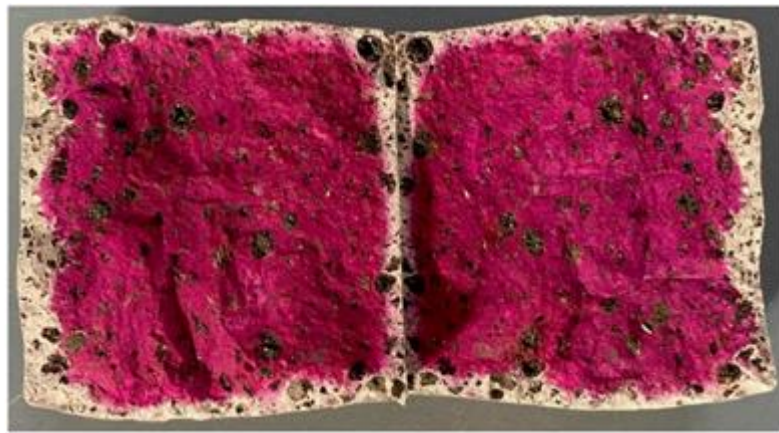


Figure 14 Carbonated specimen after phenolphthalein test.

11.2.3 Rapid Chloride Penetration Test (RCPT)

The resistance of concrete to penetration by chlorides is an important factor in protecting reinforced concrete structures from premature deterioration.

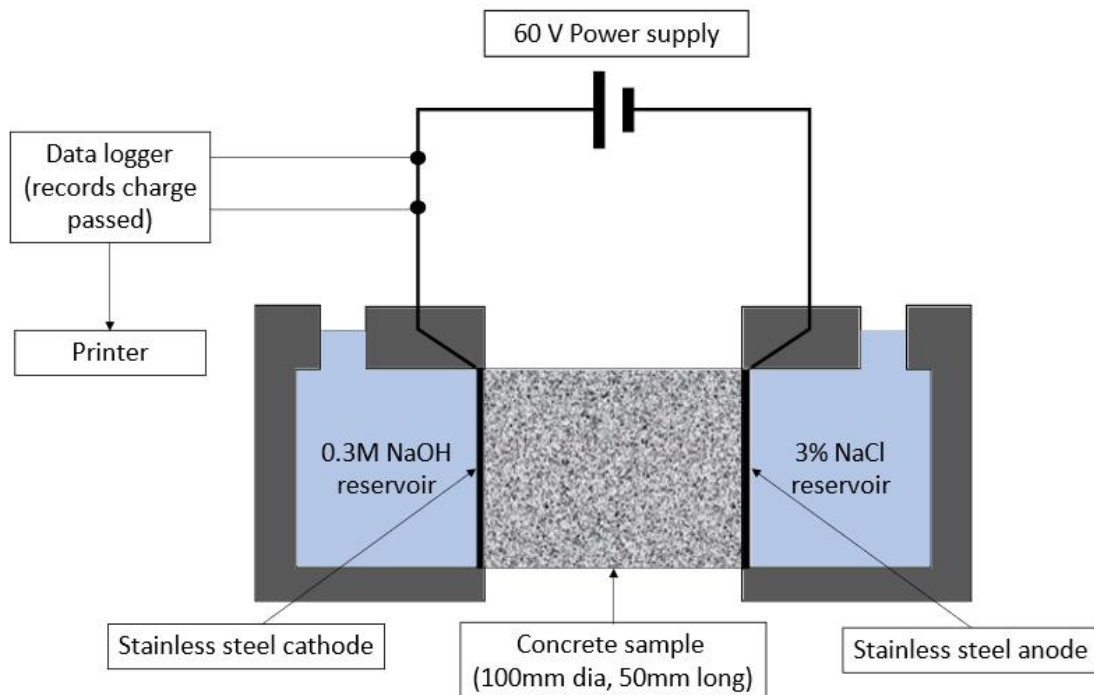


Figure 15 RCPT test setup

Therefore, measuring the movement of chlorides and the resistance of concrete against the transport of ions due to external potential will be commonly assessed using the Rapid Chloride Permeability Test (RCPT) as per ASTM C1202-19 (ASTM C1202-19 n.d.). The test is performed by monitoring the amount of electrical current that passes through a sample 50 mm thick and 100 mm in diameter in 6 hours. A voltage of 60V DC is maintained across the ends of the sample throughout the test. Figure 15 shows the schematic of the RCPT setup. The average of the RCPT results from the replicate specimens are evaluated. Based on the charge that passes through the sample, a qualitative rating is made of the concrete's permeability. A higher charge indicates higher chloride permeability and poorer quality of concrete. NIT Calicut has the completely automated testing facility to conduct RCPT tests.

11.2.4 Rapid Chloride Migration Test (RCMT)

Although RCPT is an easy test method that gives results in a short period, the current that passes through the sample during the test indicates the movement of all ions in the pore solution (that is, the sample's electrical conductivity) and not just chloride ions. The presence of fly ash, slag and even superplasticizers can create misleading results largely due to the chemical composition of the pore solution. Rapid Chloride Migration test (RCMT) as per the standard, NT Build 492 (NT Build 492 1999), measures the depth of penetration chlorides over a while due to external potential application rather than measurement of current in the case of RCPT. It is a short-term non-steady-state method allowing the expeditious determination of the chloride migration co-efficient of concrete on the 28th day of curing concrete. This methodology is essentially based on the migration of chloride ions into previously lime-saturated concrete specimens by applying an external electrical voltage, which is adjusted according to the electrical current in the system. The test consists of applying an external electrical potential for a certain amount of time, forcing the chloride ions in the sodium chloride (NaCl) solution to migrate into the specimen. The time of the test depended on the applied voltage. Then, the specimen is split into two halves and sprayed with an AgNO_3 solution. The chloride penetration depth corresponded to the visible limit of the white silver chloride precipitation. The depths of penetration at an interval of 10 mm are measured and the average depth is used for calculation of the non-steady state chloride migration coefficient as mentioned in the standard. Figure 16 shows a schematic of the RCMT test setup. NIT Calicut has the completely automated testing facility to conduct RCMT tests.

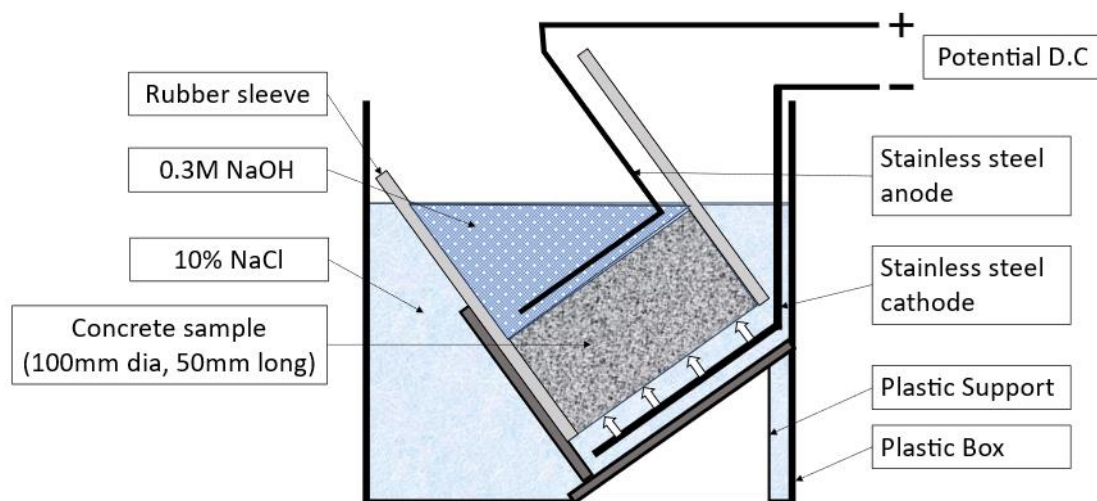


Figure 16 RCMT test setup

11.3 Evaluation of corrosion in concrete beams

The major risk involved in reinforced concrete with higher permeability is corrosion of rebars. Chloride ingress and carbonation are the most common causes of initiation of reinforcement corrosion. These conditions are aggressive: they weaken the protective coating over the rebars in case of chloride ingress or reduce the pH of the concrete in case of carbonation, allowing corrosion to occur before the minimum duration of the expected useful life has elapsed. In the presence of moisture and oxygen, chloride ions at the interface of steel and concrete can destroy the passive film around the rebars locally and initiate local corrosion. Therefore, an evaluation of both carbonation-induced and chloride-induced corrosion in reinforced concrete (RC) beams will be performed on the two different sets of samples. NIT Calicut has the complete advanced testing facility to conduct carbonation-induced and chloride-induced corrosion tests like LPR, EIS, gravimetric mass loss, half-cell potential test, etc.

11.3.1 Study on corrosion induced by carbonation

Carbonation is the process of carbon dioxide reacting with hydrated cement paste in pore water to reduce the natural alkalinity of concrete. The passive layer on the rebars becomes unstable in a lower pH environment and hence poses a risk to be corroded. Therefore, the study aims to determine the carbonation front in reinforced concrete subjected to extreme carbon dioxide conditions and find the rate of corrosion of rebars from the linear polarization resistance method.

The linear polarization resistance (LPR) technique is a non-destructive electrochemical test method that measures the corrosion current density (i_{corr}) of steel reinforcement as per BS EN 00480-14-2006. The method involves increasing or decreasing the potential of reinforcement relative to its open circuit potential by a fixed amount, ΔE , and monitoring the current decay, Δi . In the vicinity of the open circuit potential (10 to 30 mV), the voltage-current density curve is linear, and the slope of this section is defined as the polarization resistance, R_p .

$$R_p = \left(\frac{\Delta E}{\Delta i} \right)_{\Delta E \rightarrow 0}$$

Current density, Δi , is the measured current divided by the exposed steel surface area. The potential can be changed potentiostatically (potential is changed a fixed amount and current change is monitored) or galvanostatically (fixed amount of current is supplied and the change in potential is monitored). The instantaneous corrosion current density, i_{corr} , is calculated by dividing the Stern-Geary constant, B , by the R_p value as shown in

$$i_{\text{corr}} = \frac{B}{R_p}$$

where R_p is in Ωcm^2 , and B is in Volts. The Stern-Geary constant, B , is a combination of anodic and cathodic Tafel coefficients, β_a and β_c as shown in

$$B = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)}$$

The value B has been documented to vary from 13 to 52 mV for a wide range of systems. Many studies in the literature use 26mV as a B value. A three-electrode system is typically used to measure the polarization resistance of reinforcement steel that acts as the working electrode. A counter electrode of the same size as the reinforcing steel and a reference electrode is used to shift the potential 20 mV from the free corrosion potential in the anodic or cathodic direction.

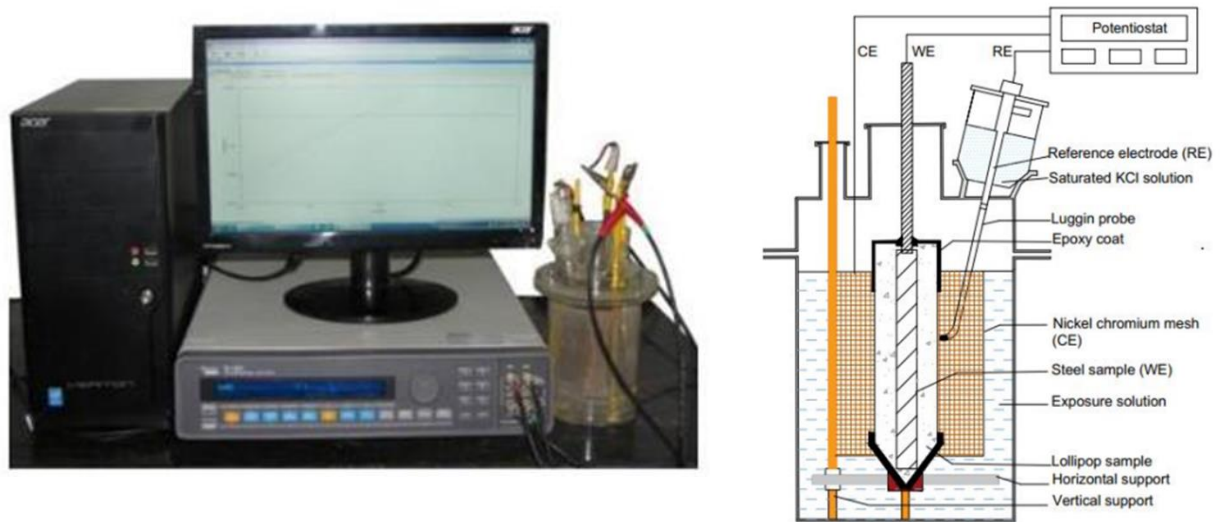


Figure 17 Linear polarization resistance (LPR) technique to measure instantaneous corrosion current density (i_{corr})

Figure 17 shows the schematic of the annular corrosion cell test setup used in this study. A lollipop-type specimen with a steel reinforcement embedded at the centre of a mortar cylinder is used. The cut steel pieces (8 mm diameter and 70 mm long) are cleaned by immersing them in an ethanol bath in the ultrasonic cleaner. The prepared steel pieces are embedded in 110 mm long cylindrical mortar and ≈ 10 mm cover. Five specimens for each of the samples are prepared and cured in a laboratory environment (25 ± 1 °C and 65 ± 5 %RH) for 24 ± 1 h. After this, the specimens are removed from the plastic mould and cured at 25 ± 1 °C and 95 ± 3 %RH for an additional 13 days. Then, the mortar surface of each specimen was coated with epoxy leaving a 50 mm long region in the middle for exposure. The specimen to test for carbonation-induced corrosion the specimen will be exposed to 3% CO₂ at 27 ± 2 °C and 40-60% RH for 120 days. Then the specimen shall be immersed 10 mm in potable water for a duration of 14 days. Once in a week since the casting, the LPR test was conducted using a scan range of ± 15 mV with respect to the measured OCP and a scan rate of 0.1667 mVs⁻¹. When the R_p decreases to a value below 10 k Ω cm² (say, threshold R_p) for two consecutive wet-dry cycles, testing was stopped, as shown in Figure 18 and the specimen was autopsied for visual observation and to determine the critical pH at the steel-cementitious interface.

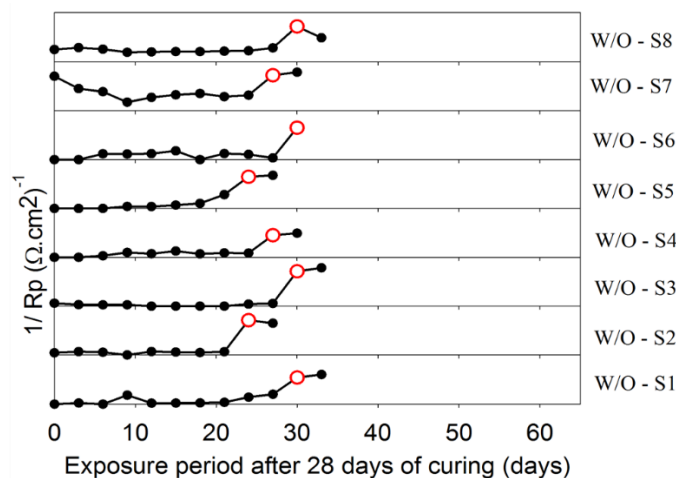


Figure 18 Typical plot showing the corrosion rate of steel exposed to carbonation.

At the end of the testing period, or considerable corrosion have progressed, the specimen will be autopsied, and the corroded steel rebar will be cleaned as per ASTM G1 procedure as shown in Figure 19.



Figure 19 Corroded steel rebar cleaned to determine the cumulative corrosion rate

Based on the results obtained from the ASTM G1 procedure, the cumulative corrosion rate will be determined using the gravimetric mass loss method, using the following formula,

$$\text{Corrosion rate } (\mu\text{m/year}) = \frac{K \times W}{A \times T \times D}$$

Where, K = a constant to estimate the corrosion in micrometres per year ($\mu\text{m/year}$) = 8.76×10^7

T = time of exposure, in hours,

A = area of steel rebar exposed to corrosion, in cm^2

W = mass loss, in grams, and

D = density of steel rebar = 7.86 g/cm^3

Finally, the results of the corrosion studies will be presented for the specimen with 2 types of clinker and 4 types of water binder ratio, and PPC by different test methods will be analysed and compared.

12 Deliverables

- A comprehensive literature review document summarizing recent developments in the production of composite cement.
- Compilation of relevant rules and regulations from Central/State governments on the manufacturing of composite cement.
- Detailed reports on observations from manufacturing unit visits, including insights into production processes.
- Completed questionnaire surveys and analysis highlighting key findings related to production, manufacturing, and consumption data.
- A detailed literature review and report on the manufacturer and laboratory visits.
- An analytical report covering all the aspects of physical, chemical, mechanical, and durability performance.
- Comparative analysis of test results against relevant standards and specifications.
- A workshop for the dissemination of the research outputs to the practising engineers (in government and private sector) will be conducted at the end of the project.
- One or more SCI/Scopus Indexed will be obtained from the outcome of the project, with the consent of the BIS project monitoring committee.
- PG and PhD students will be guided/trained to disseminate the learning through this project to society.

13 References

1. AASHTO M240. n.d. “Standard Specification for Blended Hydraulic Cement.”
2. AS 3972-2010. n.d. “General purpose and blended cements.”
3. ASTM C1202-19. n.d. “Standard Test Method for Electrical Indication of Concrete’s Ability to Resist Chloride Ion Penetration.”
4. ASTM C595/C595M-14. 2014. “Standard Specification for Blended Hydraulic Cements.” ASTM International, United States. <https://doi.org/10.1520/C0595>.
5. BDS EN 197-1: 2003. n.d. “Cement-Part 1: Composition, specifications and conformity criteria for common cements.”
6. CAN/CSA-A362-98. n.d. “Blended Hydraulic Cement.”
7. Cepuritis, R., E. J. Garboczi, C. F. Ferraris, S. Jacobsen, and B. E. Sørensen. 2017. “Measurement of particle size distribution and specific surface area for crushed concrete aggregate fines.” *Advanced Powder Technology*, 28 (3): 706–720. <https://doi.org/10.1016/j.appt.2016.11.018>.
8. CSA A3001-03-2004. n.d. “Cementitious Materials for Use in Concrete.”
9. Discussion Paper on Composite Cement, Confederation of Indian Industry (CII), 2016
10. Divsholi, B. S., T. Y. D. Lim, and S. Teng. 2014. “Durability Properties and Microstructure of Ground Granulated Blast Furnace Slag Cement Concrete.” *International Journal of Concrete Structures and Materials*, 8 (2): 157–164. <https://doi.org/10.1007/s40069-013-0063-y>.
11. EN 197-5: 2021. 2021. “Cement-Part 5: Portland-composite cement CEM II/C-M and Composite cement CEM VI.” European Standard.
12. Giergiczny, Z. 2019. “Cement and Concrete Research Fly ash and slag.” 124 (July). <https://doi.org/10.1016/j.cemconres.2019.105826>.
13. Hu, X., Z. Shi, C. Shi, Z. Wu, B. Tong, Z. Ou, and G. de Schutter. 2017. “Drying shrinkage and cracking resistance of concrete made with ternary cementitious components.” *Construction and Building Materials*, 149: 406–415. Elsevier BV. <https://doi.org/10.1016/j.conbuildmat.2017.05.113>.
14. IS 12089: 1987 (Reaffirmed 2018). n.d. “Specification for granulated slag for manufacture of Portland slag cement.”
15. IS 16353: 2015 (Reaffirmed 2020). n.d. “Portland Cement Clinker - Specification.”
16. IS 16415: 2015 (Reaffirmed 2020). n.d. “Composite Cement - Specification.”
17. IS 269: 2015 (Reaffirmed 2020). n.d. “Ordinary Portland Cement - Specification (Sixth Revision).”
18. IS 3812: Part 1: 2013 (Reaffirmed 2022). n.d. “Pulverised Fuel Ash - Specification Part 1 for use as Pozzolana in Cement, Cement mortar and Concrete.”
19. IS 4032:1985 (Reaffirmed 2005). 1985. “Method of Chemical Analysis of Hydraulic Cement (First Revision).” Bu. Indian. Stand. New Delhi.
20. Jian Zhang, Yuefeng Ma, Jiaping Liu, Xiangsheng Chen, Fangzhou Ren, Weibin Chen, Hongzhi Cui, Improvement of shrinkage resistance and mechanical property of cement-fly ash-slag ternary blends by shrinkage-reducing polycarboxylate superplasticizer, *Journal of Cleaner Production*, Volume 447, 2024,

21. Khelifi, S., F. Ayari, H. Tiss, and D. Ben Hassan Chehimi. 2017. "X-ray fluorescence analysis of Portland cement and clinker for major and trace elements: Accuracy and precision." *Journal of the Australian Ceramic Society*, 53 (2): 743–749. <https://doi.org/10.1007/s41779-017-0087-x>.
22. Konieczna, K., K. Chilmon, and W. Jackiewicz-Rek. 2021. "Investigation of mechanical properties, durability and microstructure of low-clinker high-performance concretes incorporating ground granulated blast furnace slag, siliceous fly ash and silica fume." *Applied Sciences (Switzerland)*, 11 (2): 1–18. <https://doi.org/10.3390/app11020830>.
23. Low carbon technology roadmap for Indian Cement Industry, developed by IEA and WBCSD CSI members, technically supported by National Council for Cement and Building Materials (NCB) and Confederation of Indian Industry (CII) and financially supported by International Finance Corp
24. Mithun R, Vasudev V, Pallavi P, 2018, Evaluating Strength Parameters of Triple Blended Concrete Using Composite Cement, *International Journal of Research in Advent Technology*, E-ISSN: 2321-9637
25. Mokal MP, Mandal R, Nayak S, Panda SK. Impact of slag-fly ash cementitious system on thermal controls and durability of high-strength mass concrete. *Structural Concrete*. 2023;24(5):6778–97. <https://doi.org/10.1002/suco.202300197>
26. Nath, P., and P. K. Sarker. 2013. "Effect of mixture proportions on the drying shrinkage and permeation properties of high strength concrete containing class F fly ash." *KSCE Journal of Civil Engineering*, 17 (6): 1437–1445. Springer Science and Business Media LLC. <https://doi.org/10.1007/s12205-013-0487-6>.
27. NT Build 492. 1999. "Concrete, Mortar and Cement-Based Repair Materials: Chloride Migration Coefficient from Non-Steady-state Migration experiments." 1–8.
28. Report on Cement by IBEF, January 2016
29. S.B.Hegde (2014): Secondary cementitious materials in cement and concrete, *World Cement*.
30. S.K. Chaturvedi, D. Yadav, S. Vanguri, V.P. Chatterjee, A. Pahuja and A.K. Sahu, "investigations on composite cement -containing Indian fly ash and granulated blast furnace slag"
31. Salas Montoya, A., L. I. Rodríguez-Barboza, F. Colmenero Fonseca, J. Cárcel-Carrasco, and L. Y. Gómez-Zamorano. 2023. "Composite Cements Using Ground Granulated Blast Furnace Slag, Fly Ash, and Geothermal Silica with Alkali Activation." *Buildings*, 13 (7). <https://doi.org/10.3390/buildings13071854>.
32. Sim, S., J. H. Rhee, J. E. Oh, and G. Kim. 2023. "Enhancing the durability performance of thermally damaged concrete with ground-granulated blast furnace slag and fly ash." *Construction and Building Materials*, 407 (October). <https://doi.org/10.1016/j.conbuildmat.2023.133538>.
33. Venkatachalapathy Venkitasamy, M. Santhanam, B. P. C. Rao, S. Balakrishnan, and A. Kumar. 2023. "Mechanical and durability properties of structural grade heavy weight concrete with fly ash and slag." *Cement and Concrete Composites*, 145: 105362–105362. Elsevier BV. <https://doi.org/10.1016/j.cemconcomp.2023.105362>.

Queries related to the BIS R&D project titled “Study of durability properties of composite cement (a type of hydraulic cement) currently being manufactured by the cement industry in India” (CED 0128) – Continuation to the discussion held over online meeting on July 26, 2024.

Members present during the meeting:

1. Jitendra Kumar Chaudhary, Scientist-B & Assistant Director, CED, BIS.
2. Jayachandran K., Assistant Professor, CED, NIT Calicut.
3. Sharan Kumar Goudar, Assistant Professor, CED, NIT Calicut.

The following queries have been raised and needs clarification based on the work description given in the [Terms of Reference \(TOR\) for the above titled R&D project](#) from the BIS team to execute the project to achieve the objectives. Link for the TOR:

1. **Section 4.b)** To carry out factory visit to the manufacturers of composite cement, identify/collect the information regarding the manufacturing base of the product and the testing facilities available in the country and to learn the sustainability practices adopted by the manufacturer such as energy consumption, use of renewable energy resources, waste management and disposal mechanisms and reduction of carbon footprint.

Query: The cement manufacturers are ready provide the composite cement for the study but they are not ready to reveal the proportion of the SCMs and not IN position to provide the exact proportion required for the project with varying clinker content (35%, 40%, 45% with fly ash ranges between 10 to 35% and rest with granulated slag). Further, the cement industry may not readily accommodate our request for the necessary information on base of the product of composite cements and sustainability practices like waste management, disposal mechanisms, carbon footprint, etc. Will the BIS team/committee will assist in obtaining permission or provide an authorization letter for cement plant visits, collect the cement samples as per the project requirement, and to access the required information from the factory?

2. **Section 4.c)** Visit Government/NABL accredited testing laboratories (one government and one private) to identify the testing infrastructure available in the country.

Query: NIT Calicut have all the testing facilities required for project confirming to the Indian standards and involved in industrial consultancy. Any round robin tests is required or validation study in the external NABL accredited testing laboratories?

3. **Section 4.b.d)** Collect requisite samples of composite cement from the manufacturers and conduct various tests in order to obtain data on various physical, chemical and durability requirements such as RCPT, chloride induced corrosion and carbonation induced corrosion of composite cement.

Query: The term corrosion means, usually the rusting or loss of steel rebar embedded in the cementitious system due to an electrochemical process. The test requirements for the chloride induced corrosion is not given in the document. Suggest a method to conduct the tests or shall to omit it?

4. **Section 5.d.iii)** For each concrete mix, measure carbonation depth at 3% CO₂, at $27 \pm 2^\circ\text{C}$, having 40% - 60% RH

Query: The relative humidity to maintain during the carbonation is mentioned as 40% - 60%. However, in IS 516 (Part 2/Sec 4):2021 specifies that the RH need to be $65 \pm 5\%$ with temperature of $27 \pm 2^\circ\text{C}$, and $3 \pm 0.5\%$ Carbon Dioxide concentration by volume for a period of 70 days unless the duration of the test is not specified. As the duration of testing is not specified, whether to test on 70 days or 120 days as specified in Section 5.e.ii of ToR or to test by 28 days, 56 days, 70 days, and 120 days. For determination of carbonation at multiple exposure periods, prism specimens of size 100 x 100 x 500 mm or 40 x 40 x 160 mm is required instead of 100 mm cubes which used to test at a particular exposure period. Else, the committee may suggest the test protocol.

5. **Section 5.d.i)** For each of the cement sample, three concrete mixes should be prepared using the following water/binder ratio 0.60 (usual M10 Concrete), 0.50 (usual M20 Concrete), 0.45 (usual M25 Concrete), 0.40 (usual M30 Concrete)

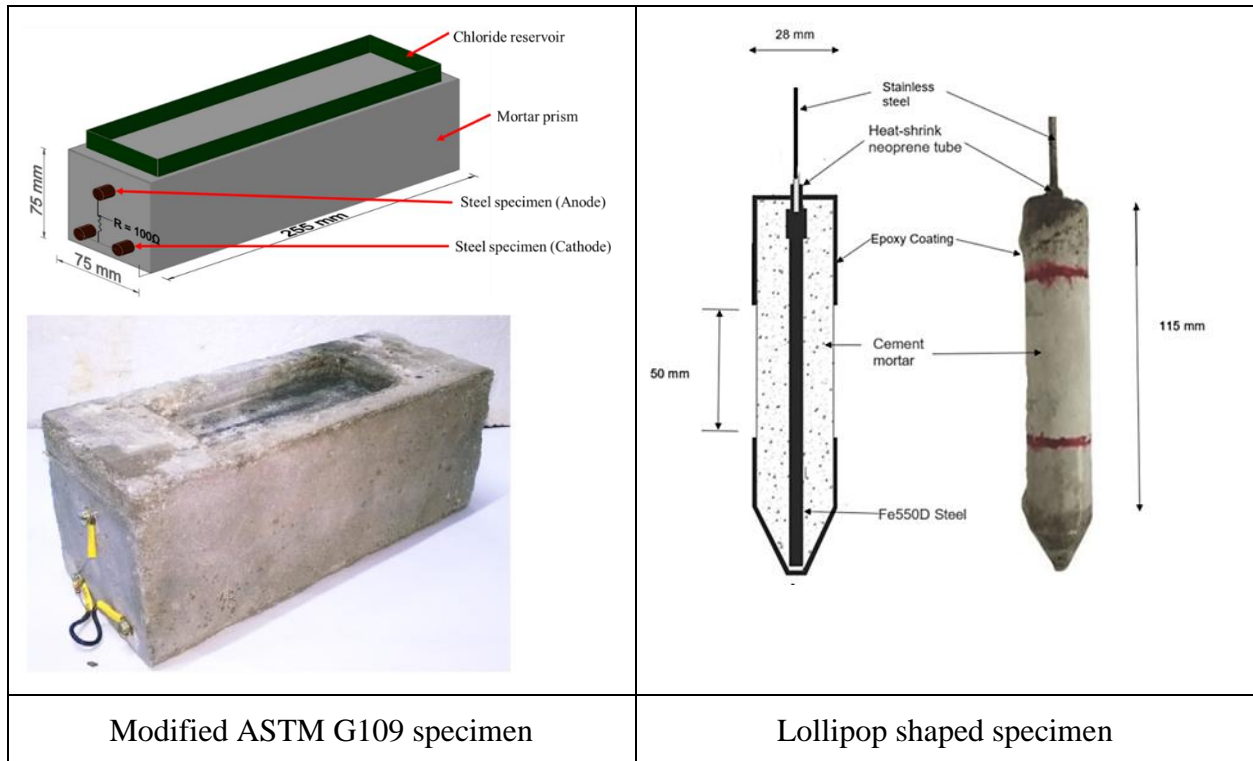
Query: For a typical mix design, the Figure 1 in the IS 10262:2019 will be used to choose the free water-binder ratio for the target compressive strength. For this study, choosing the curve 2 would be an ideal choice or any other suggestions.

6. **Section 5.d.ii)** For each concrete mix, measure 7-, 14-, 28- and 90-days compressive strength.

Query: Does the 56th day compressive strength not required?

7. **Section 5.e.i)** RC beams shall be prepared with 20 mm clear cover for each concrete mix.

Query: The geometry (dimensions, rebar diameter, rebar spacing, etc.) of the RC beam is not clear. Further, large size beams will occupy more space in the carbonation chamber. Thus, the proportionately modified ASTM G109 specimen (75 x 75 x 255 mm prism having 20 mm cover with 1 rebar as anode and 2 rebar as cathode) or the lollipop shaped microcell specimen ($\text{Ø}28$ mm cement mortar cylinder having a single 8 mm rebar with 10 mm cover) is suggested for carbonation induced corrosion in steel rebar embedded in the cementitious system is suggested. Details of the specimens are shown below and the test procedure are explained in the proposal submitted during the application. Both test methods follow a repetitive / series of test to detect the corrosion initiation and the progress of corrosion rate for the specified duration, which may be 3 to 6 months or more.



Calculation for approximate volume for all the specimens and materials required.

Number of binders types from 2 suppliers = 8 = 2 x 4 (with 30%, 40%, 45% clinker & PPC)

Number of strength grades from each binder = 4 (M10, M20, M25, M30 with defined w-b ratio)

Number of concrete mixes for each cement sample = 3

Total number of mixes considering all variables = 8 x 4 x 3 = 96 mixes

Variables	Compressive strength (10 cm cubes)	RCPT (Ø10 cm)	RCMT (Ø 10 cm)	Carbonation depth (4x4x16 cm)	Carbonation induced Corrosion (lollipop microcell specimen or mG109)
Tested on (days)	7, 14, 28, 90	28, 90	28, 90	70, 120	Once in every week till end of corrosion initiation
Specimen required per mix	12 (3x4)	2	2	6 (2x3)	10
Total specimens to cast 96 mixes	1152	192	192	576	960

Section 5.e.iii) Thereafter, the beams shall be immersed 10 mm in potable water for a duration of 14 days

Query: The process of immersing the carbonated sample is for 14 days in potable water after 120 days after carbonation is not available in the standards. Is this process being correct and purposefully specified for any reason? Usually, a wet dry cycle (2 days wet and 5 days dry) will be followed to accelerate the corrosion in the specimen.

Section 5.e.iv) Thereafter, the rate of corrosion should be measured using corrosion current (I_{corr}) measurements.

Query: Usually, the corrosion initiation or the corrosion rate will be measured by the as corrosion potential (E_{corr}) or corrosion current density (I_{corr}). The active corrosion will be estimated by comparing the significant increase in the E_{corr} or I_{corr} or Total integrated current. As per the ToR, a one-time after 120 days of carbonation, 14 days of immersion in portable water is specified which may not replicate the corrosion behaviour.

8. Will the members of the project will be added to technical committee/ working groups of Cement & Concrete (CED02) of BIS?
9. As the fund allocation under each head is uncertain. Thus, the cost may vary for the consumables, characterization of materials, travel for the materials collection, etc. Thus, strongly requested to make an official order to have a provision to change the heads or use the full fund for the project needs irrespective of the heads.
10. The date of transaction of the 1st instalment of the fund was on June 25, 2024 and it was intimated on July 22, 2024. The total project duration is 6 months, and because of the delay in the intimation of the transaction, 1 months of the given time is lost. Further, as per our institutional procedures a new account was created and the fund was transferred to the separate project account on October 21, 2024 (letter of fund transaction -proof attached in next page). Till this time, I'm not a position to utilize any fund and thus, the project officially started from the above-mentioned date only. Further the cement manufacturers are ready provide the composite cement for the study but they are not ready to reveal the proportion of the SCMs and they are not position to provide the exact proportion required for the project with varying clinker content. The extension is required, as the specimen cast is huge in volume where we do not have enough space in the environmental chambers. Thus, the casting is planned in phases to avoid accumulation of specimens to be tested altogether. Hence, requested to consider the extension of the project to end of May 2025 for the completion of the project.

CED

Office of the Dean (R&C)
NATIONAL INSTITUTE OF TECHNOLOGY CALICUT

No. NITC/DEAN(R&C)/2024-25/12/BIS/CED/JC-376

Dated, 16.10.2024

NOTE TO ACCOUNTS SECTION

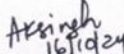
Sub: - Bureau of Indian Standards (BIS) Research Project entitled "Study of durability properties of composite cement (a type of hydraulic cement) currently being manufactured by the cement industry in India" sanctioned to **Dr. Jayachandran K**, Assistant Professor, Department of Civil Engineering - Transfer of fund - Reg.

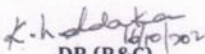
Ref: Sanction order: SCMD/R&D PROJECTS/CED0123 dated:15.07.2024 (Copy enclosed)

Bureau of Indian Standards (BIS) had sanctioned an amount of **Rs. 1,62,150/-** being the first instalment of financial assistance for the implementation of the above said project sanctioned to **Dr. Jayachandran K**, Assistant Professor, Department of Civil Engineering, NITC. Bureau of Indian Standards (BIS) deducted 10% TDS and released an amount of **Rs. 1,45,935/-** & the same is credited to the Institute R&C Account No.35981271500 on 25.06.2024 by NEFT (Copy enclosed).

We have opened a SB account with SBI NITC Branch bearing No. **43388958950** for this project. Kindly make necessary arrangements to transfer the amount of **Rs. 1,45,935/- (Rupees one lakh forty-five thousand nine hundred and thirty-five only)** from the Institute R&C A/c No.35981271500 and credit to the Project A/c No. **43388958950**. A refund bill for **Rs. 1,45,935/-** in this regard is prepared and placed below for further necessary action.


(16/10/24)
JR. ASST


16/10/24
Supdt. (R&C)


16/10/24
DR (R&C)


16/10/24
ASSOC. DEAN (R&C)


21/10/24
DEAN (R&C)

To
Accounts Section with refund bill

Copy to: Dr. Jayachandran K, Assistant Professor, Department of Civil Engineering

