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Design of low carbon high performance concrete incorporating ultrafine materials

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ABSTRACT. In general, high performance concrete (HPC) is associated with high strength and improved durability in comparison to normal strength concrete. However, HPC invariably involves high binder content at low water/binder ratio and its application has been limited to specialised concrete works. In this study, an attempt was made to design high performance concrete, at high water/binder ratio made with OPC content varying from 40%-80% in concrete mixes with low binder content of 280 kg/m3. Binary and quaternary, low carbon mixes were prepared by incorporating Supplementary Cementitious Materials (SCM) and Ultrafine (UF) materials (silica fume, ultrafine GGBS, ultrafine fly ash and metakaolin) and were characterised for strength and durability parameters such as charge passed using RCPT, electrical resistivity and carbonation depth. Findings of the study shows that with appropriate choice and combination of SCM and ultrafine materials, low carbon high performance even at 45% OPC content. Overall, performance of low carbon high performance even at 45% OPC content. Overall, performance of low carbon high performance fly ash and silica fume use along with their compatibility.

Keywords: High performance concrete, ultrafine materials, Supplementary Cementitious Materials carbonation, electrical resistivity, RCPT

1. INTRODUCTION

Usually the term HPC is used synonymously with high strength concrete (HSC), however, HPC has larger spectrum of properties than strength alone. It has been realised that the durability aspects of concrete are equally important. With the idea of HPC, aim of the study was to design a concrete having high strength ensuring more durability in addition to meeting other special requirements of constructability and serviceability. American Concrete Institute (ACI) defines high performance concrete (HPC) as a concrete that fulfils special requirements in performance and homogeneity, which cannot be normally satisfied using conventional materials and mixing, casting and curing procedures[1]. In the last few decades, generous work has been done in the field of HPC and several guidelines such as ACI-318 [2] and fib Bulletin 42 [3] have also been formulated. In India IS 10262:2019 provides guidelines for design of high strength and high performance concrete.

In contrast to normal concrete, researchers have essentially used low water to binder ratio, high binder content incorporating Supplementary Cementitious Materials (SCM) and Ultrafine materials (UF) like microsilica, metakaolin, ultrafine slag etc., in addition to use of high range water reducing admixtures for the development of HPC[4]–[19]. SCMs like fly ash and Ground Granulated Blast furnace Slag (GGBS) have been well exploited in mid-range strength concrete in terms of binary blend and ternary blends with OPC. Ultrafine

materials consist of particles having size in the range of nanometres and thus, have very high specific surface area. These materials are generally finer than cement and SCMs which help in improving the packing density thus reducing the permeability of concrete, besides pozzolanic or cementitious properties help in later stage strength development. But when it comes to developing high strength or high performance concrete further correction in the packing deficiencies is required. Thus, the appropriate combination and proportioning of SCMs and ultrafine materials is necessary to get the desired performance.

Addition of mineral admixtures, such as fly ash and GGBS, in concrete, has proven to be advantageous by many ways. Fly ash particles are finer than cement and help fill up the voids which increases the packing density thus making the concrete less permeable.

Tafraoui [20] studied the application of Metakaolin (MK) instead of Silica fume (SF) in production of UHPC to overcome the high cost associated with SF and the lack of availability, as well as the availability of kaolinite in most countries. MK was utilized as substitution of 20% of the total binder. Study highlighted that weight by weight replacement of SF with MK gave an equivalent mechanical performance for UHPC. However, the mixing time was increased slightly. Nadiger et al. [21] investigated effect replacing cement with SF or ultrafine GGBS. Replacement level for each material was kept in range of 0% to 15%. GGBS was utilized for replacing SF for its economical availability. Results showed that GGBS had same characteristics for SF in terms of mixing and workability but the strength was decreased by 5.2% compared to that of SF replacement. Apart from this cylinder compressive strength, split tensile strength and flexural strength were increased by 40–50% after addition of GGBS compared to control mix.

FA and GGBS shows [22-25] improvement in fluidity of UHPC (as compared to SF), which leads to reduction in the superplasticizer demand and also improves the compactness. In addition, the pozzolanic reaction of reactive silica and alumina in fly ash helps increase in strength and durability by producing C-S-H and C-A-H. In addition, the RCPT values are comparatively very low as compared to concrete made with only Ordinary Portland Cement(OPC) [22][23][24]. It has also been reported that chloride binding capacity of concrete increases with addition of fly ash [25][26]. Electrical resistivity of concrete incorporation SCMs is also higher as compared to concrete made with only Ordinary Portland Cement (OPC)[27]. The lower value of RCPT, higher chloride binding capacity and higher electrical resistivity indicates that due to the addition of SCM's, the resistance against chloride induced corrosion increases. In case of carbonation, it is evident from the literature that the addition of SCMs and ultrafine material improve denseness and reduce permeability thus increasing resistance to ingress of oxygen and moisture (needed for carbonation). However, the addition high volume of SCMs tend to lower the alkalinity and this reduce the resistance to carbonation. Lower water cement ratios and higher grades of concrete can be adopted to improve performance in this case[28], [29].

HPC concrete works demand skilled manpower, better quality control, machinery and various other material and technical requirements. Therefore, the use of HPC has been limited to large infrastructure such as high-rise buildings, bridges, dams etc. Although in general concrete constructions, strength requirement is not as determined as large infrastructure projects, but durability is however a matter of concern. In this study the authors have attempted to design HPC for general use by keeping low binder content (280 kg/m³) and high water to binder (w/b) ratio (fixed at 0.50). Concrete mixes, having various combination and proportions of SCM and/or UF, were designed and characterised for mechanical and durability performance by recognized laboratory test methods.

2. MATERIALS AND METHODOLOGY

2.1. Materials

Commercially available ordinary portland cement (OPC) of 53 grade conforming to the physical and chemical requirements of IS 269 [30] has been used as the main binder in all the concrete mixes. Mineral admixtures viz. fly ash (FA) conforming to requirements of IS 3812 (Part 1) [31] and ground granulated blast furnace slag (GGBS) conforming to requirements of IS 16714 [32] and ultrafine materials (UF) viz. silica fume (SF) confirming to IS 15388 [33], ultrafine FA, ultrafine GGBS conforming to IS 16715 [34] and Metakaolin (MK) conforming to IS 16354 [35] have been incorporated in different combinations and proportions to make various binary and ternary concrete mixes. The physical properties and chemical composition of these materials are given in Table-1 and Table-2: Chemical composition of OPC and mineral admixtures respectively. Coarse aggregates of nominal size 10 mm and 20 mm and fine aggregates. Polycarboxylate ether (PCE) based super-plasticizer conforming to IS 9103 [37] was used to meet the workability requirement of slump between 75-100mm for all the mixes.

| Properties | Cement | FA | GGBS | UFFA | UFGGBS | MK | SF | |
|-------------------------------|--------|------|------|------|--------|------|------|--|
| Fineness (m ² /kg) | 323 | 334 | 400 | 642 | 2026 | 7429 | 1670 | |
| Specific Gravity | 3.15 | 2.19 | 2.93 | 2.28 | 2.88 | 2.91 | 2.28 | |

| Table-1: Physical | properties of OPC | and mineral admixtures | |
|-------------------|-------------------|------------------------|--|
| | | | |

| Properties | OPC 53 | Fly Ash | GGBS | UFFA | UFGGBS | MK | SF |
|--|--------|---------|-------|-------|--------|-------|-------|
| Loss of Ignition (LOI) | 2.30 | 3.64 | 0.33 | 0.39 | 0.17 | 0.60 | 2.73 |
| Silica (SiO ₂) | 20.71 | 62.53 | 34.41 | 54.79 | 3.05 | 52.89 | 85.03 |
| Aluminum oxide (Al ₂ O ₃) | 5.15 | 23.58 | 18.45 | 33.14 | 20.40 | 41.84 | - |
| Calcium oxide (CaO) | 59.96 | 1.17 | 36.46 | 3.21 | 33.14 | 1.03 | - |
| Magnesium oxide, MgO | 4.57 | 0.50 | 7.00 | 0.0 | 7.62 | 0.37 | - |
| Alkalis | | | | | | | |
| Na ₂ O | 0.42 | 1.23 | 0.30 | 0.23 | 0.19 | 0.16 | 0.73 |
| K ₂ O | 0.56 | - | 0.37 | 1.11 | 0.58 | 0.12 | 2.96 |
| Chlorides | 0.012 | - | 0.022 | 0.009 | 0.016 | 0.05 | - |
| Insoluble Residue (IR) | 1.25 | 91.92 | 0.40 | - | 0.86 | - | - |

Table-2: Chemical composition of OPC and mineral admixtures

2.2. **Concrete mix design**

With the purpose of the study to make low carbon HPC, the Total Binder Content (TBC) for all mixes was kept constant at 280 kg/m³ and the water to binder (w/b) ratio was kept fixed at 0.50. At this w/b ratio and TBC of 280 kg/m³, binary and quarterly concrete mixes were prepared by using various combinations and proportions of OPC, FA, GGBS and UF materials. In all 9 mixes were prepared including control mix, the details are given in Table-3. M0 is control mix cast with 100% OPC 53 grade cement as binder, M1-M4 are binary mix consisting of MK, UFFA and UFGGBS and M5-M8 are quaternary mixes containing either FA or GGBS along with combinations of two UF materials like silica fume, UFFA and UFGGBS. Quaternary mixes have OPC content 50% or less which is significantly lower as compared to rest of the mixes. The preparation of mixes and casting of specimen was done at the NCCBM laboratory under controlled environmental conditions (Relative humidity > 65 % and temperature = 27 ± 2 °C). Polycarboxylate Ether Admixtures (PCE) based admixture conforming to IS 9103 was used to maintain workability of 75-100 for each mix. Specimens were cast as per the test requirement and were demoulded after 24 hours and kept in water cuing (27±2 °C) until the test day. Testing of specimens was done in laboratory conditions (temperature $27\pm2^{\circ}$ C and relative humidity $65\pm5\%$)

| Mix Type | Mix Id | OPC | FA | GGBS | UFFA | UFGGBS | MK | SF |
|------------|--------|-----|----|------|------|--------|----|----|
| Control | M0 | 100 | 0 | 0 | 0 | 0 | 0 | 0 |
| Binary | M1 | 85 | 0 | 0 | 0 | 0 | 15 | 0 |
| | M2 | 80 | 0 | 0 | 0 | 0 | 20 | 0 |
| | M3 | 80 | 0 | 0 | 20 | 0 | 0 | 0 |
| | M4 | 92 | 0 | 0 | 0 | 8 | 0 | 0 |
| | M5 | 50 | 25 | 0 | 0 | 5 | 20 | 0 |
| Quaternary | M6 | 40 | 0 | 35 | 0 | 5 | 20 | 0 |
| | M7 | 45 | 30 | 0 | 20 | 0 | 0 | 5 |
| | M8 | 45 | 0 | 45 | 0 | 5 | 0 | 5 |

Table-3: Binder mix proportions

2.3. **Experimental procedures – mechanical test**

Compressive strength test was performed on concrete cubes of 150 mm as per IS 516 (Part-1/Section-1) [38] using 3000 KN capacity compression testing machine. Test was conducted on cube specimens each at age of 7 and 28 days. At the time of testing, it was ensured that the concrete cubes specimens were tested on their moulded sides without any packing between the cube and the steel platens of the testing machine. One of the platens was carried on a base and was self-adjusting, and the load was steadily and uniformly applied, starting from zero at a rate of 14 N/mm²/min.

2.4. Experimental procedure – durability test

- 2.4.1 Rapid chloride penetration test (RCPT) RCPT is a widely used test method to evaluate the durability of concrete. In this test the resistance of concrete towards penetration of chloride ions under the influence of a voltage difference is measured in terms of charge passed (figure-1). Depending upon the amount of charge passed (in coulomb), qualitative rating of concrete's permeability is made. Lesser the charge passed, higher is the resistance of concrete, thus better is the quality of concrete. The test was conducted on three concrete cylindrical concrete discs of diameter 100 mm and thickness 50 mm at 28 and 56 days of age of curing as per ASTM 1202 [39].
- **2.4.2** Electrical Resistivity (Four point Wenner Probe method) (Figure-2) Electrical resistivity which is inverse of conductivity, is an intrinsic property of any material that reflects the ability to resist the transfer of electrical charge through it. On concrete this test is conducted using an equally spaced four-point Wenner Probes. The outer two probes induce current into the concrete and the two inner electrodes measure the resulting potential drop in K Ω -cm. Higher electrical resistance indicates better quality of concrete with respect to durability. The test was conducted at 28 days and 56 days on concrete cubes of 150 mm.



Fig-1 (a): Vacuum box and 1 (b): DC voltage system for RCPT Test



Fig-2:- Sketch of Four -point Wenner Resistivity method



Fig-3: Carbonation Chamber for Accelerated Carbonation Test

2.4.3 Accelerated carbonation test – Carbonation is the process of carbon dioxide entering into the pores of concrete and then reacting with Portlandite in pore water thus, reducing the natural alkalinity of concrete. This makes reinforcement in concrete vulnerable to corrosion in presence of water and oxygen. In laboratory the process of carbonation is accelerated by placing the concrete in a carbonation chamber (Figure-3) which has concentration of carbonation higher in comparison to the atmospheric concentration of CO₂. For this study the accelerated carbonation test was conducted as per ISO 1920 (Part 12) [40] on

concrete prism specimens of size $100 \times 100 \times 500$ mm after 28 days of curing and 28 days of conditioning in laboratory environment. After laboratory conditioning, side faces were sealed using epoxy mainly araldite and carbonation was allowed on the cast and shutter face. After the sealing, the concrete beams were shifted to the carbonation Chamber with CO₂ (4±0.5) %, temperature $27\pm2^{\circ}$ C and relative humidity $65\pm5\%$. The carbonation depth was measured by cutting a slice of 50 mm from the beam and exposing the cut surface to 1% phenolphthalein solution. The concrete specimens were exposed to carbon dioxide for the exposure upto 280 days.

3. RESULTS AND DISCUSSION

3.1. Compressive strength

3.1.1. Effect of UF materials on 28-day strength

Compressive strength results at 7-day and 28-day age are shown in Figure 4. The results are the average for three specimens per mix, obtained by dividing the load sustained by the specimen per unit area. The 28-day strength of binary concrete mixes containing only ultrafine materials (such as M1, M2, M3 and M4) was found in the range of 52-56 MPa and is more than 30% higher in comparison to control mix. Among all the binary concrete mixes, M1 mix containing 15% metakaolin gave higher strength at 7-day as well as 28-day in comparison to other binary mixes. This shows that metakaolin as ultrafine material performed better in comparison to ultrafine fly ash and ultrafine GGBS with respect to compressive strength. Indeed, 20 percent replacement of OPC by ultrafine fly ash also produces strength greater than 50 MPa but was slightly lower when compared to that of concrete made with metakaolin and ultrafine GGBS.

All quaternary blends containing 50% or less OPC, except M6, produced higher strength in comparison to control. The 28-day compressive strength of quaternary concrete mixes was found in the range of 35-51 MPa. This large scale difference in the compressive strength value of quaternary blends may be attributed towards the difference in the amount of OPC content as well as combination of ultrafine materials. Although the strength of these mixes is less as compared to that of binary mixes, but given such lower percentage OPC, yet these quaternary blends yielded higher strength as compared to OPC alone. The strength of quaternary concrete mixes also depends upon the synergy of different components with each other. For example, alike M5, M7 and M8, mix M6 is also quaternary mix, but the strength of M6 is much lower when compared to other mixes. Therefore, in order to achieve the best possible results, the combination and proportion of different binders is very important. From the test results, it is quite evident that OPC content less than 45% has detrimental effect on the compressive strength.

From the compressive strength results of quaternary mixes in this study, it can be said that the combination of metakaolin is synergic with fly ash rather than GGBS, while silica fume goes well with fly ash and GGBS. Ultrafine fly ash when added along with fly ash also yielded strength of 47 MPa at 28 days which is about 15% higher when compared to that of control at same age.

Both the binary concrete mixes as well as quaternary concrete mixes have higher strength in comparison to control concrete. This is due to the formation of secondary CSH and CAH that are produced during the pozzalanic reaction. Formation of secondary hydration products leads to densification and makes the concrete stronger. Therefore, it is possible to design concrete mixes containing high percentage of SCMs along with ultrafine materials provided selection of ultrafine materials has been done judiciously.



Fig-4: Compressive strength results

3.1.2. Strength development with age

It is generally believed that for normal concrete about 67 percent of 28-day strength is achieved on 7day. In this study also about 70% percent of the 28-day strength is achieved at 7-day for control mix (see Figure 5). For binary concrete mixes containing Ultrafine materials, this ratio is a bit higher, while for quaternary mixes M5, M7 and M8, this ratio is on much higher side. This shows that incorporation Ultrafine materials along with mineral admixtures like fly ash and GGBS can lead to early age strength development. This may be due to the nucleation effect which might be signification in early age[8], [22], [23], [41], [42].



Fig-5: Strength development with age (7D/28D)

3.2. Rapid Chloride Permeability Test

Rapid Chloride Permeability test (RCPT) method is widely used by researchers for the performance evaluation of the electrical conductance of concrete samples and it provides a rapid indication of their resistance to chloride ion penetration. Low RCPT value indicates lower chloride ingress which means concrete is more durable as it shows higher resistance against penetration of chloride and other external agencies. The results of RCPT obtained for mixes in this study at 28 days and 56 days of curing are given in Figure 6.

ASTM C1202 provides qualitative indications of the chloride ion penetrability based on the measured values from this test method, see Table-4. From Fig, it can be seen that the control mix has yielded the highest

RCPT value and is fairly close to high penetrability class, while in case of binary mixes incorporating metakaolin, ultrafine GGBS and ultrafine fly ash and all the quaternary concrete mixes, the charge passed is in very low category, however, binary concrete mix (M4) with ultrafine GGBS produced RCPT in low category. A slight reduction in the RCPT value had been observed in all concrete mixes with the age with no change in the chloride penetrability class. From the test results, it can be inferred that incorporating ultrafine material can help to reduce chloride ion penetration to a significant extent and able to produce durable concrete for the RCC structures located in chloride laden environment.

| Charge passed (coulombs) | Chloride ion penetrability | | |
|--------------------------|----------------------------|--|--|
| > 4000 | High | | |
| 2000-4000 | Moderate | | |
| 1000-2000 | Low | | |
| 100-1000 | Very low | | |
| <100 | Negligible | | |

Table-4: Chloride ion penetrability based on charge passed (ASTM C1202)



Fig-6: RCPT test results

3.3. Electrical resistivity

Degradation and corrosion of reinforcement in concrete largely depends upon the concrete microstructure, quantum and size pores and pore interconnectivity. Better pore connectivity will lead to stress-free transport aggressive ions in concrete which will lead to concrete degradation. One of the ways to compare the transport mechanism in different concrete mixes is by measuring the electrical resistivity of concrete. Higher electrical resistivity signifies higher resistance of concrete towards passage of ions through it. Electrical resistivity was measured at 28 and 56 days of age using four point Wenner probe the results of which are shown in Figure 7.

With age concrete develops strength and durability on account of hydration of C_3S and C_2S and pozzolanic activity in case of addition of pozzolanic materials, thus electrical resistivity of concrete is bound to increase with age, which is evident in the resistivity results at 56 days for all the mixes. Lowest resistance was obtained for control concrete and the resistance of binary mixes incorporating metakaolin and ultrafine fly ash was more than thrice as compared to control while in case of quaternary concrete mixes resistance was found to be much higher as compared to that of control. Electrical resistivity of quaternary concrete mixes is 2 to 3 times higher than that of binary concrete mixes. The results positively show that use of ultrafine materials independently as well as along with fly ash and GGBS increases the electrical resistance of concrete mixes, Mix M8 (45 % OPC + 45 %GGBS+5 % ultra-fine GGBS +5 % silica fume) had shown highest electrical resistivity value. In case of binary concrete mixes, electrical resistivity of concrete mix containing 20 percentage metakaolin is the highest in comparison to other binary concrete mixes.



Fig-7: Electrical resistivity of concrete mixes

3.4. Accelerated carbonation test

Carbonation is a complex physiochemical process which involves reaction of atmospheric carbon dioxide (CO_2) with calcium rich phases in cement matrix like $Ca(OH)_2$, C-S-H and yet unhydrated C₃S and C₂S. Thus, leading to the formation of CaCO₃. The consumption of Ca(OH)₂ leads to reduction in pH of the pore solution which creates ambient conditions for reinforcement corrosion in presence of water and oxygen[31]. In laboratory the phenomenon of carbonation is accelerated by keeping concrete specimens in carbonation chamber and exposing to CO₂ concentrations much higher than that in atmosphere. The results of carbonation test are shown in Figure-8.

Addition of SCMs and ultrafine materials, reduces the alkalinity that would outweigh the increased resistance against ingress of CO₂ and moisture and hence causes more carbonation as compared to control sample containing only OPC [43]. The depth of carbonation after 280-Day exposure period is least for control concrete M0 (10 mm). Carbonation depths comparable to control concrete were obtained for binary concrete mixes: M1 (containing 15% metakaolin) and M4 (containing 8% percent ultrafine GGBS). A marginal increase in carbonation depth in comparison to control concrete was obtained in case of binary concrete mixes: M3 (containing 20% metakaolin) and M4 (containing 20% ultrafine fly ash). From the test results it can be observed that as the OPC content is replaced beyond 20% by ultrafine materials, resistance against ingress of CO₂ reduces.

In case of quaternary concrete mixes, for 280-day exposure period carbonation depth was found to be more than 1.5-2.0 times than that of control concrete. At the same OPC content i.e. in concrete mix M7 and M8, the carbonation depth for concrete mix M7 (containing fly ash and ultrafine fly ash) is higher as compared to M8 (containing GGBS and ultrafine GGBS). This may be due to the consumption of Portlandite due to higher extent of pozzolanic activity of fly ash as compared to that of GGBS[41][43]. This is also evident in the carbonation depth results for M5 and M6. Although the OPC content is less in M6 than M5, but carbonation depth is lower as compared to M5. It may be noted that M5 and M6 have same combination of ultrafine materials i.e. 5% ultrafine GGBS and 20% metakaolin except for the difference in type and quantity of SCM content (M5 contains 25% fly ash and M6 contains 35% fly ash).



Fig-8: Accelerated carbonation test results

From the results of electrical resistivity and RCPT, it can be observed that low carbon high performance concrete mixes have reduced porosity and discontinuous pore structure. It is widely known that presence of moisture and oxygen is vital for corrosion. Service life design model as proposed by Tuutti [44] for RC structure comprises of two phases; phase-1 corresponds to initiation phase and phase-2 corresponds to propagation phase. The initiation time refers to the ingress of the external aggressive agents like CO₂, chlorides, SO₄ etc. into the cover concrete and the propagation phase is the time taken for evolution of different forms of deterioration[45]. More carbonation depth in low carbon high performance concrete mixes reduces its initiation period as compared to control mixes but the denser and improved microstructure may decrease the rate of corrosion in the propagation phase on account of increased resistance against the ingress of moisture and oxygen. Carbonation induced corrosion in low carbon high performance mixes is a matter of future research.

4. CONCLUSIONS

- On the basis of compressive strength and durability test results, design of low carbon high performance concrete mixes offers a sustainable solution to the ongoing problem of global warming.
- Durability issue related to carbonation is one of the major concerns associated with the low carbon concrete. The depth of carbonation in all low carbon high performance concrete mixes like binary and quaternary is higher in comparison to control concrete. However, higher electrical resistance as well as lower RCPT value suggests reduced porosity and discontinuous pore structure in low carbon high performance concrete mixes. This is possibly due to synergistic effect between SCMs and ultrafine materials.
- Higher carbonation depth in low carbon high performance concrete mixes results into reduced initiation period as compared to control mixes but denser and improved microstructure may decrease the rate of corrosion in propagation phase due to increased resistance against the ingress of moisture and oxygen. Thus, propagation period in case of low carbon high performance concrete mixes may be longer than that of control mixes. This is a subject of further research.
- In this study low carbon high performance concrete mixes with strength up to 50 MPa with improved durability performance even at 45% OPC content was achieved. Overall, performance of low carbon high performance concrete mixes depends on the type and extent of SCM as well ultrafine materials such as metakaolin, ultrafine GGBS, ultrafine fly ash and silica fume use along with their compatibility.

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