Experimental Investigation of Electrochemical Corrosion and Chloride Penetration of Concrete Incorporating Colloidal Nanosilica and Silica fume

Rishav Garg\textsuperscript{2}, Rajni Garg\textsuperscript{1,*}, and Sandeep Singla\textsuperscript{3}
\textsuperscript{1}Rayat Bahra University, Mohali, India
\textsuperscript{2}Galgotias College of Engineering & Technology, Greater Noida, India
\textsuperscript{3}RIMT University, Mandi Gobindgarh, India

ABSTRACT
Enhancement of durability and reduction of maintenance cost of concrete, with the implementation of various approaches, has always been a matter of concern to researchers. The integration of pozzolans as a substitute for cement into the concrete is one of the most desirable technique. Silica fume (SF) and colloidal nanosilica (CS) have received a great deal of interest from researchers with their significant performance in improving the durability of concrete. The synergistic role of the micro and nano-silica particles in improving the main characteristics of cemented materials needs to be investigated. This work aims to examine the utility of partial substitution of cement by SF and CS in binary and ternary blends in the improvement of the durability characteristics linked to resistance for electrochemical corrosion using electrical resistivity and half-cell potential analysis and chloride penetration trough rapid chloride penetration test. Furthermore, the effects of this silica mixture on the compressive strength of concrete under normal and aggressive environment have also been investigated. Based on the maximum compression strength of the concrete, the optimal cement substituent ratios have been obtained as 12% SF and 1.5% CS for binary blends. The optimal CS and SF combination mixing ratios has been obtained as 1.0% and 12% respectively for ternary blends. The ternary blends with substitution of cement by optimal percentage of CS and SF exhibited decreased rate for electrochemical corrosion. The strength and durability studies were found in consistence with the microstructural analysis signifying the beneficiary role of CS and SF in upgrading the performance of concrete.

Keywords: Corrosion, Electrical Resistivity, Half-Cell Potential, Strength, Chloride Penetration

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1. Introduction

Durability of a cementitious structure is the ability to resist against various deterioration processes viz. chemical, weathering, abrasion etc. in aggressive environmental conditions without any adverse effect on the identity, quality & performance of the cementitious structure during its service life [1,2]. Many physical, chemical & electrochemical processes affect the durability of the cement mortar leading to cracking [3]. The responses inside of the cement mortar as it sets and fortifies are hard to control and this is a progressing issue in the construction business [4]. The major issue in the cement mortar at the solidified state is the voids and cracks furthermore, its ensuing issues because of salt silica response, which is a concoction response that causes gaps in the cement matrix. Aside from the above, penetrability of gasses through pores and small scale cracks in the cement, which prompts erosion issue in the fortification of the matrix brings on additional weakening and causes disintegration, and chemical leaching mainly due to the excess calcium hydroxide [5]. The pores present in the matrix of concrete make it vulnerable towards the invasion of ions in the aggressive environment. Recently, many chemical admixtures have been evaluated to play a role as supportive cementitious materials (SCMs) that
change the porous microstructure when added as a substitute for a small portion of cement.

The term “Pozzolan was initially used to cover the mineral admixtures with discovery in between 700-600 BC by Greek masons. These materials are used in cement mortar/concrete to partially replace cement to obtain the required properties of the more economical products along with the comparable performance of the cementitious materials [6]. Since 1930, many developing countries drove attraction for supplementation of cement with pozzolans. Nowadays, the term “Pozzolan is used to include the very fine particle-sized siliceous and/or aluminous materials with little or no cement-like activity, but in presence of water react with calcium hydroxide or lime to form calcium silicate hydrate (C₃S₂H₃ or CSH) gel similar to that of cement hydration [7].

Thus, the basic function of a pozzolan is to induce a silica-lime reaction in the cement matrix resulting in the formation of CSH gel [8]. However, the presence of pozzolans results in the consumption of calcium hydroxide and an enhanced chemical resistance and strength development due to nano-filler effect [9]. This impact prevents entry of water and chloride ions in the reinforced concrete steel structures, thereby, improving the performance of concrete in exposure to deteriorating conditions. The usage of pozzolanic materials have been proven beneficial to affect the structural and durability properties of cementitious materials [10]. The partial substitution of cement reduces the cement demand and hence, the global carbon dioxide emission decreases, leading to sustainable development [11]. Hence the comparison of different pozzolanic materials & their combination with optimized content is relevant for understanding their effect on the performance of concrete. In this context, the research was initiated by selecting micro silica & nano-silica to partially replace cement and study the effect on fresh, hardened, microstructural, and durability properties of concrete.

Corrosion of the reinforced concrete structures presents the biggest threats for the structural stability in reinforced concrete buildings and is a major concern for the construction industry. The corrosion tendency of the concrete frames is strongly affected by the atmospheric temperature, humidity, and ionic conductivity within the concrete matrix. The safeguarding and maintenance of buildings and bridges requires corrosion assessments of the reinforcements [1,2]. Especially, the corrosion of the embedded steel in concrete owes to the electrochemical action involving chloride ions. Chloride binding is of great importance in the prediction of the service life of cementitious structures in an aggressive environment such as seawater or solutions containing NaCl that results in salt weathering [12]. Deterioration of cementitious materials takes place on coming in contact with an aggressive and corrosive solution containing chloride ions mainly due to the reaction of calcium hydroxide with these ions (Equation 1) [13].

\[
\text{Ca(OH)}_2 + 2\text{NaCl} \rightarrow \text{CaCl}_2 + 2\text{NaOH} \tag{1}
\]

This reaction may proceed further leading to the formation of expansive compounds that are also responsible for the destruction of the materials (Equation 2)[13].

\[
\text{CaCl}_2 + \text{Ca(OH)}_2 \xrightarrow{\text{w,H,O}} \text{CaCl}_2\cdot\text{Ca(OH)}_2\cdot\text{H}_2\text{O} \tag{2}
\]

Several studies have examined blocking of chloride ions from interaction with embedded steel by various techniques and many electrochemical techniques are available for getting information regarding corrosion tendency of concrete. Determination of electrical resistivity, half-cell potential and rapid chloride permeability are the most common techniques used for the same. The goal of the present work is to assess the strength and durability of concrete in presence of various percentages of nano-materials as the partial substitute of cement.

| Table 1. Physical and Chemical Characteristics of OPC, CS and SF |
|---------------------------------------------------------------|
| Property          | OPC | CS  | SF  |
| Specific Gravity  | 3.14| 1.31| 2.2 |
| Specific Surface (m²/g)| 0.31| 140 | 28  |
| Composition (%)   |     |     |     |
| CaO              | 62.2| -   | 0.1 |
| SiO₂             | 20.82| 99.9| 98  |
| Al₂O₃            | 5.2 | -   | -   |
| FeO              | 3.35| -   | -   |
| MgO              | 2.65| -   | 0.1 |
| TiO₂             | 0.27| -   | -   |
| L.O.I            | 3.09| 2.8 | <2  |
2. Experimental

Ordinary Portland cement (OPC) meeting the IS 516, river sand, coarse aggregates, CS (average particle size 40 nm), and SF (average particle size 20 μm) were used. The physical and chemical characteristics of CS and SF have been shown in Table 1. Polycarboxylate based superplasticizer (SP) was used to ensure workability. On a whole, nine mixtures were designed using the mixing design technique as per IS 516 and are summarized in Table 2.

The specimens were prepared at a water-binder ratio of 0.32, while 0-2.0% of CS and 0-16% SF were added to replace cement by weight. The optimization study was carried out for the optimum percentage of CS and SF in binary blends. Subsequently, the optimum percentage of CS and SF for ternary blends was analyzed at the constant water-binder ratio. The components were mechanically mixed and poured into moulds that were left at room temperature for 24 h. Proper hydration of specimens were ensured by curing in a water bath maintained at room temperature [14].

The compressive tests were performed at various curing ages of 28, 56, 90 and 180 days in accordance with IS 516[15]. Rapid chloride penetration test was performed as per method specified in ASTM C1202 at 28, 56, 90, and 180 days [13]. The set up for rapid chloride penetration test using 3.0% NaCl solution and 0.3 M NaOH solution as electrolytes has been represented in Fig. 1. Table 3 shows the criteria for rating of the concrete using the total charge passed. Microstructure analysis was carried out using Philips XL20 Scanning Electron Microscope.
Steel reinforcement bar (150 mm length and 10 mm diameter) was embedded into mould and the reinforcement concrete specimens were prepared and cured before exposing to corrosive environment. Half-cell potential measurements were carried out after curing in accordance with ASTM C876 [16]. The set up for half-cell potential measurement has been represented in Fig. 2. Cu/CuSO$_4$ electrode was used as a reference electrode. The probability of corrosion was evaluated using the criteria given in Table 4.

The electrical resistivity, $\rho$ ($\text{K}\Omega \text{cm}$) of the concrete specimens was determined from the resistance, $R$ ($\Omega$) of the concrete specimens with surface area, $A$ ($\text{cm}^2$) and length, $l$ (cm), using the equation 3 [17].

$$\rho = \frac{R \times A}{l} \quad (3)$$

The rate of corrosion was estimated by the criteria given in Table 5 and the set-up has been represented in Fig. 3.

### Table 4. Criteria for Probability of Corrosion

| Half-cell Potential (mV) | Probability of Corrosion |
|--------------------------|-------------------------|
| <-350                    | More than 90%           |
| -200 to -350             | Uncertain               |
| >-200                    | Less than 10%           |

### Table 5. Criteria for Rate of Corrosion

| Electrical Resistivity (K$\Omega$ cm) | Rate of Corrosion |
|--------------------------------------|-------------------|
| <5                                   | Very High         |
| 5-10                                 | High              |
| 10-20                                | Moderate          |
| >20                                  | Low               |

3. Results and Discussion

3.1 Compressive Strength

The impact of micro and nano-particles on the compressive strength of concrete with the percentage of SF and CS has been shown in Figs. 4-6.

Compressive strength for samples SF1, SF2, SF3 and SF4 with the inclusion of SF and CS1, CS2, CS3 and CS4 with the inclusion of CS was higher relative to that of the control mix (CM) regardless of substitution percentage (Fig. 4). In comparison to CM, an increase in strength values of 5.0%, 11%, 18% and 13% at 28 days, 3.0%, 9.0%, 17% and 14% at 56 days, 3.0%, 8.0%, 17% and 15% at 90 days and 3.0%, 10.0%, 16% and 13% at 180 days was observed respectively for SF1, SF2, SF3 and SF4 specimens. An increase in strength values of 10%, 18%, 24%, 20% at 28 days, 11%, 17%, 22% and 21% at 56 days, 9.0%, 19%, 23% and 20% at 90 days and 12%, 20%, 25% and 22% at 180 days respectively was observed for CS1, CS2, CS3 and CS4 speci-
men’s comparative to CM. Thus, CS improved the compressive strength of concrete in a better way as compared to SF. It has been documented that CS and SF have high pozzolanic potential [18,19]. Ultra-pure silica particles are the main components of CS and SF that act not only as a chemical filler but also as nucleation sites for the cement hydration that produces C-S-H gel. As a result, the increased C-S-H gel production improves the pore structure of the matrix resulting in higher strength [20].

The maximum compressive strength was observed when the specimen contained 1.5% of CS (CS3) or 12% SF (SF3). Nevertheless, with the increasing content of 1.5-2.0% CS and 12-16% SF, the compressive strength was found to decrease. The results are consistent as already reported in the literature specifying the increase in compressive strength up to an optimum dosage of additives and/or substituents [21,22]. The results for compressive strength analysis of specimens at 28, 56, 90, and 180 days have been shown in Fig. 5. The enhancement in compressive strength with an increase in the extent of curing as observed for all the specimens. The pozzolanic behavior of CS is more pronounced at an early age as compared to that of SF due to ultra-fine silica particles [23]. Thus, the substitution by CS tended to increase the compressive strength better than SF even at an early curing age. It was also observed that the substitution by CS and SF gives best results only at optimum substitution percentage as the compressive strength decreases later on with increasing concentrations of the substituent [9]. Taking into account for higher compressive strength when 12% of SF was used, the impact of CS substitution was analyzed for further studies at a fixed content of 12% SF as represented in Fig. 6. In this case, the synergistic effect of CS and SF was observed as the maximum compressive strength was observed at 1.0% of CS in the pres-
ence of 12% SF. The increase in strength values as compared to CM was 27%, 35%, 31% and 30% at 28 days, 25%, 31%, 27% and 27% at 56 days, 25%, 30%, 27% and 26% at 90 days and 27%, 32%, 30%, 29% at 180 days respectively was observed for TC1, TC2, TC3 and TC4 specimens. This result can be attributed to the improved homogeneity of the matrix by simultaneous filling of the unoccupied voids by the micro and nano-silica particles [11]. Thus, the optimized use of CS in the presence of SF can produce better results.

3.2 Electrochemical Corrosion Probability Test

The probability of electrochemical corrosion was determined using half-cell potential test. An increase in half-cell potential (hcp) values and hence decreased corrosion probability of CM, SF1, SF2, SF3 and SF4 concrete specimens with an increase in curing age was observed due to strength development obvious from compressive strength studies (Fig. 7). In comparison to CM, an increase in hcp values of 15%, 28%, 32% and 29% at 28 days, 16%, 29%, 34% and 31% at 56 days, 3.0%, 18%, 21% and 19% at 90 days and 5.0%, 13%, 16% and 14% at 180 days was observed respectively for SF1, SF2, SF3 and SF4. Best results have been obtained for SF3 with 12% SF.

The conventional concrete has highly porous structure that results in depassivation of the reinforcing steel bar due to invasion of chloride ions. Further, interaction with the dissolved oxygen in the pore solution initiates the electrochemical reaction of corrosion. The increase in hcp values indicates the decreased permeability for these specimens for the corroding ions and gases [17]. The microparticles not only fill the pores but also participate in the pozzolanic reaction resulting in occupation of pores by CSH gel. In addition, the reaction of the chloride ions with
these hydration products limits their concentration and controls the probability of corrosion [24]. However, at increased dosage of SF up to 16% results in mutual friction of the microparticles and results in slightly increasing the chances of formation of weak interfacial zones leading to progression of electrochemical corrosion reaction [25].

The use of CS was found to result in decreased probability of corrosion of the reinforced concrete specimens. An increase in hcp values of 53%, 58%, 69%, 64% at 28 days, 55%, 61%, 71% and 62% at 56 days, 47%, 50%, 64% and 57% at 90 days and 48%, 51%, 62% and 55% at 180 days respectively was observed for CS1, CS2, CS3 and CS4 specimen comparative to CM. The results can be ascribed to the nano-filler and pozzolanic effect of CS nanoparticles resulting in densification of the matrix and reduced corrosion probability [26]. However, at higher content of CS up to 1.25% CS (Fig. 8) results in agglomeration in the matrix causing weak zones in the matrix leading to increased corrosion rate [16].

Corrosion rate of TC1, TC2, TC2, and TC4 specimens was found to decrease with curing age as shown in Fig. 9 due to increased densification of the matrix. The hcp values of the specimens >-200 mV at late curing age indicate the very low corrosion rate as compared to CM [17]. The cooperative filler effect of SF microparticles in the presence of CS nanoparticles fills the pores in a better way (Fig. 9). The increase in hcp values as compared to CM was 71%, 77%, 75% and 73% at 28 days, 70%, 79%, 76% and 75% at 56 days, 65%, 74%, 72% and 71% at 90 days and 65%, 75%, 70%, 69% at 180 days respectively was observed for TC1, TC2, TC3 and TC4 specimens as shown in Fig. 6. The densified microstructure at the optimized content of SF and CS results in enhanced
corrosion resistance of the reinforced concrete specimens [27].

3.3 Electrical Resistivity Test

All the specimens were comparatively analyzed for electrical resistivity at the carbonation ages of 28, 56, 90 & 180 days. The determined values have been plotted in Figs. 10-12. The electrical resistivity for CM, SF1, SF2, SF3, and SF4 specimens was observed to increase with increasing curing age. The observation is in correlation with the reduction in the pore structure of the cement matrix with CSH development at increased curing age [28]. Lesser the porosity of the cement mortar, lesser is the ionic strength in the pore matrix and hence, lesser is the conductivity (Fig. 10). Electrical resistivity of specimens with SF was found to be higher in comparison to that of CM with an increase of 18%, 35%, 49%, 42% at 28 days, 11%, 17%, 30% and 27% at 56 days, 9.0%, 14%, 27% and 22% at 90 days and 12%, 25%, 38% and 31% at 180 days respectively for SF1, SF2, SF3 and SF4 specimens.

SF3 specimen exhibited the maximum electrical resistivity due to least conductivity among these specimens. Electrical conductivity is a function of ionic strength of the solution present in pores of the matrix. SF particles consume calcium hydroxide in pozzolanic reaction and improve the pore structure by extra CSH gel formation. Hence, the ionic strength decreases leading to a reduction in the conductivity [29]. The high value of electrical resistivity indicate low rate of corrosion in presence of SF [30]. When compared to CM, the CS1, CS2, CS3, and CS4 specimens recorded an increase in electrical resistivity as 66%, 72%, 87%, 80% at 28 days, 44%, 55%, 75% and 67% at 56 days, 40%, 51%, 68%, and 60%
at 90 days and 48%, 52%, 58% and 51% at 180 days respectively. Maximum electrical resistivity was obtained for CS3 specimens in consistence with strength analysis (Fig. 11). An increase in electrical resistivity with curing age was observed CS1, CS2, CS3, and CS4 specimens in consistence with earlier trends as shown in Fig. 11. Further, the electrical resistivity of these specimens was found to be least in comparison to CM as well as the specimens with individual SF or CS at the respective curing ages. This indicates that these specimens have very low rate of electrochemical corrosion. The results support the strength studies for the improvement of pore structure due to better pozzolanic action of CS as compared to SF leading to increased resistivity [31].

When compared with CM, the TC1, TC2, TC3, and TC4 specimens exhibited an increase in electrical resistivity of 89%, 98%, 94%, 91% at 28 days, 78%, 89%, 86% and 84% at 56 days, 82%, 96%, 86%, and 84% at 90 days and 85%, 92%, 78% and 76% at 180 days respectively. The results are much better than for CM and the specimens with SF and strengthen the fact of improved pozzolanic activity of micro silica particles in the presence of nano-silica particles. The electrical resistivity of TC2 specimens was found to be highest indicating the enhanced durability of these specimens towards electrochemical corrosion (Fig. 12). A marginal decrease in the values may be due to the agglomerate formation of nano-silica particles and friction among the micro silica particles at higher content and destruction of homogeneity of the cement matrix. However, the difference is not significant to support the statement [28].

3.4 Rapid Chloride Penetration Test

A decrease in RCPT values and hence chloride ion permeability of CM, SF1, SF2, SF3 and SF4 concrete specimens with an increase in curing age was
observed due to strength development obvious from compressive strength studies (Fig. 13).

Best results have been obtained for SF3 with 12% SF. In comparison to CM, a decrease in RCPT values of 7.0%, 11%, 20% and 15% at 28 days, 5.0%, 10%, 14% and 13% at 56 days, 5.0%, 9.0%, 15% and 12% at 90 days and 12%, 15%, 21% and 18% at 180 days was observed respectively for SF1, SF2, SF3 and SF4. The decrease in RCPT values indicates the decreased chloride ion permeability for these specimens [32]. Permeability of the cement matrix is related to the extent of hydration, pore structure, and pore continuity. The micro silica particles increase the hydration of the cement matrix and the pozzolanic reaction of SF particles yields additional CSH gel that not only improves the cement matrix but also results in discontinuity of the pores leading to decrease in permeability [33]. A marginal increase in permeability of SF4 specimens with 16% SF can be due to friction among the microparticles at a higher content [13].

The better performance of specimens with CS as compared to specimens with SF was observed well correlated with the strength studies (Fig. 14). A decrease in RCPT values of 17%, 18%, 20%, 19% at 28 days, 13%, 17%, 17% and 17% at 56 days, 13%, 16%, 17% and 16% at 90 days and 19%, 20%, 22% and 21% at 180 days respectively was observed for CS1, CS2, CS3 and CS4 specimen comparative to CM. The decrease in RCPT values with an increase in CS content is again consistent with strength studies attributed to the filler effect of CS particles and pozzolanic action that improves the structure [11]. A marginal increase in RCPT values of CS4 specimens with 1.25% CS can be due to the formation of agglomerates of nano-silica particles at higher content as observed in earlier studies [9].

The total charge passed through TC1, TC2, TC2,
and TC4 specimens was observed to decrease with curing age as shown in Fig. 15 and the trend has been as per expectation. The RCPT values of the specimens below 1000 Coulombs at late curing age indicate the very low permeability as compared to CM. Thus, the pozzolanic activity of SF gets enhanced in the presence of CS. The RCPT values decreased up to content substitution by 10% SF in the case of the TC2 specimens and increased slightly afterward at all the curing ages (Fig. 15). The decrease in RCPT values as compared to CM was 20%, 22%, 21% and 20% at 28 days, 18%, 22%, 21% and 20% at 56 days, 18%, 20%, 19% and 19% at 90 days and 23%, 25%, 24%, 24% at 180 days respectively was observed for TC1, TC2, TC3 and TC4 specimens. The earlier decrease in RCPT is linked to the better pozzolanic action of micro silica particles at the optimized content of nano-silica particles, while the slight increase may be due to the deterioration in pore structure by agglomeration tendency of CS particles [21]. The study reveals the best performance of TC2 specimens at all the studied curing ages with a maximum decrease in RCPT values as compared to CM as well as specimens with individual SF or CS as shown in Fig. 15.

3.5 SEM Analysis

Fig. 16 shows the micrographs for CM, SF3, CS3 and TC2 concrete specimens at 180 days of curing. The various phases in the microstructure of the matrix can be characterized into crystals of calcium hydroxide, needles of ettringite, sheets & flakes of CSH gel [18]. Crystals of ettringite and calcium hydroxide are visible in the matrix of CM specimen along with many pores and scanty flakes of CSH gel leading to heterogeneity (Fig. 16a). This heteroge-

Fig. 16. Micrographs of a) CM b) SF3 c) CS3 and d) TC2 specimens.
neous and porous microstructure of CM is responsible for poor performance observed in compressive results [13]. Fig. 16b shows the micrograph of SF3 with higher compressive strength than CM. This micrograph exhibits comparatively denser matrix with improved pore structure due to the presence of CSH gel flakes. The increased compressive strength of this specimen is due to the decreased amount of strength reducing calcium hydroxide and ettringite needles in the matrix having comparatively lesser number of pores [34]. Fig. 16c illustrates the formation of additional CSH gel due to the enhanced pozzolanic reaction in the matrix of CS3 specimen. The morphology is more homogeneous as compared to CM as well as SF3 specimens, showing the existence of dense CSH gel along with scantly pores. Fig. 16d represents the micrograph of TC2 specimen with highest compressive strength among the investigated specimens. The microstructure of TC2 specimen is better than the former specimens in terms of better packed structure with dense matrix occupied by CSH gel [9]. The improved microstructure leads to enhanced strength as well as durability properties of TC2 with cooperative effect of micro and nano-silica particles in consistence with the high carbonation resistance and RCPT analysis [13].

4. Conclusions

The study has been performed to determine the strength and durability properties of concrete containing CS and SF in binary and ternary blends. The analysis was done on the basis of compressive strength, carbonation, and chloride permeability testing that were correlated with microstructural studies. The following conclusions can be drawn on the basis of the results obtained:

1. The substitution of cement by CS and SF significantly affected the hardened properties of concrete specimens.

2. CS significantly improved the compressive strength in the early age as compared to the SF. Highest increase in compressive strength was observed for CS3 followed by SF3 specimen in binary blends. On the whole, TC2 provided the highest compressive strength among all specimens.

3. The inclusion of CS and SF has a significant effect on the durability of concrete. For binary blends, highest resistance for electrochemical corrosion and chloride penetration was observed for CS3 among CS specimens and SF3 among SF specimens. In case of ternary blends, TC2 provided the best results signifying the cooperative role of CS and SF in improving the resistance properties of concrete.

4. CS3, SF3 and TC2 specimens were observed to provide the higher electrical resistivity, with the highest electrical resistivity for TC2.

Thus, optimal use of SF and NF can be used to tailor the hardened as well as durability properties of concrete for sustainable construction.

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