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Experimental study on strength and microstructure of mortar in presence of micro and nano-silica

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ABSTRACT

Sustainable construction practices require cementitious materials with high strength that is strongly dependent upon the nature of binding materials and pore structure. The physico-chemical properties of these materials can be tailored suitably by preferential substitution of cement by materials having comparatively small particle size resulting in improved pore structure. This study is aimed to investigate strength and microstructure of the preferentially substituted cement mortars with incorporation of microsilica (MS), nanosilica (NS) and their combined use at 3, 7 and 28 days of curing. The substituent MS (5.0–20%) and NS (0.5–1.25%) have been used at a water binder ratio of 0.5. The specimens were analyzed for the fresh (consistency, setting time, flow) and hardened (compressive and split tensile strength) properties and a correlation between compressive and split tensile strength was obtained. Mortar containing NS was found to develop better strength as compared to the mortar containing MS. The optimum usage of MS with incorporation of NS was further found to increase the strength of mortar significantly. SEM-EDX was used for the analysis of the microstructure of the specimens and the correlation between Ca and Si content was used to analyze the cement matrix. The findings show that the optimized usage of micro and nano silica can give beneficial effects to improve the fresh properties as well as strength with dense microstructure.

Selection and peer-review under responsibility of the scientific committee of the International Conference on Advanced Materials Behavior and Characterization.

1. Introduction

Cementitious materials are the composite materials produced from Portland cement with paramount importance in all the construction fields due to their extensive applications. Day-by Day increasing world population and tremendous technological & industrial advancement leading to massive infrastructure requirement has further increased the demand of cement [1]. The production of cement and fine aggregates is not only highly energy intensive, but also pose a threat to the environment due to release of harmful pollutants including particulates and greenhouse gases [2]. As per estimation, cement clinker production is one of the main sources of global pollution generating greenhouse gases (GHGs) with identification of construction industry as one of the major contributors of GHGs emissions. With the ever-increasing demand of cement, the emission is expected to become more significant [3]. Further, strength, durability and maintenance cost of construction material are another important issue of prime concern. Sustainable development demands new techniques that must involve control and reduction of the GHG emissions and energy use along with substitution of cement by supportive cementitious materials (SCM) such as Pozzolans [4]. The conventional pozzolans include volcanic ash [5], diatomaceous earth [6], silica fume [7], bottom ash [8] and fly ash [9]. However, with tremendous growth in nanotechnology, various nanomaterials such as carbon nano tubes [10], nano-SiO_2 [11–14], nano-TiO_2 [15,16], nano-Al_2O_3 [17,18], nano-metakaolin [2,19], nano-CuO [20] and nano-Fe_2O_3 [1,21] have also been explored recently. These materials are used in cement mortar/concrete to preferentially replace cement so as to obtain the required properties of the more economical products along with the better performance of the cementitious materials.

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Due to finer particle size, nanoparticles possess high pozzolanic activity and also act as filler, resulting in homogeneous, dense and compact microstructure. It drastically improves the physico-chemical and mechanical behaviour of the cement structures [22]. The ultra-fine nanoparticles invade the voids present in the cement matrix resulting in refinement of the pore size, reduction of the pore volume and disconnection of the capillary mechanism of the pores. Consequently, there is a decrease in water permeability capacity of cement matrix along with simultaneous increase in the chemical resistance of the cementitious structures. In cement mortar, the nanoparticles create a micro dense structure which leads to extremely compact cement mortars and improves the mechanical characteristics of the cement mortar [23]. Literature reports better results for the systems with incorporation of nanoparticles in presence of microsilica. Micro silica can exhibit both cementitious and pozzolanic behaviour and is quite worthwhile in amending the fresh and hardened behaviour of cement mortars and concrete [24]. The nucleation effect of nanoparticles facilitates hydration reaction involving formation of CSH gel with the development of microstructure accounting for closing of cracks. The fresh properties such as setting time also gets lessened due to extensive reaction of nano silica particles resulting in early hardening [25].

The fresh and hardened properties of mortar not only depend upon the application techniques, water/binder ratio but also upon the nature, concentration and particle size of the binder and additive particles. Nanosilica can be used either as dry powder or in the colloidal form [26]. Colloidal nanosilica is in aqueous suspension form that consists of amorphous hydroxylated silica nanoparticles with particle size lying in the range of 1–500 nm. The particles of colloidal nanosilica show lesser segregation and better dispersion in cement mortar and is considered to facilitate the production of CSH gel with high stiffness [27]. Although the reports are available on the individual use of micro silica & nano silica to modify the behaviour of the cement mortar yet not much data is available on the use of amorphous silica nanoparticles on behaviour of cement mortars. This study is aimed to understand the outcomes of preferential substitution of cement by amorphous nanosilica nanoparticles in reference to fresh, hardened and micro structural properties of cement mortar in presence and absence of microsilica and to design the statistical model for prediction & validation of the various studied properties at the desired curing ages.

1.1. Materials

Grade 43 Ordinary Portland Cement was used for preparation of the samples used in the study. Fine aggregates in the form of Standard Ennore sand and potable water was used to prepare all specimens. Colloidal NS of average particle size 40 nm and microsilica with average particle size 0.20 μm were used with physical properties listed in Tables 1 and 2 respectively. The % content of MS and NS was varied from 5.0% to 20% and 0.5% to 1.25% respectively at a water/binder ratio of 0.5 as listed in Table 3. The fresh properties viz consistency, initial and final setting time of the cement were determined confirming to IS 4031-2019 part 4 and 5 respectively [28,29]. Flow of mortar specimens was determined by using Flow table as per IS: 5512-1983 [30]. Compressive strength of mortar cubes was determined in accordance to IS 2250-1981 [31] and the split tensile strength was determined in accordance of IS 5816-1999 at 3, 7 and 28 days of curing [32]. The microstructural analysis has been investigated through SEM-EDX studies.

Table 1
Physical characteristics of micro silica.

Property	Value
Average Particle size (μm)	0.20
Bulk density As-produced (kg/m^3)	135–475
Bulk density Slurry (kg/m^3)	1,420–1,540
Bulk density Densified (kg/m^3)	550–700
Specific gravity (g/cm^3)	2.21
Specific Surface (m^2/g)	15–30

Table 2
Physical characteristics of nano silica.

Property	Value
Average Particle size (nm)	40
Density (g/cm^3)	2.40
Molar Mass (g/mol)	59.90
Melting Point (°C)	1610
Boiling Point (°C)	2225
Specific gravity (g/cm^3)	1.31
Specific Surface (m^2/g)	140

2. Result and discussion

2.1. Normal consistency

Cement paste is characterized in terms of Normal Consistency that further deduces the water demand and workability of cement paste. Normal Consistency of controlled mix (CM) paste was examined and then compared with that of the cement pastes with preferential substitution of cement by micro silica and/or nano silica as represented in Fig. 1. The consistency of cement pastes M1, M2, M3 and M4 increased by 8.0%, 14%, 23% and 26% respectively as compared to CM. Further as the percentage of MS increased from 5.0% to 20%, the water required for consistency also increased. An increase in the content of MS particles, with more specific surface, results in more surface area available for interaction with water [26]. It can be observed from Fig. 1 that the water required for consistency in N1, N2, N3 and N4 cement pastes increased in comparison to CM. The consistency of N1, N2, N3 and N4 cement pastes increased by 6.0%, 12%, 13% and 13% respectively as compared to CM. As the percentage of NS was increased in specimens N1, N2, N3 and N4, a gradual increase in consistency was observed. However, the consistency of these cement paste specimens was lesser as compared to specimens with MS. Although, the particle size of NS is lesser as compared to the particles of MS, yet due to much difference in content of MS and NS, the effect of higher content compensates the effect of particle size leading to increased consistency of specimens with MS [33]. The consistency in case of MN1, MN2, MN3 and MN4 cement pastes not only increased as compared to CM but also increased as compared to pastes containing individual MS (M1, M2, M3 and M4) or NS (N1, N2, N3 and N4). It indicates comparatively higher water demand in presence of MS + 1.0% NS. The consistency of MN1, MN2, MN3 and MN4 cement pastes increased by 22%, 25%, 26% and 28% respectively as compared to CM. The increased consistency of these specimens can be related to the availability of micro silica particles with more surface area with increased content of MS. Hence, water demand of binder particles increases due to enhanced interaction of binder particles with water leading to increased consistency [27].

2.2. Setting time

Setting time of CM paste was examined and then compared with that of the cement pastes with preferential substitution of cement by micro silica and/or nano silica. The setting time of M1, M2, M3 and M4 pastes was found to be higher as compared to CM as evident from Fig. 2. The initial setting time of M1, M2, M3 and M4 cement pastes increased by 6.0%, 10%, 16% and 24% respec-

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Table 3
Composition of specimens.

Specimen	Water (mL)	Cement (g)	Sand (g)	W/B	MS (g)	NS (g)
CM	100.00	200.0	600	0.50	–	–
M1	100.00	190.0	600	0.50	10	–
M2	100.00	180.0	600	0.50	20	–
M3	100.00	170.0	600	0.50	30	–
M4	100.00	160.0	600	0.50	40	–
N1	97.670	199.0	600	0.50	–	1.0
N2	96.500	198.5	600	0.50	–	1.5
N3	95.340	198.0	600	0.50	–	2.0
N4	94.175	197.5	600	0.50	–	2.5
MN1	95.340	188.0	600	0.50	10	2.0
MN2	95.340	178.0	600	0.50	20	2.0
MN3	95.340	168.0	600	0.50	30	2.0
MN4	95.340	158.0	600	0.50	40	2.0

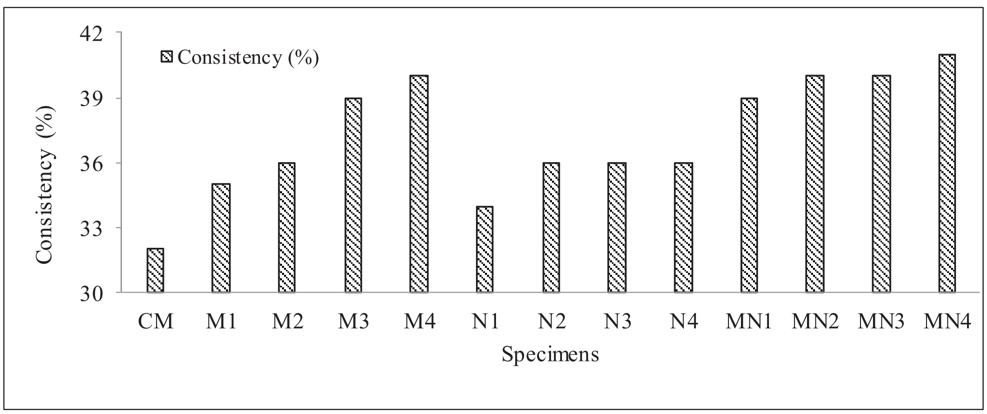

Fig. 1. Consistency of Specimens.

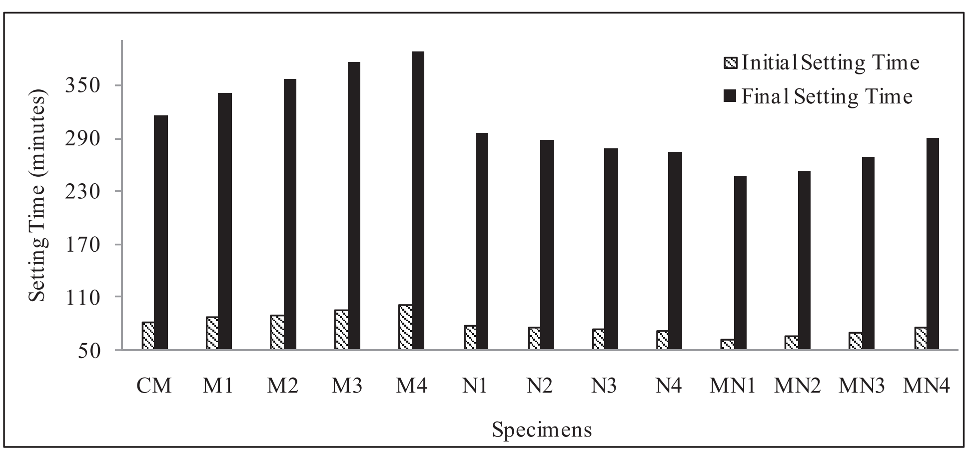

Fig. 2. Setting time of Specimens.

tively as compared to CM. Whereas, the final setting time of M1, M2, M3 and M4 cement pastes increased by 8.0%, 13%, 19% and 23% respectively as compared to CM. It is quite obvious that as the content of MS with finer micro particles increases, the hydration process is further delayed resulting in an increase in the setting time [22]. The initial setting time of N1, N2, N3 and N4 cement pastes decreased by 4.0%, 7.0%, 9.0% and 12% respectively as compared to CM. Whereas, the final setting time of N1, N2, N3 and N4 cement pastes decreased by 6.0%, 9.0%, 12% and 13% respectively as compared to CM. The nano-silica particles are finer as compared to micro-silica and cement particles. As a result, increased content of NS further increases the thickness of the paste due to more specific surface of NS particles in comparison to MS and cement particles [26]. Consequently, there is facilitation of early hydration leading to a decrease in the setting time. The initial setting time decreased significantly as 24%, 18%, 14% and 6.0% as well as final setting time as 22%, 20%, 15% and 8.0% in case of MN1, MN2, MN3 and MN4 cement pastes in comparison to CM

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as well as the cement pastes containing MS or NS due to increased thinning effect of micro silica particles with increase in their content that retards the setting process [25].

2.3. Flow or workability

The flow of CM was found more as compared to the flow of M1, M2, M3 and M4 cement mortars as evident from Fig. 3. Further, flow of mortar decreased with increase in content of MS. The flow of cement pastes M1, M2, M3 and M4 decreased by 3.0%, 4.0%, 8.0% and 11% respectively as compared to CM. The flow of N1, N2, N3 and N4 cement mortars was observed to be lesser as compared to flow of CM as well as M1, M2, M3 and M4 cement mortars with some exceptions due to much higher substitution content of micro silica in comparison to nano silica. The finer nano silica particles introduced better packing due to filler effect and decreased the flow [34]. Further, flow of mortar decreased with increase in content of NS. The flow of cement mortar N1, N2, N3 and N4 decreased by 5.0%, 6.0%, 8.0% and 10% respectively as compared to CM. The increased content of finer particles increased the stiffness of cement matrix leading to decrease in flow. The flow of MN1, MN2, MN3 and MN4 cement mortar with preferential substitution of cement by optimized dosage of MS and NS decreased significantly as compared to CM as well as mortars with preferential substitution of cement by individual MS or NS. The flow of cement pastes MN1, MN2, MN3 and MN4 decreased by 23%, 27%, 33% and 39% respectively as compared to CM. This observation can be attributed to the enhancement in packing of cement matrix because of combined effect of micro and nano silica particles [35].

2.4. Compressive strength

The prime factor responsible for cement mortar is its compressive strength that primarily depends upon the curing age, water/binder ratio, nature and content of the binder material. It can be seen that all the specimens having preferential substitution of cement by MS exhibited increased compressive strength in comparison to that of CM and with increasing curing age as illustrated in Fig. 4. The reason behind this observation is the pozzolanic activity of the micro-silica particles that accelerates the compressive strength [35]. The specimens with 5.0%, 10%, 15% and 20% MS showed an increase of compressive strength by 3.0%, 8.0%, 13% and 11% at 3 days, by 6.0%, 11%, 16% and 13% at 7 days and 9.0%, 15%, 18% and 16% at 28 days respectively. However, this increase was found up to substitution with 5.0% to 15% MS and substitution with 20% MS caused a comparative decrease afterwards. This observation can be due to the friction among the micro-silica particles at higher %age leading to decrease of homogeneity and compressive strength. These specimens with preferential substitution of cement by NS exhibited increased compressive strength in comparison to CM and M1, M2, M3 and M4 specimens in terms of comparative % substitution [26].

The mixes with 0.5%, 0.75%, 1.0% and 1.25% NS showed an increase in compressive strength by 16%, 22%, 26% and 24% at 3 days, by 15%, 21%, 23% and 22% at 7 days and 15%, 19%, 22% and 21% at 28 days respectively in comparison to CM. The observed increase is attributed to the filler effect and pozzolanic behavior of NS that not only increases the packing of the cement matrix but also improves its homogeneity due to production of extra CSH gel. The difference in the developed compressive strength pertains to difference in the pozzolanic response of MS and NS. The pozzolanic activity of a substance depends upon its particle size, packing ability and uniform dispersion of the particles. NS has greater SiO_2 content but smaller particle size than MS. Hence, NS has more uniform dispersion of particles and greater packing ability than MS to provide more filler and is more effective than MS in the pozzolanic reaction with CSH gel [13]. It was observed that among the N1, N2, N3 and N4 cement mortar specimens with substitution by 0.5%, 0.75%, 1.0% and 1.25% NS respectively, the N3 specimen showed maximum compressive strength at 28 days indicating a loss of homogeneity in the cement matrix with increasing content. It has been observed that pozzolanic materials tend to agglomerate at higher content in the cement matrix. NS particles have higher tendency to agglomerate as compared to other pozzolanic materials. In comparison to other binders, NS particles have smaller size and stronger van der Waal's interactions resulting in the formation of agglomerates that disrupts the homogeneity of the cement matrix and decreases the compressive strength [12]. Hence, 1.0% content of NS can be designated as the optimum percentage, used further in mortars with MS + 1.0% NS.

The MN1, MN2, MN3 and MN4 cement mortars with substitution of cement by MS + 1.0% NS showed further enhancement in compressive strength in comparison to that of CM as well as specimens with individual substitution. This observation exhibits the better performance of MS and NS in combination. The NS particles are distributed in the unoccupied voids of MS-cement matrix to increase the homogeneity and the compressive strength of the specimen [23]. Fig. 4 shows that MN1, MN2, MN3 and MN4 specimens developed compressive strength with increase in curing age. When compared with CM, the mixes with 5.0%, 10%, 15% and 20% MS (MN1, MN2, MN3 and MN4) at 1.0% NS showed an increase in compressive strength by 29%, 48%, 41% and 37% at 3 days, by 26%,

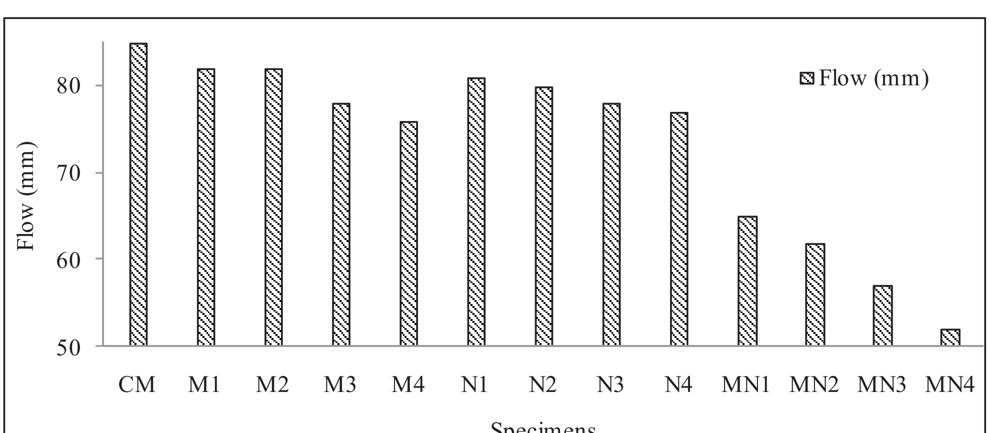

Fig. 3. Flow of Specimens.

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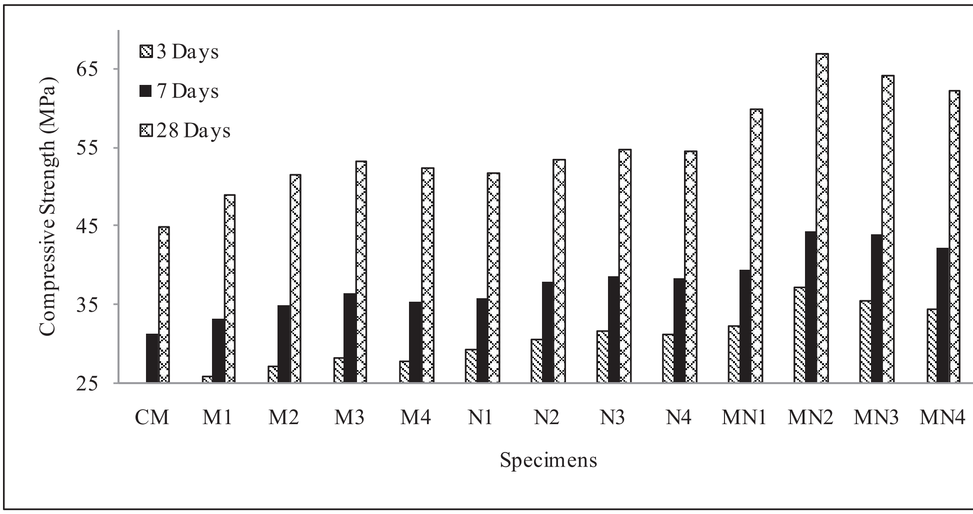

Fig. 4. Compressive Strength of Specimens at 3, 7 and 28 Days.

41%, 40% and 35% at 7 days and 33%, 49%, 43% and 38% at 28 days respectively. It has been observed by other researchers also that higher content of MS or NS alone may not result in enhanced compressive strength of the cement mortars and give better results in presence of other pozzolanic materials [24]. In these cases, the compressive strength was found to increase on substitution of cement by 5.0% to 10% MS, but decreased further at substitution of cement by 15% to 20% of MS at standard curing age. This decrease in compressive strength indicates to the reduction in homogeneity of the cement matrix that can be linked to the two possibilities. First being the non-uniform packing at higher content of MS + 1.0% NS due to increased friction between micro and nano silica particles and second the agglomeration tendency of NS particles at higher content. As a result, the pozzolanic action of micro and nano silica particles decreases leading to decreased densification and strength as reported by other workers [25].

2.5. Split tensile strength

Fig. 5 represents the trends for gain in split tensile strength of CM, M1, M2, M3 and M4 mortar specimens with curing age as expected. When compared to CM, an increase in split tensile strength of 4.0%, 6.0%, 9.0% and 8.0% at 3 days, 4.0%, 7.0%, 10% and 10% at 7 days, 7.0%, 9.0%, 11% and 10% at 28 days, was observed for M1, M2, M3 and M4 respectively. In these specimens, the maximum split tensile strength was observed for M3 specimens with 15% MS indicating the development of cement matrix. The N1, N2, N3 and N4 specimens also developed better split tensile strength as compared to CM, M1, M2, M3 and M4 specimens in terms of percentage content of substitution from 0.5% to 1.0% and then decreased marginally afterwards. The results further support the better performance of nano silica with better pozzolanic activity as compared to micro silica. For N1, N2, N3 and N4 specimens, the increase was observed as 12%, 14%, 19% and 15% at 3 days, 20%, 22%, 23% and 21% at 7 days and 20%, 19%, 21% and 19% at 28 days respectively as compared to CM. The results indicate the extra generation of CSH gel due to nucleation effect and increase of homogeneity of cement matrix due to filler effect of nano silica particles leading to enhanced tensile strength [22].

Tensile strength development trend for MN1, MN2, MN3 and MN4 mortar specimens was similar to the trends obtained for CM and specimens with individual MS or NS as shown in Fig. 5,

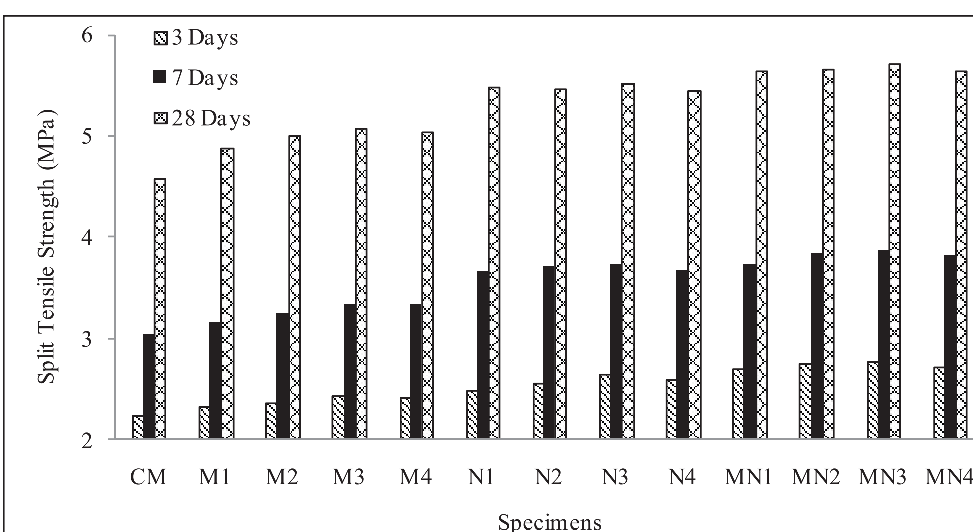

Fig. 5. Split Tensile Strength of Specimens at 3, 7 and 28 Days.

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but the increase was not significant at standard and later curing ages. However, comparatively a remarkable increase supports the better performance of MS particles in presence of optimum dosage of NS particles (1.0% NS). Best results were obtained for MN3 specimens in consistence with compressive strength analysis with an initial increase followed by a slight decrease. In comparison to CM, the increase in split tensile strength for MN1, MN2, MN3 and MN4 respectively was 21%, 23%, 24% and 22% at 3 days, 22%, 26%, 27% and 25% at 7 days and 23%, 24%, 25% and 23% at 28 days. The results again support the better effect of MS at an appropriate content and in presence of optimized content of NS [24].

2.6. Correlational studies between compressive and split tensile strength

Statistical analysis has been carried out to find correlation between the two strength properties at the studied curing ages for all the mixes to represent the two properties, compressive strength (X) and split tensile strength (Y) as per the equation (1) and plotted in Fig. 6 indicating a good correlation at early age with high value of R^2.

$$Y = a_o + a_1X + a_2X^2 \quad (1)$$

Where, a_o; $a_1$and a_2 are the coefficients.

The value of Correlation Coefficient (R^2) has been obtained as 0.9795, 0.8895 and 0.8402 at 3, 7 and 28 days respectively indicating the good validation of the results.

2.7. SEM-EDX analysis

Figs. 7–10 represent SEM micrographs of cement mortars, without substitution of cement, with substitution by MS, with substitution by NS and with substitution by MS at 1.0% NS respectively. The micrographs of all the specimens can be distinguished into six phases on the basis of morphology. The darkest phase can be identified as the pores, the brightest phases as the unreacted anhydrous cement grains and the less dark needles and hexagonal plates can be identified as CH. The small needle like portions can be identified as ettringite and the massive fibroid/crown like phase as CSH gel [33]. For CM, a heterogeneous matrix with many pores along with CSH crystals interconnected with CH plates were observed. The micrographs of cement mortars with substitution by MS appeared comparatively more compact as compared to CM in support of gain of compressive strength while the micrographs of cement mortars with substitution by NS were found to represent more compact, dense and homogeneous microstructures in comparison to that of CM as well as the specimens with preferential substitution of cement by MS [24]. The microstructures of all specimens were fur-

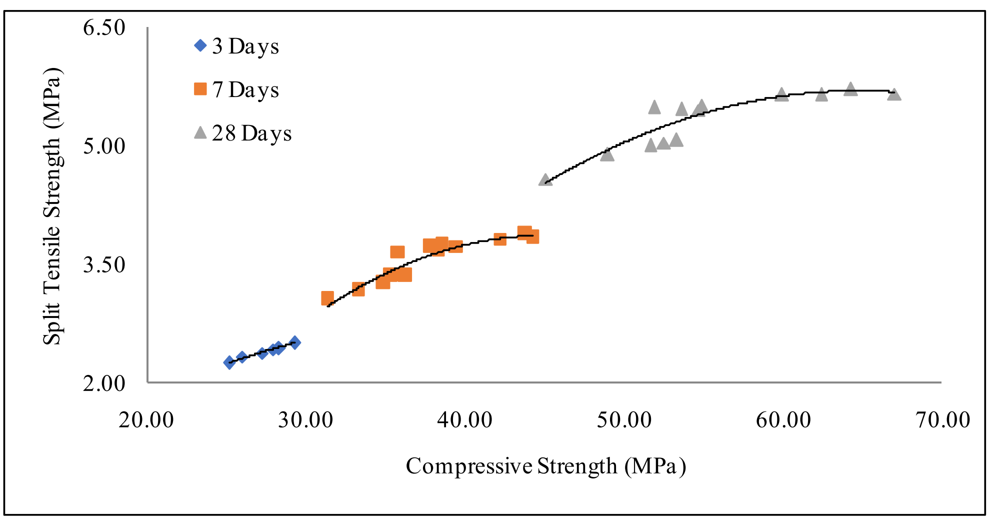

Fig. 6. Correlation analysis at 3, 7 and 28 Days.

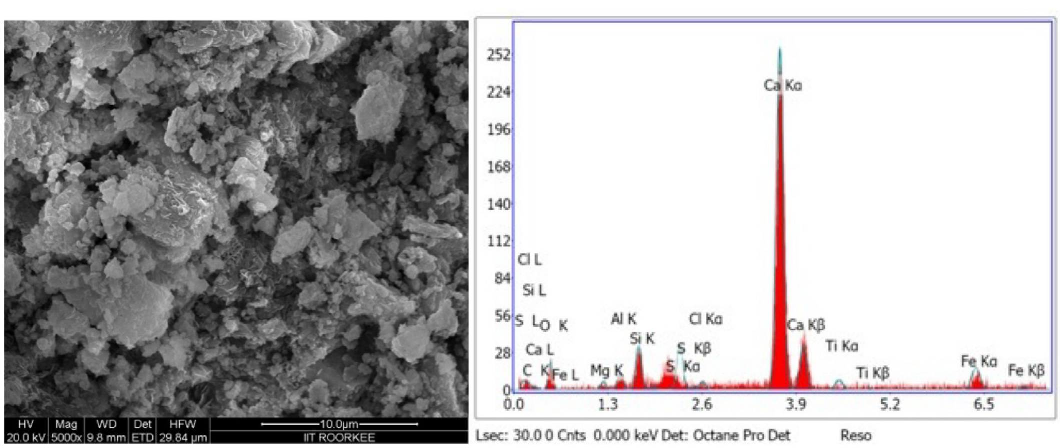

Fig. 7. SEM-EDX Micrographs of CM specimen at 28 Days.

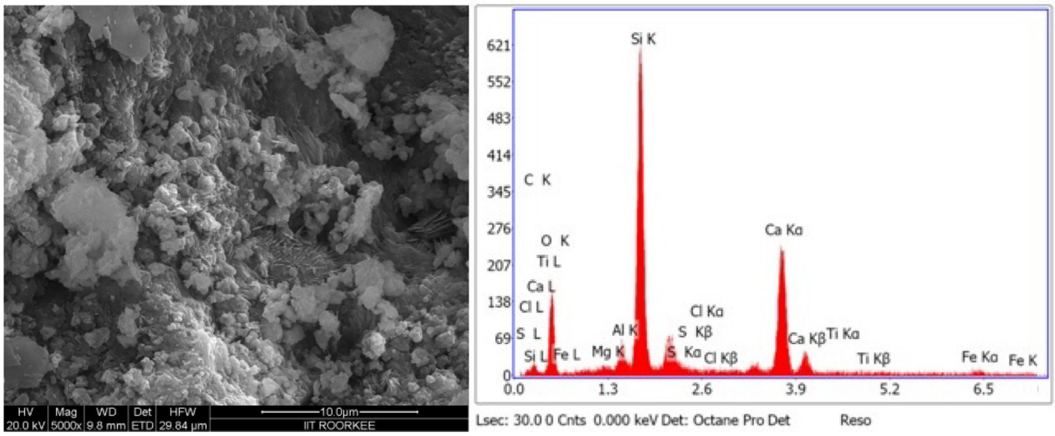

Fig. 8. SEM-EDX Micrographs of M3 specimen at 28 Days.

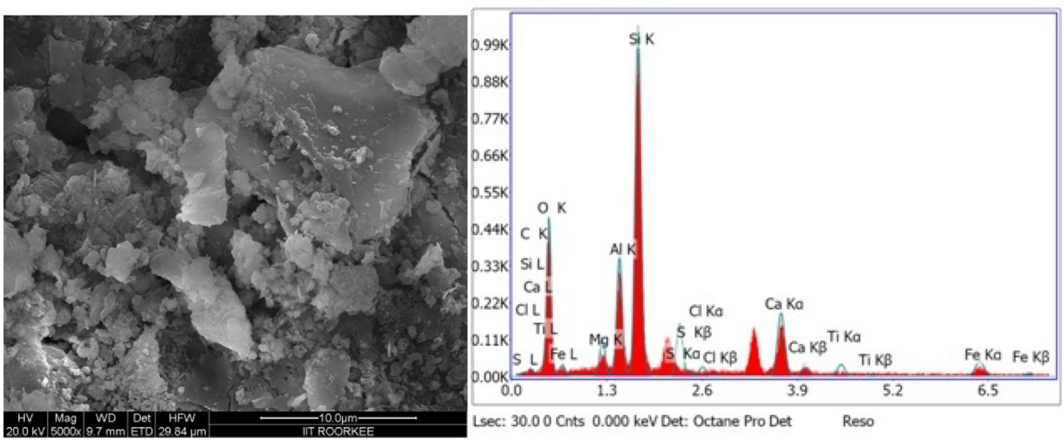

Fig. 9. SEM-EDX Micrographs of N3 specimen at 28 Days.

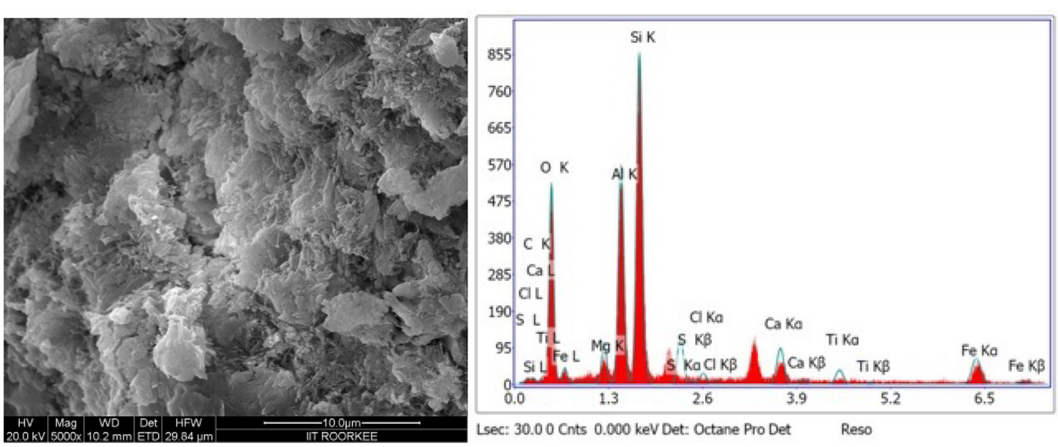

Fig. 10. SEM-EDX Micrographs of MN2 specimen at 28 Days.

ther found to develop with curing age. The stoichiometric Ca/Si ratio is a measure of the crystals in various regions of the specimens and is determined as the ratio of the atomic % of the Ca and Si as given by EDX analysis. The decrease in this ratio indicates the development of CSH phase with decrease in content of CH while the increase indicates the excess of CH with decrease of pozzolanic reaction [12]. The value of Ca/Si ratio gradually decreased with increase in content of MS from 5.0% to 15% in support of increase in pozzolanic reaction. The very low Ca/Si ratio in M3 specimens with 15% MS confirmed the increased strength in these specimens at 28 days. However, a slight increase in Ca/Si ratio was obtained for M4 specimens with preferential substitution

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of cement by 20% MS. The analysis supports the slight decrease in strength of M4 specimens as compared to M3 specimen [7].

However, the ratio still indicates the presence of congruent CSH gel with tobermorite like phases. Likewise, the value of Ca/Si ratio was found to reduce with increase in curing age and content from 0.5% to 1.0% NS in confirmation to increase in pozzolanic activity of nano silica with increase in content. However, the N4 specimen exhibited a slight increase in Ca/Si ratio indicating the slight decrease in microstructure development at all the studied curing ages. The microstructure of these specimens represented the existence of agglomerates of nano silica at higher content of NS in consistence with reports available in literature [12]. As a result, the pozzolanic activity of NS decreases causing a decrease in compactness and strength of the cement mortar. In case of mortars with substitution by MS at 1.0% NS, a significant decrease in the value of Ca/Si ratio of the specimens were found with increasing curing age and increasing MS content from 5.0% to 10%. The very low Ca/Si ratio in MN2 and MN3 specimens with 10% and 15% MS + 1% NS confirmed the increased strength in these specimens at 28 days. The value also supports the highly compact and homogeneous microstructures of these specimens as observed in the micrographs. However, the ratio was found to increase slightly in case of MN3 and MN4 specimens with 15% and 20% MS indicating the decrease of pozzolanic activity at increased content of MS as discussed earlier in mechanical strength studies. Further, the slight variation in the Ca/Si ratio in these specimens indicates the difference in the composition of the CSH content. The reason behind this observation may be the onset of heterogeneous reaction taking place on the particle surface and agglomeration at higher content of micro silica and nano silica particles [13].

3. Conclusion

The study of effect of preferential substitution of cement by MS and/or NS on fresh as well as hardened properties & microstructure of cement mortars in comparison to control mix has been carried out. The consistency, initial and final setting time of the specimens were found to increase while the flow was found to decrease in content of MS as well as that of NS but the effect was more pronounced in case of ternary blends. An increase in compressive and split tensile strength was obtained with increasing content of MS and NS from 5.0% to 15% and from 0.5% to 1.0% respectively along with a slight reduction in strength afterwards. The results were confirmed with SEM-EDX analysis with profound decrease in Ca/Si ratio accordingly. This decrease in strength was because of the reduction in homogeneity of the cement matrix at higher content of MS while the decrease in strength at higher content of NS was attributed to the agglomeration of nano particles at higher content in the cement matrix. SEM-EDX analysis confirmed the loss of pozzolanic activity of MS and NS at higher content due to agglomeration and friction among particles.

CRediT authorship contribution statement

Rishav Garg: . : Conceptualization, Methodology, Data curation, Investigation, Resources, Writing - original draft. **Rajni Garg:** Formal analysis, Software, Writing - review & editing. **Manjeet Bansal:** Project administration, Validation. **Yogesh Aggarwal:** Supervision, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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