CHARACTERIZATION STUDY ON THE SYNERGISTIC EFFECT OF NANO METAKAOLIN AND EXPANSIVE AGENT ON THE SHRINKAGE MITIGATION AND STRENGTH ENHANCEMENT OF SELF CURING- SELF COMPACTING CONCRETE

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Insufficient and ineffective curing may decrease the strength of self compacting concrete forming high shrinkage cracks and voids. The shrinkage due to evaporation is the main reason for the deterioration of concrete structures. The use of internal curing agents in combination with expansive agents can solve the problems of shrinkage and self desiccation caused due to self curing and has the potential for achieving predefined properties. The present research works aims at the production of self compacting concrete with higher strength and durability by utilizing GGBS, alccofine and nano metakaolin as cement admixture along with copper slag as partial substitute for fine aggregate. Several investigations were carried out to analyze the influence of using MgO based compounds as expansive agents in the concrete on the mechanical, fresh state and durability tests. The results show that addition of expansive agent provided desirable strength values and durability properties in the self compacting concretes. The use of highly free flowing concrete that are self compacting in nature with high strength and dimensional stability is also achieved due to the synergistic effect of expansive agents and mineral admixtures in the concrete.

Keywords: *self compacting concrete, nano metakaolin, alccofine, copper slag, expansive agent, self curing agent, strength, durability*

1. Introduction

Self compacting concretes possess special ability to flow through its self weight and hence being widely used in the repair and rehabilitation works [1]. The use of repair work agents should be of good strength and should not deteriorate due to insufficient hydration. SCC that proves to be eco friendly, productive and cost effective when compared to the conventional concrete mix [2]. The essential ingredients of the concrete marks cement as its major component and the production of cement involve a huge labour and cost [3]. The fine aggregate which is another essential component of concrete is also facing on ever increasing demand and exploitation [4, 5]. These two facts always remind the engineers and researchers to develop a concrete that can partially or completely mitigate the above said problems. Copper slag is one such a hazardous industrial waste that can replace fine aggregate without affecting the strength of the concrete [6-8]. The silica fume and metakaolin are the commonly used mineral admixtures in concretes [9-14]. Wastes from the industries when used in concrete not only prove to be beneficial from the economic consideration but also from the ecological point of view [15,16]. The use of industrial wastes and by products can thus produce sustainable concrete with increase strength results [17]. Hence the study tries to develop a self compacting concrete which contains all the three admixtures namely silica fume and metakaolin as cement replacement and copper slag as fine aggregate replacement.

One of the most commonly used industrial byproduct in concrete industry is copper slag. Successful results have been obtained by using copper slag as fine aggregate by several researchers. Number of works reported on the utilization of copper slag as fine aggregates in normal concrete [18]. The strength and workability of concretes were found to increase due to copper slag as natural sand substitution. The strength and durability of concrete was increased and the maximum increment was observed at 40% substitution rate of copper slag. Little of the literature is available on the usage of copper slag in self compacting concrete [19] and the workability of copper slag incorporated self compacting concretes were acceptable as per EFNARC guidelines with minimal segregation and bleeding [20]. The incorporation of copper slag in concrete also remarkably enhanced the shrinkage resistance of the concrete [21].

Nano metakaolin can act as fillers in concrete and can also function as nucleus for inducing hydration in concrete. The nano metakaolin can make the structure of concrete highly dense by reducing the porosity and also promoting the early hydration of cementitious materials. Self compacting concrete suffer from volume deformation and micro cracking due to the moisture consumption during hydration especially when metakaolin is added to the concrete excess shrinkage occurs [22]. Hence effective use of expansive agent is required to mitigate both autogenous and drying shrinkage thereby preventing the internal and external moisture loss of the concrete.

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Curing is essential for the improvement of strength and attainment of stability of the chemical components of the concrete [23]. One of the major challenges faced by the construction industry during the production of concrete is curing. The adoption of best type of curing is essential for any concrete. In areas where water scarcity is a major problem, the adoption of water curing poses a severe issue. The attainment of maximum strength in concrete is possible only when they are subjected to normal curing. In situations where normal curing is not possible proves a new methodology to be introduced for curing by using suitable external admixtures or agents [24,25]. The self curing agents can effectively hold moisture thereby functioning as internal curing agent [26]. The use of expansive agents in self compacting concretes can reduce the formations of cracks formed during hydration process [27-29]. Shrinkage cracks are more common in concretes with certain admixtures like metakaolin and silica fume [30,31]. Also workability is the main criterion that affects the strength and durability of a self compacting concrete and hence the use of low W/C ratio with high amounts of superplasticizer can also cause cracks due relatively low volume hydration products [32]. The reduced quantity of water may result in the requirement of use of agents that can hold the moisture and release when hydration progresses to attain high strength [33,34]. The self curing agents are expected to perform this job well and thus can effectively hold up to moisture for completion of hydration.

Followed by the various study initiated through the domains of SCC no sufficient studies are available on expansive self curing concretes and the simultaneous usage of both the self curing agents and expansive agent in the self compacting mixture remains unexplored. Hence the novelty of the present study lies on the essentials of studying the effect of SCC containing mineral admixtures using different internal curing agent and expansive agents. The main objective of the production of the self compacting concretes using expansive agents in combination with mineral admixtures is to establish a deeper understanding of the SCC containing different proportions of expansive agents in modifying the strength properties of SCC.

2. Materials and methodology

Ordinary Portland cement of 53 Grade conforming IS 12269-1987 is used. The properties of the cement are given in Table 1. Natural River bed sand available in the local market and satisfying the standard codal recommendations of IS 383-1989 is used as fine aggregate. Gravel of nominal size 10mm availed locally is used as coarse aggregate. Blast furnace slag (GGBS), Copper slag, alccofine and nano metakaolin used in the study were obtained from the local manufacturers and the chemical composition as identified through ED-XRF technique is presented in Table 1. Commercial grade polyethylene glycol named as PEG 400 self curing agents is used as 1% by the weight of binder's content.

The production of SCC was done as per the procedure stated in BIS methodology and M30 grade self compacting concretes are produced. The mix design for the production self compacting concrete was done as per the EFNARC specifications. The key proportions for the production of concrete mixes were performed based on mass calculations rather than the volume considerations. The water cement ratio was constantly maintained at 0.38 throughout the concrete mixes. The superplasticizer dosage was maintained at 2.5% of the binder content for the concrete mixes. The specimen designations and percentage replacements of the binder and fine aggregate contents with the corresponding levels of additions of self curing and expansive agents are presented in Table 2.

3. Experimental techniques

The workability of the fresh self compacting concrete mixes were investigated using J-ring, L box, slump flow, T50 and V funnel test as per the

Table 1

Chemical Oxide composition of the materials used in concrete

Oxide (%)	Cement	Alccofine	GGBS	Metakaolin	Copper slag
SiO_2	20.85	35.30	35.43	53.75	33.05
Al_2O_3	4.78	21.40	13.00	43.82	2.79
Fe_2O_3	3.51	1.20	0.37	0.45	53.45
CaO	63.06	32.20	40.52	0.16	6.06
MgO	2.32	8.20	8.00	0	1.56
SO_3	2.48	0.13	0.50	0.02	1.89
K_2O	0.55	0	0	0.18	0.61
Na_2O	0.24	0	0	0.26	0.28
TiO_2	0.25	0	0.50	0.86	0
Mn_2O_3	0.05	0	0.61	0	0.06

Table 2

Mix proportion of self compacting concrete mixes

Mix Id	Cement Replacement (kg/m^3)				FA Replacement (kg/m^3)		Coarse Aggregate (kg/m^3)	MgO (EA)
	Cement	Alccofine	GGBS	Metakaolin	Sand	Copper Slag		
REF	630.00	-	-	-	846.00	-	787.00	-
SMk1	535.50	15.75	47.25	31.5	803.70	42.30	787.00	-
SMk2	504.00	15.75	47.25	63.00	761.40	84.60	787.00	-
SMk3	472.50	15.75	47.25	94.50	719.10	126.90	787.00	-
SMk4	441.00	15.75	47.25	126.00	676.80	169.20	787.00	-
SMg1	535.50	15.75	47.25	31.50	634.50	211.50	787.00	31.50
SMg2	504.00	15.75	47.25	63.00	592.20	253.80	787.00	63.00
SMg3	472.50	15.75	47.25	94.50	549.90	296.10	787.00	94.50
SMg4	441.00	15.75	47.25	126.00	507.60	338.40	787.00	126.00

procedure stated in EFNARC 2002. The weight loss of the concrete due to evaporable water content and measurement of non evaporable water content in the concrete specimen were done by burning in muffle furnace at 1050^{0}C as per the methodologies suggested by El-Dieb [35]. The shrinkage measurements were done in rectangular concrete specimens of size 100x100x400 mm using measuring apparatus with micrometer gauge, measuring frame, invar rods as per IS 1199:1959. Using the Compression Testing Machine(CTM) of 2000 kN capacity, the compressive strength of the concrete at various ages of 14, 28 and 90 days were done on cubic specimens of size 150 x 150 x 150 mm confirming to the standard IS 516-1959. The flexural strength of the beams of size 100 x 100 x 500 mm was determined by two point load method at 7, 28, 90 and 180 days as per IS 516-1959. The water absorption, porosity and sorptivity of the 100 mm cube specimens were tested by using 28 days cured specimens meeting the RILEM recommendations. All the specimens were subjected to curing under sealed conditions until the testing is done. Five specimens were cast for each test and the average forms the final result. The test setup for all experiments was shown in Figure 1.

Fig. 1 - Test setup (a) Compressive strength (b) Flexural strength (c) Water absorption and porosity (d) Sorptivity.

4. Results and discussion

4.1. Workability

Figure 2 showed the workability results of self cured self compacting concrete specimens obtained through Slump, T 50, V- funnel, J- Ring and L-Box. The fresh state results showed that all the SCC concrete mixes yielded good workability and thus fulfilled the compacting requirements. The improvements in the workability were due to the use of copper slag that induced the lack of cohesiveness in the concrete thereby increasing the workability. The glassy surface texture of copper slag and their low water absorption characteristics increased the flowing ability of the SCC which were in concomitance with the previously established research works [6, 36]. The use of nano metakaolin generally restricts the fluidity where in the present study the use of copper slag partially restricted the resistance to the flowability. The use of self curing agents can reduce self compacting ability of concrete due to their ability to bond with hydrogen bonds. In addition the higher specific surface area and pozzolanic effect of the blast furnace slag and alccofine also increases the wettable surface area thereby requiring more water which leads to reduction in workability. But in the present study an improvement in the fluidity of concrete is obtained due to the incorporation of copper slag and expansive agent that improves the rheology of the paste material. The nano metakaolin though demanded more water for attaining workability was compensated by the increased copper slag and expansive agent concentration in the mixes that effectively distributed the nano metakaolin in the concrete without causing agglomeration. Moreover the fineness content of the ingredients used in concrete plays a role in improving the workability. Thus the property of agglomeration and

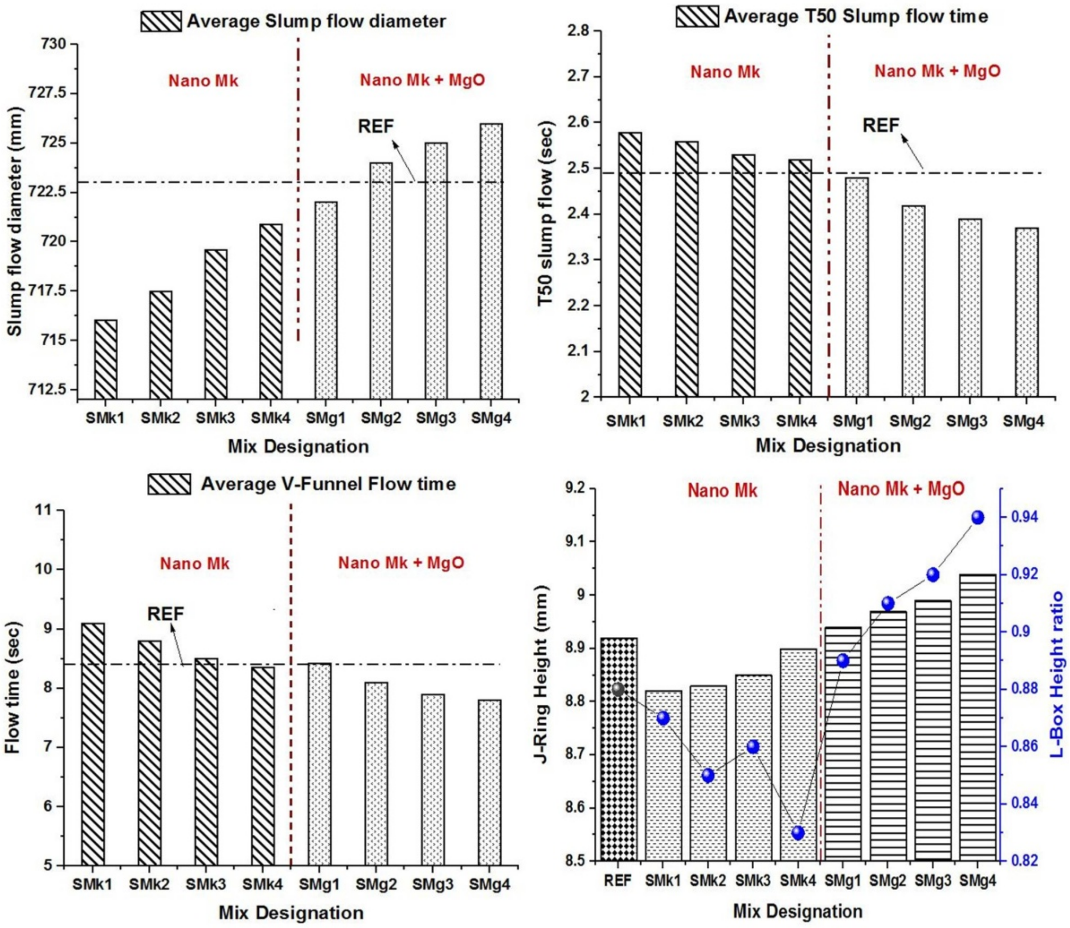

Fig. 2 - Fresh state results of self cured self compacting concrete specimens.

compounding effect of metakaolin and their negative influence on the workability of the concrete was reduced significantly.

4.2. Weight loss

Figure 3 shows the weight loss of the concrete mixes due to the loss of evaporable moisture content in the concrete. The results indicate higher values of weight loss for the mixes containing nano metakaolin when compared to the mixes containing expansive agent. This shows the improvement in the water retention capacity of the self curedself compacting mixes due to the expansive agent addition which results in minimized weight loss. The measurement of the non evaporable water content was performed on unsealed concrete specimens at various ages is presented in Figure 4. The presence of high amount of non evaporable water due to higher water retention capacity of the concrete also results in improved degree of hydration.

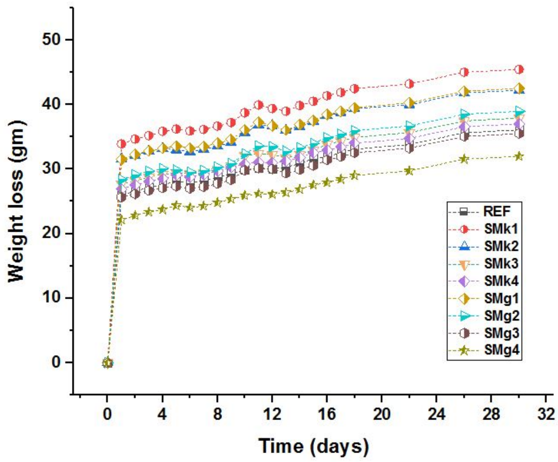

Fig. 3 - Weight loss with time for self cured self compacting concrete specimens.

4.3. Shrinkage behaviour

The results of the shrinkage values of the self cured self compacting concrete specimens are shown in Figure 5. The incorporation of nano metakaolin increased the shrinkage values due to the large amount of water that resulted in significant expansion. Whereas the addition of expansive agent mitigated the shrinkage values by forming expansive components thereby promoting self desiccation and compensate the shrinkage of concrete. The early age shrinkage is also reduced due to the high hydration activity of the MgO based expansive agent. The later age expansion also showed excellent reduction due to the hydration of MgO grains. The MgO based expansive agent further breaks into smaller particles due to the increased expansive stress occurring on the grain

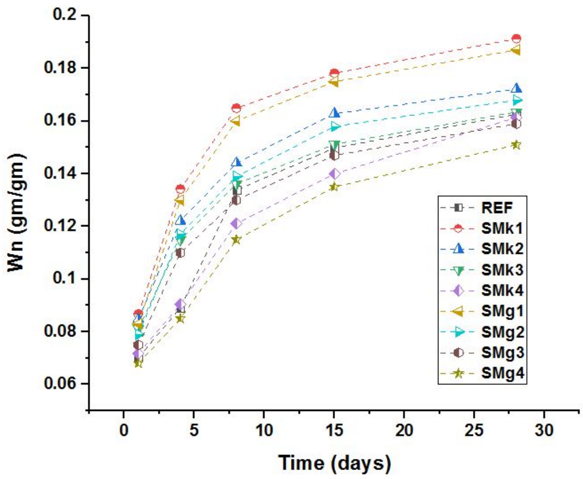

Fig. 4 - Non-evaporable water versus time for self cured self compacting concrete specimens.

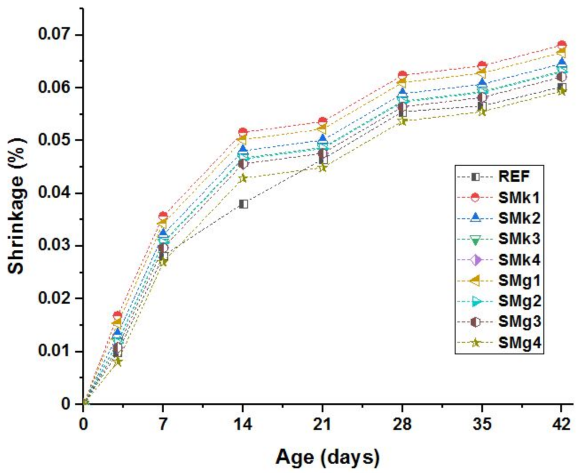

Fig. 5 - Shrinkage behavior of self cured self compacting concrete specimens.

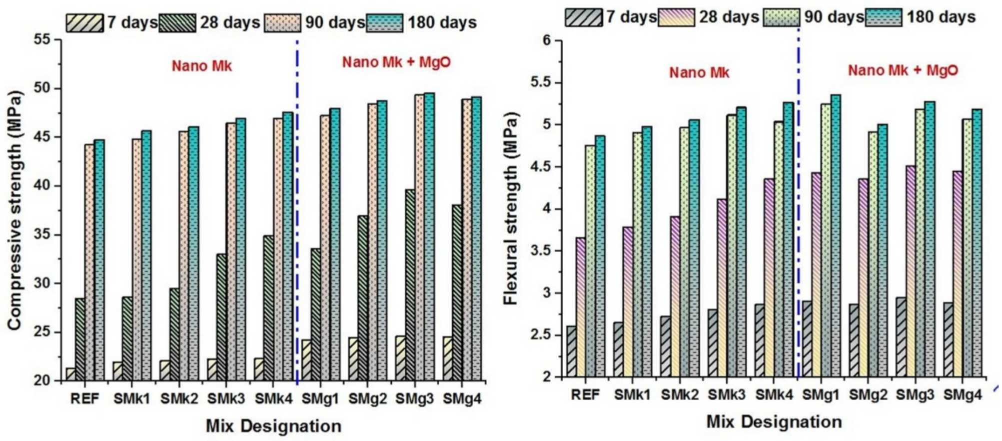

Fig. 6 - Mechanical behavior of self cured self compacting concrete specimens.

boundary thereby increasing the expansion and rate of hydration [37]. Furthermore the reduced expansion of the SCC particularly with expansive agents and nano metakaolin addition indicates the higher moisture consumption of the nano metakaolin which is compensated by the expansive agents that contribute to the decline of self desiccation in the concrete [38]. The shrinkage reduction can also be attributed to the greater volume of hydration products resulting from the replacement of cement by nano metakaolin in combination with expansive agents. The observed smaller expansion values of the SCC specimens were likely an outcome of the synergistic effect of expansive agent and nano metakaolin which caused a subsequent increase in the volume of hydration products thus mitigating the shrinkage effects [39,40].

4.4. Mechanical Strength Properties

The compressive strength of the self compacting concrete containing self curing and expansive agents showed improved performance at all curing ages as shown in Figure 6. MgO based expansive agent added to PEG 400 resulted in a concrete mix having higher strength than the control concrete mix. The combined agents in the mixture exhibited increase in compressive strength on 28, 90 and 180 days of curing ages. Increase in strength was recorded in both the mixtures on 28, 90 and 180 days. The strength improvement due to the combined additions of mineral admixtures and expansive agent in the concrete has been observed in all the ages. The reduction in the plastic shrinkage and minimized evaporation of water from the concrete due to the inclusion of expansive agent also caused an improvement in the strength of the concrete. The expansive agents in combination with GGBS functioned as accelerator and helped in the continued formation of hydration products without causing self desiccation. The formation of pores inside the concrete is also reduced due to the minimized loss of evaporation of waters present inside the concrete thus the strength reduction is minimized to a great extent even when the concrete is not subjected to water curing.

The increase in the flexural strength was observed due to the additions of self curing and expansive agents blended with copper slag and silica fume results in excellent flexural strength, due to the filling effect of self curing agent, due to the filling effect of metakaolin and high fineness of copper slag that has large surface area. This is further enhanced by adding the combined curing and expansive agent that complemented each other. This replacement exhibited improved strength in all ages of 7, 28, 90 and 180 days as shown in Figure 6. This shows the reaction of combined agents and effective participation of curing and expansive agent in the mixture. Progressive increase is also obtained because of the curing and expansive agent proportion increment in the SCC. Thus combined effect of silica fume, metakaolin, copper slag and mixture of curing and expansive agents has resulted in increased flexural strength.

The strength of the concrete increases with appropriate nano metakaolin content and when the quantity of nano metakaolin exceeds the optimum percentage the strength of the concrete is reduced. The decrement in the strength occurs due to the agglomerating tendency of nano metakaolin that gets adsorbed around the cement grains leading to delayed hydration reaction. In addition the inclusion of nano metakaolin beyond certain percentage reduces the contact points which function as binding centre of the cement grains thereby causing ineffective dispersion and weaker interfacial transition zones. But according to the current research the negative effects of nano metakaolin on the strength reduction was compensated by the mix proportion adopted such as increased superplasticizer ratio and the inclusion of expansive agent and copper slag addition. Therefore the research showed enhanced strength than the previous studies reported on nano metakaolin and this was mainly due to the fact that the agglomeration between the nano metakaolin was destroyed by copper slag addition which improved its dispersing ability in the cement matrix. Simultaneously the expansive agent addition provided sufficient shrinkage mitigation property thereby minimizing the crack formation due to the excess addition of nano metakaolin in the concrete. At the same time the combined addition of nano metakaolin with alccofine and GGBS shows the best effect owing to the higher degree of hydration leading to formation of dense layers of hydration products.

4.5. Water absorption

The water absorption values of the self compacting concrete containing self curing and expansive agent in addition to the supplementary cementitious materials are shown in Figure 7.The water absorption values of the self compacting concretes containing expansive agents and nano metakaolin exhibited reduced values due to their viscous nature and pore filling characters. Generally nano metakaolin is hydrophilic in nature and hence tendency to absorb water increases. But the present study showed reduced water

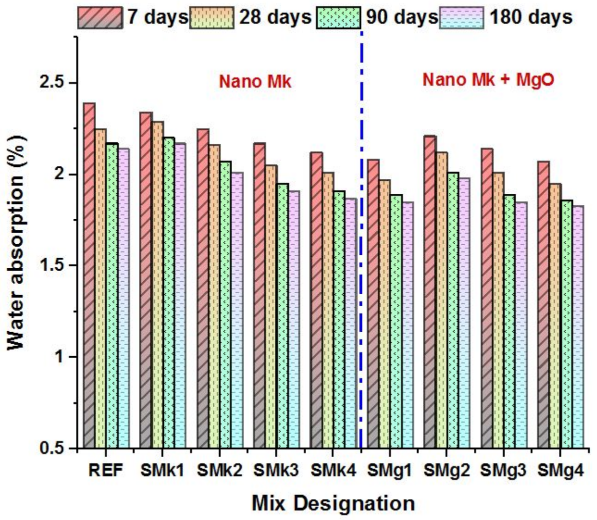

Fig. 7 - Water absorption of self cured self compacting concrete specimens.

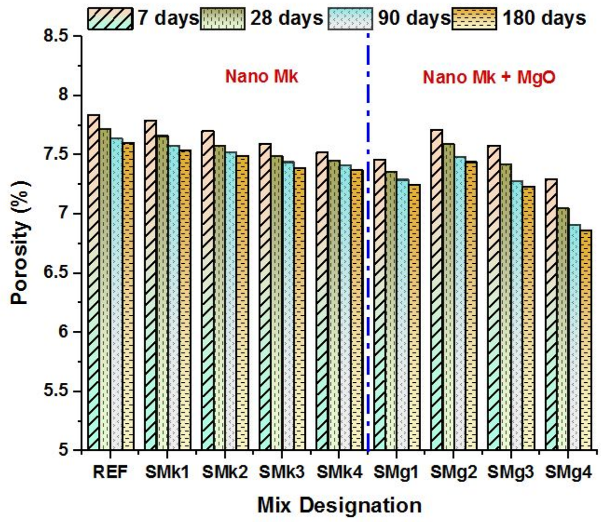

Fig. 8 - Porosity of self cured self compacting concrete specimens.

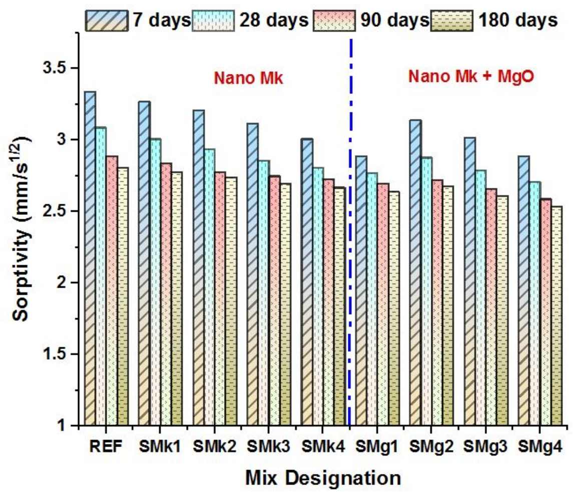

Fig. 9 - Sorptivity of self cured self compacting concrete specimens.

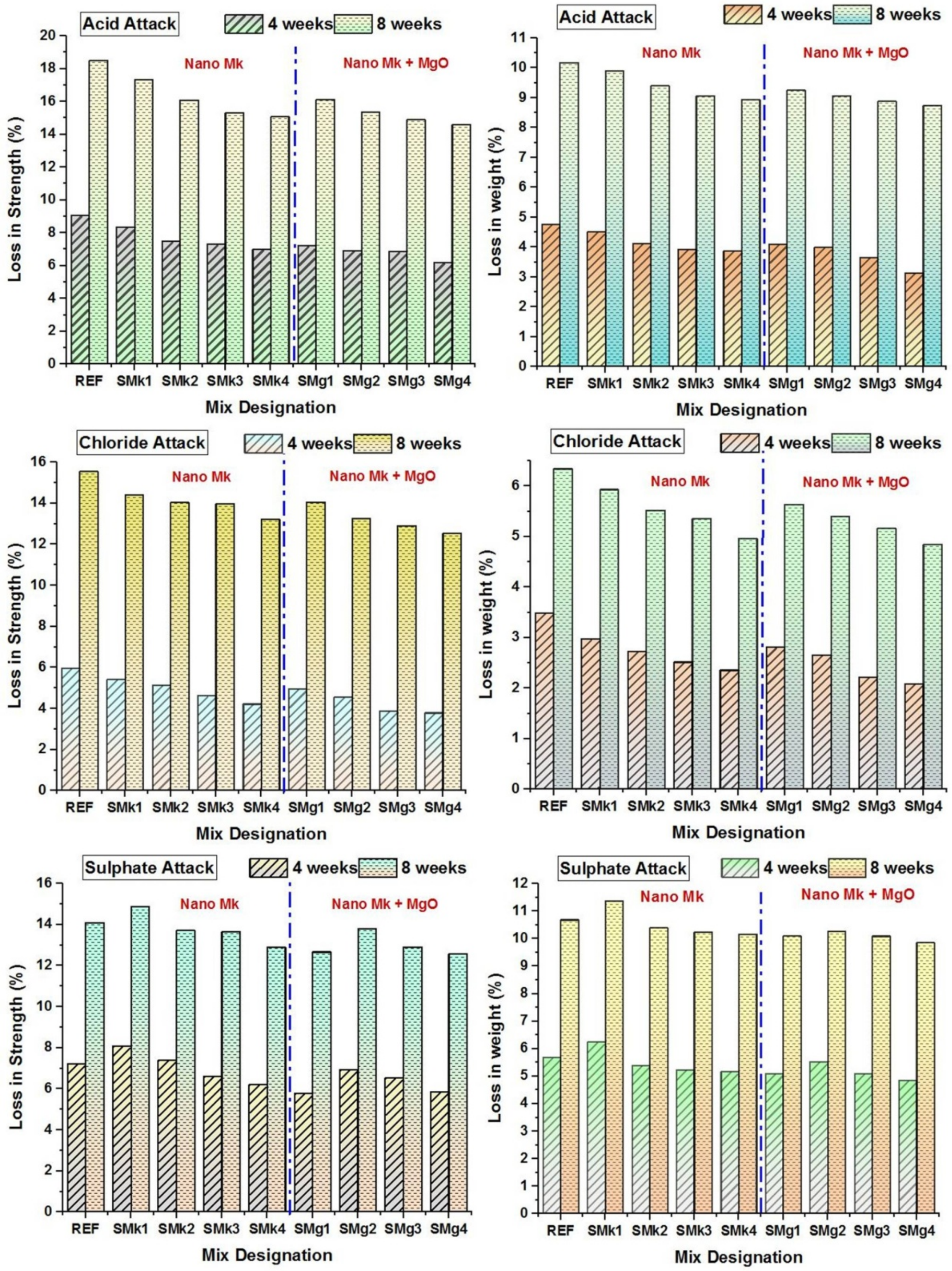

Fig. 10 - Loss in weight and strength of self cured self compacting concrete specimens under chemical exposure.

absorption and porosity values due to the classification of the matrix caused by the hydration of CH crystals into CSH gels due to the pozzolanic actions. The copper slag also exhibited fine granular sizes that occupied the interstitial pore spaces through which water can penetrate through the concrete.

4.6. Porosity

The porosity values of the self compacting concrete mixes were reduced due to the additions of nano metakaolin and expansive agents in the self curing self compacting concrete as shown in Figure 8.The self curing agents generally have the ability to reduce the amounts of water that are evaporated into the atmosphere since these concretes are subjected to air curing and this property was further promoted due to the additions of expansive agent. The reduction in the volume of pores caused due to evaporable water has caused a reduction in the porosity of the concrete. The reduction in the amounts of water absorbed through the capillary absorption test clearly signifies the reduced amount of pores and void volume in the concrete. The pores are effectively filled by the thin polymeric film of the self curing agent in combination with nano metakaolin and expansive agent that surrounded the aggregate spaces forming dense structure with lesser porosity.

4.7. Sorptivity

The sorptivity values of the self compacting concretes containing varying proportions of nano MK and expansive agents in self cured self compacting concrete are shown in Figure 9. The capillary water absorption tests conducted due to the uniform availability of water throughout the concrete has caused the reaction of the cement grains with the supplementary cementitious materials thereby forming stable hydration products. The self curing agents have also caused reduced surface evaporation of water thereby minimizing the sorptivity values. The influence of expansive agents and self curing agents on the reduction the sorptivity of the concrete is significant since the densification of the microstructure and compactness of concrete has been increased due to the expansive agents and self curing agents.

4.8. Chemical attack

The resistance to the attack of harmful chemical agents define the durability of concrete. When concrete is subjected to the attack of acids, sulphates and chlorides as in Figure 10, they undergo drastic dimensional changes due to the flow of deleterious ions through the pores in concrete. The depth of penetration of the harmful substances into the concrete was decreased due to the use of nano metakaolin and expansive agent into the self curing concrete along with the additions of mineral admixtures. The durability was improved very effectively with the increase in the self curing and expansive agents. The transport properties of concrete were severely restricted due to the filling effect of GGBS, alccofine and the metakaolin which was further supported by the expansive agent. The micro crack formation due to shrinkage was severely reduced due to the use of expansive agents. The use of expansive and self curing agents reduced the ingress of chloride ions and OH^{+} ions into the concrete due to theabsence of free flowing pore water solution. The water binder content reduction also reduced the excess free water that can be evaporated. Moreover the improved hydration products formed resulted in the formation of tortuous pore structure by forming high precipitates of CSH gel. The depth of penetration of deleterious ions were also prevented by the high water retention capacity of the expansive agent that increased the pore packing density thereby forming low porous concrete.

5. Contribution and potential Applications

The benefits of using self curing agent and expansive agent can be generally practiced in construction applications due to the provided experimental results that make their usage reliable. The present study also enhances the better understanding of the usage of self cured self compacting expansive concrete mixes widening their possibility for use in structural applications conserving the natural resources through the simultaneous replacement of cement and fine aggregates. The study also tries to provide experimental data to support the performance characteristics of GGBS- copper slag SCC incorporating expansive and self curing agents thereby drawing the attention of end users to employ the developed concretes for potential applications.

6. Conclusion

By the use of expansive agents in SCC the fluidity is enhanced due to the dispersive effect on nano metakaolin produced by these agents. The self curing self compacting concrete mixes containing nano metakaolin showed acceptable values of strength and durability and hence suggests their usage in areas where external curing is practically impossible. The results also indicate that the workability of self curedself compacting concrete containing nano metakaolin resemble the performance of control concrete and hence satisfy the EFNARC requirements. The use of expansive agent also provided desirable results in terms of chemical resistance behaviour and shrinkage performances. The utilization of superplasticizers can also be reduced due to the expansive agents thereby the construction cost is reduced. Thus the developed self compacting concrete mixes containing expansive agents can also be commercialized for use in specific applications.

Thus the present study has bridged the gap existing in the use of self curing and expansive agents combined with mineral additives for use in self compacting concrete and have yielded positive results.

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