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Mechanical Properties and Micro Structure Characteristics of Ternary Blended Concrete with Ceramic Powder and SiO_2

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ABSTRACT
The chemical compositions of ceramic powder and micro silica were checked with XRF and were found to be satisfactory as both micro silica and ceramic powder possess chemical components related to cement. A high strength concrete of M50 grade was made using ceramic powder obtained from used or broken tiles at 4, 9 and 14% replacement levels and micro silica at 1%, both by weight of cement. The strength after 7 and 28 days curing were checked and was found that the blended concrete initially showed less strength but reached 65% of the desired mean strength. Control concrete strength was found to be more after 7 days curing. The blended concrete started showing increased strength at later age from the 28th day. Concrete with 9% ceramic powder and 1% micro silica showed better strength-increase from 7-28 days. It is understood that addition of ceramic powder and micro silica contributes to later age strength development. The microstructure of the concrete for various compositions were also checked using SEM and it was found that the C9+MS1% combination showed dense packing of concrete with the presence of C-H (Calcium-Hydroxide) which contributes to strength development.

Key words: Ceramic powder, micro silica, SEM, ternary, strength development, Calcium-Hydroxide

INTRODUCTION
After water, concrete is considered to be the largest consumed material and the most widely used construction material in the world (Reddy *et al*., 2013). For high strength and high performance concrete mixes, use of supplementary cementitious materials has become an integral art since, high strength or high performance concrete using cement without any mineral admixtures require high paste volume which may lead to problems such as excessive shrinkage, high evolution of heat due to hydration and more significantly increase in cost (Raj *et al*., 2013). Use of supplementary cementitious materials as a partial replacement for cement can overcome such problems (Rajamane *et al*., 2003).

Not all materials can be used as partial replacements; only those supplementary cementitious materials, which can improve the mechanical properties, show less permeability and high resistance to chloride ion penetration or sulphate attack by possessing pozzolanic reactions. Such partial replacements can be done by natural materials such as calcined clay, metakaolin, by products such as silica fume and industrial wastes such as ground granulated blast furnace slag, fly ash, etc. Many work has been reported regarding use of mineral admixtures as supplementary cementitious material in concrete, since they posses pozzolanic characteristics. Ganesh and Rao (1993) reported the studies on effect of fly ash in concrete. Kartikeyan *et al*. (2014) performed micro structure analysis and strength properties of concrete with silica fume. Ground granulated blast furnace slag was used as partial replacement in the strength and durability characteristics study of concrete by Karthikeyan and Dhinakaran (2014). Shanmugavadivu *et al*. (2014) used ultra-fine sized metakaolin as the supplementary cementitious material in the preparation of high strength concrete.

In addition to the mineral admixtures listed above many industrial, construction wastes possessing pozzolanic activity or having chemical composition related to cement are also being tried as supplementary cementitious materials. The use of waste glass powder in concrete as a cement replacement material was reported by Bajad *et al*. (2015). Ergun (2011) used waste marble powder an industrial by product obtained during the process of sawing, shaping and polishing of marble as one of its partial replacement materials for cement, which otherwise is not recycled or used in any industries. The report stated that using waste powder marble at 5% individually and along with diatomite 10% yielded better strength results.

Ceramics, which are widely used in many forms such as electrical insulators, utility items and tiles also find its way as a construction material now. Many previous studies on ceramic focused on using it as the replacement for coarse and fine aggregates, in concrete Halicka *et al.* (2013) presented studies on using ceramic-ware waste as coarse aggregate in concrete. Gonzalez-Corominas and Etxeberria (2014) reported the properties of high performance concrete made using recycled fine ceramics and coarse mixed aggregate. Vejmelkova *et al*. (2012) presented experimental works related to use of fine ground ceramics as a partial replacement for cement and reported that fine ground ceramics can successfully be used as a supplementary cementitious material in the production of high performance concrete. Pacheco-Torgal and Jalali (2010, 2011) performed several experimental studies on compressive strength and durability properties on ceramic wastes based concrete.

Also many works involving binary, ternary blended concrete were also being tested by researchers. The reason being the improvement in the performance of concrete due to the combined action of the different pozzolonas added with cement (Bai *et al*., 2002). Thomas *et al*. (1999) investigated the application of ternary cementitious system containing silica fume and fly ash in concrete. The authors reported that even a minor addition of silica fume by 3-6% when added with fly ash in concrete resulted in reducing the alkali silica reactivity and improving sulphate resistance effectively. Antiohos *et al*. (2007) performed studies on ternary blended concrete by adding three different types of fly ashes with concrete. The authors justified that the beneficial aspects of one of the fly ashes would compensate the defects in other type resulting in improved performance.

From the above literature, we infer that use of mineral admixture either in single, binary or ternary can have positive effect on concrete. A very small percentage of micro silica can also be used as a partial replacement of cement to improve the early age strength, making the concrete ternary blended with similar strength characteristics. Micro structure analysis of the broken concrete specimen was done using Scanning Electron Microscope (SEM).

MATERIALS AND METHODS

Ordinary portland cement with a specific gravity of 3.16 was used for this research. Table 1 shows the chemical composition of cement used. Ceramic powder was prepared by grinding waste ceramic tile in dry ball mill. Waste ceramic tiles are fed into the dry ball mill with 7 steel balls of diameter 30 mm weighing 400 g each. The ceramic tiles were subjected to grinding for different grinding periods such as 30, 45 and 60 min and then the powder obtained was sieved through a 75 µm sieve to collect particles finer than cement. Figure 1 shows the ball mill used for grinding and Fig.2 shows the images of ceramic particles before grinding and after grinding. The samples of ceramic powder ground at different durations were subjected to particle size analysis and the samples which are finer were used for the research work.

The chemical composition was found using X-Ray Fluorescent test (XRF). The XRF analyzer model of Tiger 88 was used in the analysis (Table 1). The particle size of the ceramic powder was checked by zeta analyzer to ensure that their size is less than the size of cement (Table 2). Micro-silica (SiO_2), which is a by-product obtained from Ferro silicon industries was used as the other mineral admixture and is obtained from Oriental Exporters, Navi Mumbai, Maharashtra. The particle size of micro-silica was analyzed using PSA and the chemical composition of the micro-silica was found using XRF (Table 1 and 2).

River sand was used as fine aggregate and the coarse aggregate passing through 16 mm sieve and retained on 12.5 mm sieve was used as coarse aggregate in the concrete mix. The specific gravity of the aggregates and mineral admixtures used are shown in Table 3. The specific gravity of coarse aggregate used was determined by wire basket method prescribed in ASTM C127 (2012) standard. The mixture composition was obtained determined by doing a mix design as per ACI 211.1 (1991). To obtain the workable concrete mix, CONPLAST SP 430, a commonly available super

Table 1: Chemical composition of cement and micro silica

Compounds	Cement concentration (%)	Micro silica concentration (%)	Ceramic powder from waste ceramic tiles (%)
SiO_2	17-25	97.36	55.80
MgO	0.4-0.5	0.79	4.28
Al_2O_3	0.5-0.6	0.53	19.13
SO_3	2-3.5	0.51	0.54
K_2O	-	0.29	1.36
Fe_2O_3	3-8	0.15	7.88
CaO	60-67	0.14	7.85
P_2O_5	-	0.09	0.13
Na_2O	-	0.06	1.17
Cl	-	0.02	0.13
MnO	-	0.01	0.04
PbO	-	0.01	
TiO_2	-	0.01	1.31
Cr_2O_3	-	100 ppm	0.05
ZnO	-	70 ppm	0.10
CuO	-	51 ppm	0.04
Ru	-	47 ppm	90 ppm

Table 2: Size of Micro silica and ceramic powder

Sample type	Size
Silica fume	0.638 µm
Ceramic powder	2481 nm

Table 3: Specific gravity of materials

Materials	Specific gravity
Cement	3.16
Fine aggregate	2.63
Coarse aggregate	2.55
Micro silica	2.20
Ceramic powder	2.18

Fig. 1: Ball mill used for grinding ceramic tiles

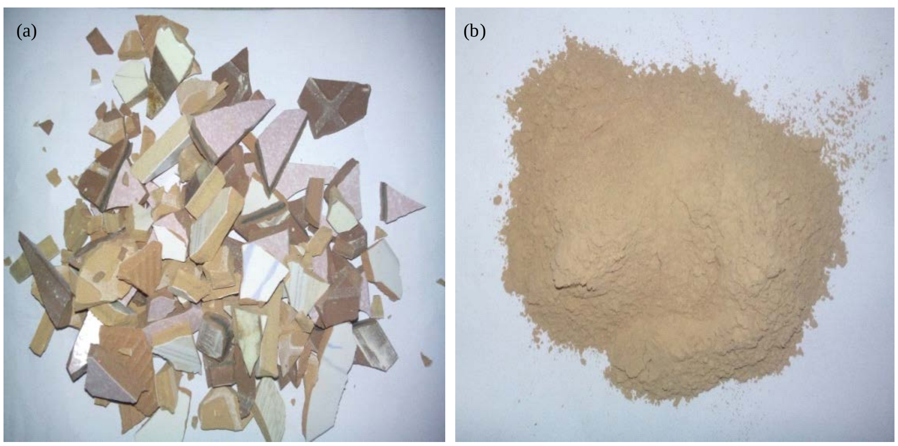

Fig. 2(a-b): Waste ceramic tiles before and after grinding in Ball Mill

plasticizer obtained from FOSROC Company was used. The dosage of super plasticizer was decided based on the desired workability of concrete by conducting slump cone test according to ASTM C143 (2012).

In the present study a high strength concrete grade of M50 has been adopted. ACI method was adopted to arrive at the mix design. A total of 4 combinations were prepared. First combination was a control concrete with no mineral admixtures. The second, third and fourth combinations replaced cement with 4, 9 and 14% of waste ceramic powder and uniform 1% of micro silica in all the three mixes. The obtained mix ratio was 1:1.04:2.13 (Cement: Fine aggregate: Coarse aggregate). Table 4 shows the mix proportion details.

Concrete cubes of size 100×100×100 mm and cylinders of size 100×200 mm were cast to study the compressive and tensile strengths. BS 1881 codal provisions were used for casting specimens. Compressive strength of concrete at the ages of 7 and 28 days were tested to understand the effect of age of concrete. The test to assess compressive strength was conducted using digital compression testing machine of 3000 kN capacity at 200 kN min^{-1} as rate of loading. For testing compressive strength of concrete, a total number of 24 specimens were cast [2 ages (7 and 28 days) ×4 different percentages of waste ceramic powder (0, 4, 9 and 14%) with constant percentages of micro silica

×3 (numbers of specimen)]. Similarly 12 cylindrical specimens were prepared to assess the tensile strength by conducting split tension test.

The morphology of the concrete specimens was checked with SEM. After the curing period the specimens underwent strength tests and samples were collected from the broken concrete specimens for each combination and their microstructures, studied.

RESULTS AND DISCUSSION

Effect of ceramic powder on compressive strength: Table 5 shows the 7 and 28 days compressive strength values of concrete, replaced with different percentages of ceramic powder and micro silica. The 7 days strength results showed that the concrete (control concrete and other combinations) reached more than 65% of the desired strength of 50 MPa. Control concrete showed maximum strength among all combinations. This is because, the concrete with mineral admixtures gain strength by continuous hydration process and initially, the strength will be less since, they are unable to compensate the loss of the cement by the mineral admixtures. After 28 days, considerable strength increase was noted in all combinations as shown above. Control concrete specimens showed a strength increase of 20.39% in 28 days compared to 7 days. Though the increase in strength by C4%+1% is the least, it has shown a strength rise by 10.104%. C9%+MS1% showed the highest rise in strength by 54.19%. All combinations, except C4%+MS1% have achieved the mean target strength and rapid strength increase in 28 days.

Ceramic powder can be obtained from a variety of means such as from brick powder, sanitary wastes, roof tiles etc. and several works were done using many ceramic materials as fine aggregates, coarse aggregates and cement replacement materials. This project is confined to use of ceramic powder from waste ceramic tiles as a supplementary cementitious material only and exact combination of projects done in this field may be difficult to find. However, few works which are relevant to this research are found and they are discussed here. The optimum strength for Pacheco-Torgal and Jalali (2010) and when ceramic powder was used as a partial replacement for cement was found at 20% of replacement of cement. Vejmelkova *et al*. (2012) reported that the mechanical properties of concrete made with fine ground ceramics were better than control

Table 4: Mix proportions adopted in the present study

Parameters	Mix reference Control	C4%+MS1%	C9%+MS1%	C14%+MS1%
Cement (kg m^{-3})	q522.57	475.54	470.31	444.18
Ceramic powder (kg m^{-3})	-	20.90	47.03	73.16
Silica fume (kg m^{-3})	-	5.23	5.23	5.22
Fine aggregate (kg m^{-3})	544.18	544.18	544.18	544.18
Coarse aggregate (kg m^{-3})	1113.84	1113.84	1113.84	1113.84
Water (L m^{-3})	182.9	182.9	182.9	182.9
Super-plasticizer	1-3 (L) per 100 kg of cement	1-3 (L) per 100 kg of cement	1-3 (L) per 100 kg of cement	1-3 (L) per 100 kg of cement
w/c	0.29	0.29	0.29	0.29
Mix ratio	1:1.04:2.13			

Table 5: Compressive strength

Mix reference	Strength after 7 days (MPa)	Strength after 28 days (MPa)	Increase in strength (%)
Control	50.25	60.50	20.39
C4%+MS1%	39.74	43.75	10.10
C9%+MS1%	39.55	60.99	54.19
C14%+MS1%	41.75	58.96	41.22

concrete upto 20% replacement levels but started decreasing when ceramic was added more than 20%. Ay and Unal (2000) presented in their report that cement replaced with waste ceramic powder beyond 35% yielded better 7 days strength but failed in 28 days strength and concluded that ceramic powder can be replaced by a maximum of 35% by weight of cement.

On comparing the above presented research works it is understood that the maximum compressive strength for concrete ceramic was obtained for a replacement percentage between 20% and 35%, but in this research better strength was shown for 10% replacement i.e., for combination with 9% ceramic and 1% micro silica, which is found to be optimum than the above mentioned works because there is considerable time and money saved in grinding the ceramic tiles, which helps in reducing the overall economy of the project. Though, the reason for increase in strength is due to the addition of micro silica, it is added in a much smaller percentage which will not affect the economy of the project.

Heidari and Tavakoli (2013) had done experimental analysis on concrete with waste ceramic tiles powder and nano SiO_2 and reported that maximum compressive strength was obtained for the mix with 10% ceramic powder and 1% nano SiO_2. In our research work without using nano SiO_2, the optimum strength was obtained for the same replacement level but with 9%ceramic powder and 1% of normal micro silica.

Effect of ceramic powder in tensile strength: Table 6 shows the results of split tensile test conducted on cylindrical concrete specimens. Control specimens showed a strength increase of 11.77% in 28 days from 7 days. Maximum strength increase of about 24.35% was obtained for C14+MS1% and a minimum increase in strength was shown by C9%+MS1%. So, it can be seen that there is an impact on tensile strength, after addition of mineral admixtures.

Scanning Electron Microscope (SEM) observations of micro structure: The morphology of ground ceramic powder and microsilica were checked with scanning electron microscope and it was observed that the majority of ceramic particles were spherical in shape. Irregularly shaped particles were found to be less in number probably due to gringing effect. Silica particles also possessed similar spherical shaped particles (Fig. 3 and 4). The micro structure of concrete with ceramic powder and microsilica as mineral admixtures were analyzed with scanning electron microscope (Fig. 5-8) showing different morphologies of the concrete with different combinations of ceramic powder and micro-silica. The results were interpreted in reference to ASTM C1723 (2010). The microstructure of control concrete (Fig. 5) showed that the pores of larger width are present in the concrete while, the concrete is not densely packed. But the presence of ettringite, which plays major role in decreasing the strength is not seen in the image. Ramachandran and Beaudoin (2001) observed that the addition of mineral admixtures in concrete makes the concrete closely packed. It is understood from Fig. 6 that, the concrete is closely packed, compared to the control concrete but here too the necessary components which are responsible for strength, such as Calcium Silicate Hydrate (C-S-H), Calcium-hydrate (C-H) are missing, though the presence of

Table 6: Tensile strength

Mix reference	Strength after 7 days (MPa)	Strength after 28 days (MPa)	Increase in strength (%)
Control	3.19	3.56	11.77
C4%+MS1%	3.31	3.56	7.61
C9%+MS1%	3.64	3.70	1.76
C14%+MS1%	3.27	4.07	24.35

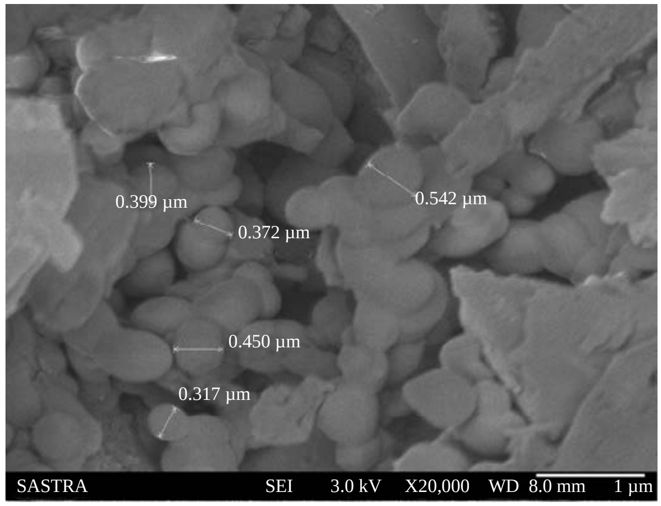

Fig. 3: Micro structure of ground ceramic powder

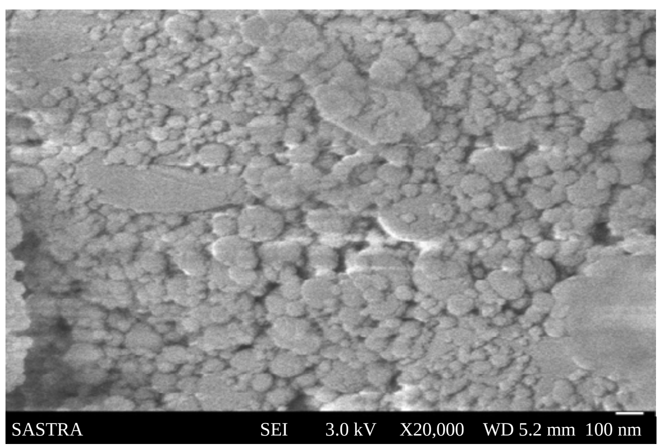

Fig. 4: Micro structure of Micro silica powder

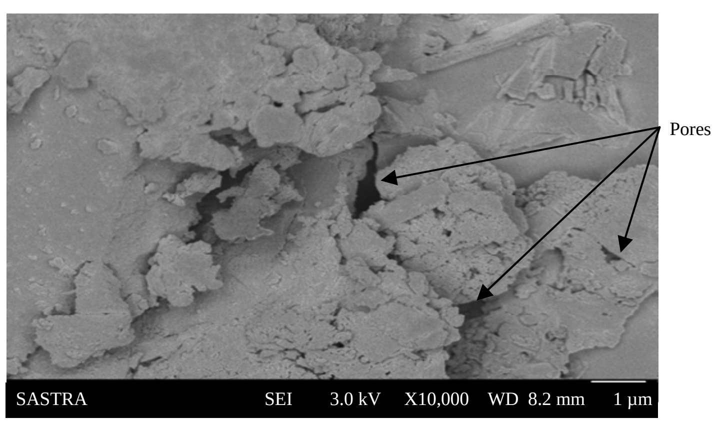

Fig. 5: Micro structure of control concrete

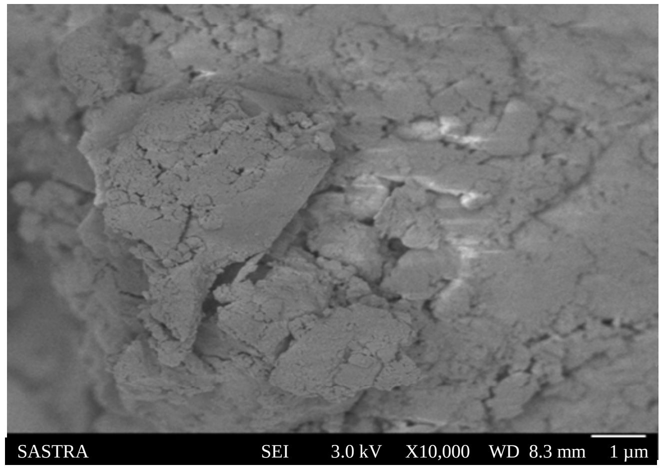

Fig. 6: Micro structure of concrete C4%+MS1%

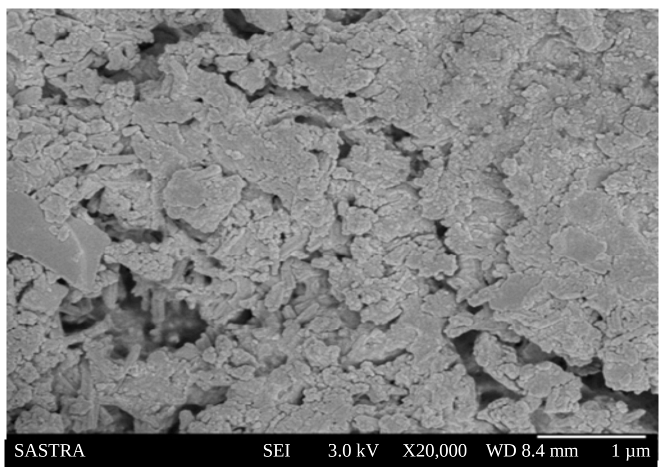

Fig. 7: Micro structure of concrete C9%+MS1%

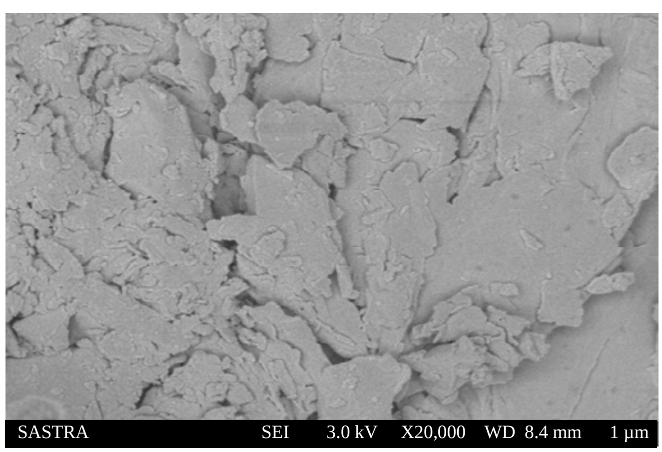

Fig. 8: Micro structure of concrete C14%+MS1%

large width pores have reduced. Small pores (dark patches) are present in a large numbers, which may be the reason for strength reduction for this combination.

From Fig. 7, it can be seen that the concrete is closely packed with less number of pores. The components which are responsible for strength, such as Calcium-Hydrate (C-H) are also seen in the image. Calcium Silicate Hydrate (C-S-H) contribute more towards the strength of concrete, as their presence in cement paste is about 50-60% and Calcium-Hydrate (C-H) making up about to 20% of solids. The presence of C-S-H is not visible at this magnification, but the presence of C-H can be seen in the form of plate like structures present in the image. Since C-H is embedded in C-S-H, it is assumed that C-S-H is also present in the combination, contributing for the strength increase of this combination.

Figure 8 shows the SEM images of concrete with ceramic powder and micro silica added in 14 and 1%, respectively. Here too, closely packed concrete particles can be seen, pores are present which may lead to decreased strength. Silica fume particles might have been dissolved and the added ceramic powder may not have been sufficient to compensate for the reduction of cement content by 15%, all leading to a reduction in strength.

CONCLUSION

Following conclusions were arrived from this experiment:

- The size of ceramic powder obtained after subjecting to grinding for 1 h was in the size range of 2412 nm
- The micro structure of ceramic powder showed the presence of more spherical particles and less irregularly shaped particles
- Much variation is not seen in split tensile strength of the specimens cast. All the combinations showed almost equal strength
- The SEM images of concrete clearly indicate the defects in the concrete and the components responsible for strength increase/decrease
- From SEM images it is under stood that concrete added with 9% ceramic powder and 1% micro silica showed a closely packed concrete, with a less number of pores and with the presence of C-H components all leading to increased strength
- In the overall comparison it is inferred that ternary blended concrete with C9%+MS1% is capable of showing strength almost equal to control concrete
- Concrete with partially replaced mineral admixtures may provide better strength when subjected to longer curing period (>28 days). The strength of specimens after more than 28 days curing showed strength equal to control specimens and possibly will show higher strengths if cured for longer periods

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