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Influence of Zinc Oxide Nanoparticle on Strength and Durability of Cement Mortar

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Abstract: This paper presents the mechanical and durable properties of cement mortar having nano-particle of Zinc Oxide (NZ) with the average particle size of 60nm. The cement was partially substituted by NZ of 0, 1, 3 and 5% by weight of cement. The blended mortar was prepared using cement-sand ratio of 1:1 and 1:2 by weight with water-binder ratio as 0.35. The compressive and split tensile strength was obtained at 7 and 28 days. Durability properties such as impact strength, water absorption, sorptivity and also microstructure of mortar were studied. The results indicate that the mechanical properties of sample comprising 3% and 5% ZnO nanoparticles are desired than traditional mortar. The comparable improvement was seen with durability properties. Scanning Electron Microscopy (SEM) study about microstructure of cement mortar enhanced with ZnO nanoparticles and plain cement mortar illustrated ZnO nanoparticle fills the pores completely and accelerate the hydration process of cement particles, which leads to increase in mechanical strength and durability of Nano-cement mortar specimens.

Keywords: *Cement mortar, ZnO nanoparticle, Mechanical and durable properties, SEM*

1. Introduction

The introduction of nanomaterials or lamellar solids in cement mortars and concrete to improve their mechanical properties has been widely employed in modern concrete technology. The recent studies on nanomaterials and nanotechnologies have highlighted the probable use of these materials in various fields such as medicine, construction, automobile industry, energy, etc., this is due to the special appearances of materials at the nano scale. Building materials domain can be one of the main beneficiaries of these investigates, with applications that will improve the characteristics of concrete, steel, glass and insulating materials. The use of nanomaterials in the configuration of some materials, such as cement, will result in major reductions of CO_2 pollution and the use of performance thermal insulations will result in efficient use of energy for air conditioning [1]. Presently, the use of nanomaterials in construction is condensed, mainly for the following reasons: the lack of knowledge about the design and implementation of the construction elements using nanomaterials; the reduced proposal of nanoproducts; the deficiency of detailed information concerning the nanoproducts content; great costs; the unknowns of health risks associated with nanomaterials.

Many investigations have studied the mechanical and durable properties of cement based materials containing nanoparticles. Studies done shows that adding nanoparticles to cement based materials increases the mechanical properties. Sattawat Haruehansapong *et.al* [2], have examined the compressive strength of cement mortar enclosing the nano silica and silica fume. The results show that the compressive strength of mortar containing nano-silica has improved compared than mortar containing silica fume. Hui Li *et.al* [3], proved that addition of Fe_2O_3 and SiO_2 nanoparticles increase the mechanical properties of mortar. The SEM observations also displayed that the nano-particles were not only acting as filler, but also as an activator to promote hydration proves and to progress the microstructure of the cement paste if the nano-particles were evenly dispersed. Nano particles of SiO_2 can fill the voids between particles of gel of C-S-H, acting as nano filler. Also, by pozzolanic reaction with calcium hydroxide, the amount of C-S-H growths, resulting a higher densification of the matrix, which improves the strength and durability of the material. The influence of nano silica on consistency and setting time are dissimilar. Nano SiO_2 creates cement paste thicker and accelerates the cement hydration process [4]. Ali Nazari *et al* [5], investigated influence of Al_2O_3 nanoparticle on the compressive strength and workability of blended concrete. The results presented that adding this nanoparticle up to maximum replacement level of 2% produces concrete with improved strength, workability of fresh concrete was reduced by increasing the content of Al_2O_3 nanoparticle. The results showed that adding Fe_2O_3 nanoparticle up to 5% reduce the mechanical properties of cement mortar [6]. Rahmat Madandoust *et.al* have studied the influence of nanoparticles on the properties of self-compacting mortar containing nano-SiO_2, nanoFe_2O_3 and nano-CuO. It was observed that the workability improved slightly and also nanoparticles could improve mechanical and durability properties of SCM specimens. The SEM micrographs proved more packed pore structure of the mortars containing nanoparticles which leads to rise in strength and durability of SCM specimens [7]. Sonebi *et al* [8], evaluate the effects different substances of nS, FA and different SP amounts on the fresh properties and plastic and drying shrinkage of cement mortars. The results show that, variations in nS content have a more prominent effect on flowability and on plastic shrinkage, which is considerably reduced when nS content is increased. M.Aly *et al* [9, 16], focused on properties of mortar containing nano clay and finely ground glass dust.

Addition of small amount of nano clay accelerate the pozzolanic activity, enhanced the workability, mechanical and thermal performance of cement paste. Meral Oltulu *et.al* [10], have investigated individually and paired the effect of adding nanoparticles of nano-SiO_2, nano-Al_2O_3 and nano-Fe_2O_3 to cement mortars containing silica fume. The experimental results show that adding these nanoparticles individually causes the increase of compressive strength. But, the mutual of nanoparticles has a negative effect on the mechanical properties of a cement mortar. The nano powders such as nano-SiO_2, nano-Al_2O_3 and nano-TiO_2 used individually or in combination increased the compressive strength of silica fume containing mortar [11]. The replacement of OPC by RHA and nSO in simultaneous use in mortars of plastic consistency is beneficial of physical, mechanical and corrosion resistance properties. Based on results, declined in porosity of mortar up to 5.7% and improved compressive strength and corrosion of 7.8% and 51.1% respectively [12]. Use of nano structured waste materials such as spent catalyst, copper slag and cement kiln dust were improved mechanical and durability properties of mortar [19]. If Engineered or manufactured nanomaterials (ENMs), which are purposefully produced nanomaterials such as nano-SiO_2, nano-Al_2O_3, nano-Fe_2O_3, nano-ZnO, nano-ZrO_2, CNT, etc., are integrated with cement based building material, the new material might possess certain outstanding properties.

In this work, the influences of nano-ZnO on mechanical and durable properties of blended cement mortar have been investigated. The reason for using ZnO nanoparticle as a partial replacement of cement is a unique material that exposes semiconducting and piezoelectric dual properties and ZnO improvements the processing time and struggle of concrete against water in the manufacturing of concrete [1]. An attempt has been made to prove that using ZnO nanoparticles, it is possible to obtain blended mortar with high mechanical and durable properties with slight increase in cost.

2. Experimental Program

2.1 Materials

Materials used in this study were 53 grade Ordinary Portland Cement, Natural River sand, nano particle of zinc oxide (NZnO) with average particle size of 60nm, containing 99.5% Zno. The main properties of nano particle are given in Table 1. Tap water was used in this study mixed with superplasticizer Enfiiq sp400 in order to achieve the desired workability of blended cement mortar and better dispersion of nanoparticles.

Table-1: *Properties of Nano-ZnO*

Average particle size (nm)	Specific surface area (m^2/g)	Density (g/cm^3)	Content of ZnO (%)	Form
60	18	0.25	>99.5	White

2.2 Mix Proportions

The mix proportions of traditional mortar and blended mortar are given in Table 3. Fresh mortars were prepared with different binder/sand ratio of 1:1 and 1:2. The ratio of water to binder ratio was chosen 0.35. In this investigation, the nanoparticle contents in the mortar specimens were 1, 3 and 5% by weight of cement.

Table-2: *Description of Mortar*

S.NO	Type of Mortar	Description of Mortar
1	A	Conventional Mortar 1:1
2	B	Conventional Mortar 1:2
3	A1	1% of cement replacement by ZnO nanoparticle in mortar 1:1
4	A3	3% of cement replacement by ZnO nanoparticle in mortar 1:1
5	A5	5% of cement replacement by ZnO nanoparticle in mortar 1:1
6	B1	1% of cement replacement by ZnO nanoparticle in mortar 1:2
7	B3	3% of cement replacement by ZnO nanoparticle in mortar 1:2
8	B5	5% of cement replacement by ZnO nanoparticle in mortar 1:2

Table-3: *Mix proportions of the Specimen (By volume, m^3)*

Mortar type	W/C Ratio	SP % of Cement	Cement	NP of Zno	Sand
A		-	1	-	1
B		-	1	-	2
A1		0.5	0.9	0.1	
A3	0.35	0.5	0.7	0.3	1
A5		0.5	0.5	0.5	
B1		1	0.9	0.1	
B3		1	0.7	0.3	2
B5		1	0.5	0.5	

SP- superplasticizer, NP-Nanoparticle

2.3 Sample Preparation

The blended mortar mix design using natural river sand, cement with partially replaced by nanoparticle of zinc oxide. Nanoparticles are not easy to disperse evenly due to their high surface energy. Thus, mixing was performed as follows; the ZnO nano powder was mixed with water. At first, the suspension of the ZnO nanoparticles and superplasticizer were mixed in the laboratory mortar mixer for 30 second, where the cement was added to this mixture instantaneously. Then, the Mortar was transferred into the standard mould. For compressive strength test and durability study such as water absorption and sorptivity test, the mortar cube specimens with 7.6 x 7.6 x 7.6 cm dimension were used. In order to prepare the specimens of the split tensile strength test, the mortar was poured into the molds to from cylinders of size 100x200 mm, six specimens were prepared for each test and the average test result was reported.

2.4 Test Methods

Compressive strength test were carried out according to the ASTM-C109 [22] and split tensile strength test were carried out according to ASTM C496 [23]. Water absorption and Sorptivity test were carried out according to the ASTM C642 [24] and ASTM C1585 [25] respectively. The microstructure of the mortar specimens was studied by Scanning Electron Microscopy (SEM).

3. Results and Discussions

3.1 Strength

Results of compressive and split tensile strength after curing for 28 days is given in table 3. It can be understood from the table that the sample having 3% ZnO nanoparticles, the mechanical properties of mortar has increased than the traditional mortar. But, the increased ZnO nanoparticle up to 5%, the strength of mortar reduced. Figure 1 and 2 shows the compressive and split tensile strengths of sample respectively. It can be seen that mechanical properties of specimens with mixtures A3 (mortar with 3 % nano-ZnO) at the 7^{th} and 28^{th} day were all greater than that of a traditional mortar. The specimen having 5NZ is reduced the mechanical properties of blended mortar. Weak zone in the form of cavities will be produced due to unsuitable dispersing of nanoparticles. Nanoparticles fill the cement pores, thus improvement of strength will be expected. Since the content of NZ is too large, this particle cannot be well dispersed and consequently by combining nanoparticles, weak zones are formed.

Table-4: *Compressive Strength and Split Tensile Strength of Samples*

Mortar type	28 days			
	Compressive strength (MPa)		Split tensile strength (MPa)	
	Target	Enhanced extent (%)	Target	Enhanced extent (%)
A	44.80	-	3.18	-
B	41.12	-	2.07	-
A1	50.11	11.85	3.96	25.20
A3	***54.51***	23.88	***4.45***	39.93
A5	51.60	15.17	4.05	27.35
B1	48.15	17.09	3.10	49.75
B3	***50.60***	23.05	***3.34***	61.35
B5	49.65	20.74	3.26	57.48

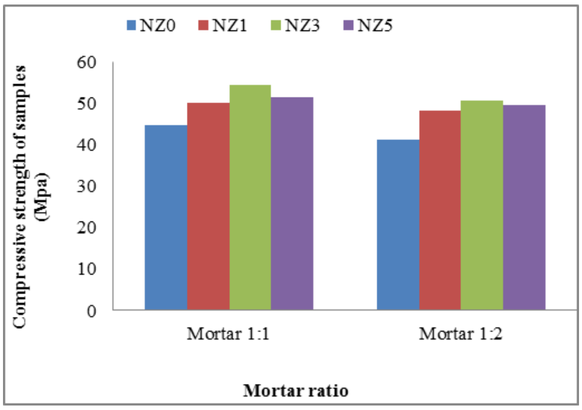

Figure-1: *Compressive strength of mortar*

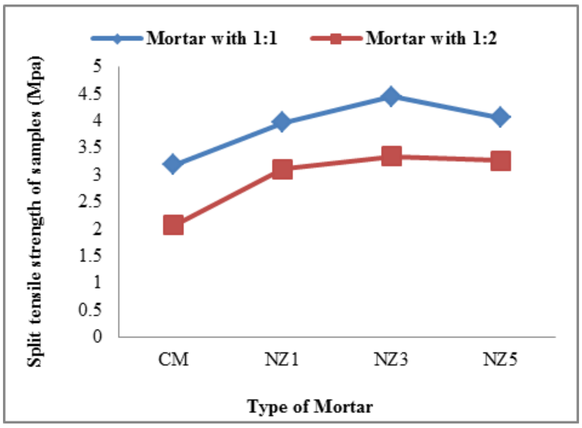

Figure-2: *Split tensile strength of mortar*

3.2 Durability of Mortar

3.2.1 Water Absorption

The cubic specimens were excluded after 28 days of curing and were prepared for water absorption test. These specimens were then oven dried for 24 hours at the temperature85°C up to the mass became constant and again weighed. This weight was noted as the dry weight (W1) of the cube. After that the specimen was kept in water at 85°c for 24 hours. Then this weight was noted as the wet weight (W2) of the cube.

$$\% \text{ water absorption} = [(W2 - W1) / W1] \times 100$$

Where, W1 = Oven dry weight of cubes in grams;
W2 = after 24 hours wet weight of cubes in grams.

3.2.2 Sorptivity

The sorptivity can be determined by the measurement of the capillary rise absorption rate on reasonably homogeneous material. Water was used of the test fluid. Sorptivity (S) is a material property which characterizes the tendency of a porous material to absorb and transmit water by capillarity. The cumulative water absorption (per unit area of the inflow surface) increases as the square root of elapsed time (t).

$$I=S.t^{1/2};$$
$$S=I/t^{1/2}$$

Where;

S= sorptivity in mm,
t= elapsed time in mint.
I=Δw/Ad
ΔW= change in weight = W2-W1
W1 = Oven dry weight of cylinder in grams
W2 = Weight of cylinder after30 minutes capillary suction of water in grams.
A= surface area of the specimen through which water penetrated.
d= density of water.

Figure 3 and 4 gives the water absorption and Sorptivity test results of % replacement of NZ in mortar for 28 days curing. The result shows higher water absorption and sorptivity.

Table-5: *Water absorption at 28thday*

Mortar type	Dry weight in grams (W_1)	Wet weight in grams (W_2)	% of water absorption
A	779.0	807.6	3.60
B	755.8	787.2	4.15
A1	802.6	829.6	3.36
A3	771.8	796.4	3.24
A5	799.6	826.8	3.40
B1	758.9	789.6	4.04
B3	759.6	786.8	3.58
B5	780.4	811.6	3.90

Table-6: *Sorptivity at 28th day*

Mortar type	Dry weight in grams (W1)	Wet weight in grams (W2)	Sorptivity value in 10^{-4} mm/min$^{0.5}$
A	1230	1260	6.97
B	1050	1082	7.43
A1	1250	1270	4.70
A3	1208	1211	0.69
A5	1237	1246	2.09
B1	1190	1202	2.77
B3	1146	1152	1.39
B5	1137	1148	2.59

3.3 Impact Resistance of Mortar

The method recommended by ACI Committee 544 was used in this study. Drop hammer impact tests based on mortar specimens with different contents of zinc oxide nano powder were carried out. The experimental results on impact strength indicated that cement mortar with addition of nano-ZnO to the matrix distribute the stresses over large area resulting increase in energy absorption capacity due to impact. Table 7 shows the impact failure energy of first and failure crack of conventional and mortar with nanoparticles. Figure 5 shows cement paste having 3 % of NP in 1:1 gives higher impact energy compared than other mortar type.

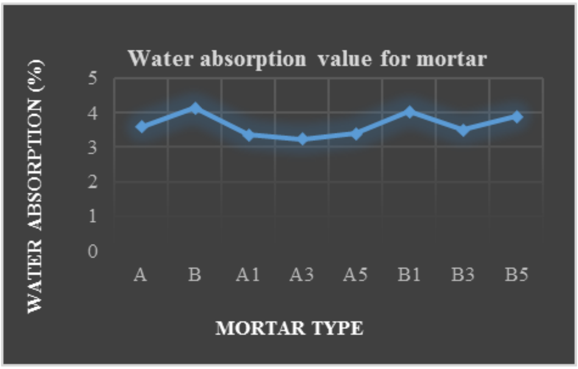

Figure-3: *Water absorption of specimens*

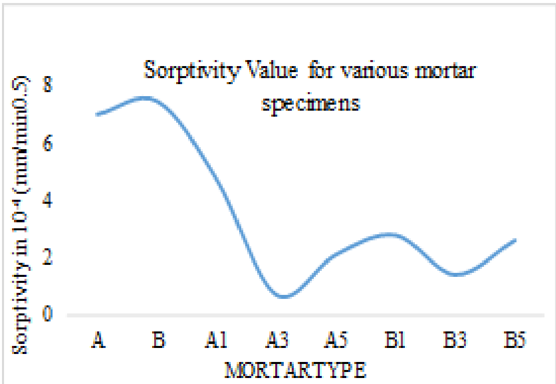

Figure-4: *Sorptivity of specimens*

Table-7: *Impact resistances of mortar specimens*

MORTAR TYPE	Height (m)	Weight (N)	No.of blows		Impact Energy (N-m)	
			First Crack	Failure Crack	First Crack	Failure Crack
A	0.457	44.5	12	14	244.03	284.71
B			9	11	183.02	223.70
A1			19	20	386.39	406.73
A3			24	26	507.30	549.57
A5			16	17	338.20	359.33
B1			15	16	317.06	338.20
B3			18	20	380.47	422.75
B5			12	13	253.65	274.78

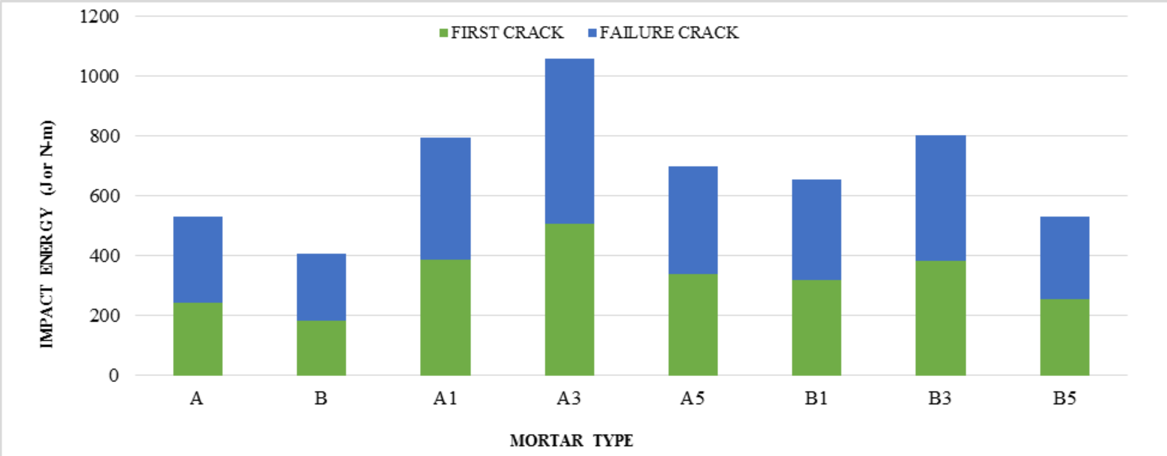

Figure-5: *Impact energy of mortar*

3.4 Microstructure of Samples

The mechanical and durability properties are influenced by the microstructures and are explained by the SEM micrographs. Figure shows the SEM micrographs of mortars without and with 3% of nanoparticles after 28 days of curing in water. After compression testing the crushed specimens are collected to analysis the microstructure by using scanning electron microscopy. Nano ZnO particle act as a filler to increase the density of mortar which can be ensued to reduce the porosity of samples.

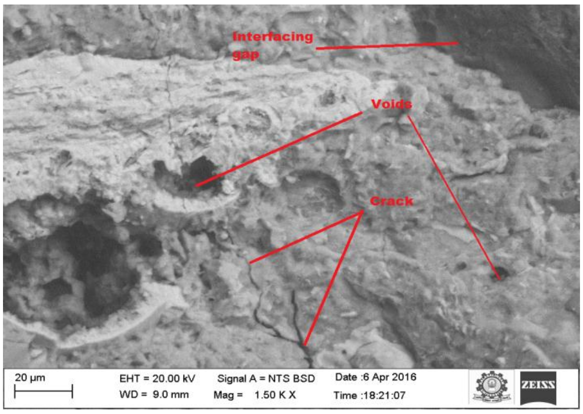

Figure-6: *SEM photograph of without nanoparticle in 1:1*

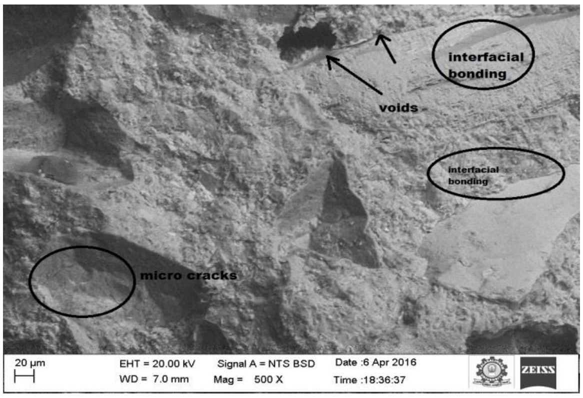

Figure-7: *SEM photograph of with nanoparticle in 1:1*

As it is shown in figure there are large pores and cracks are observed in control specimens, but by addition of zinc oxide nanoparticle, these voids are occupied and microstructure develops denser. The structure of the important calcium-silicate-hydrate (C-S-H) gel which is liable for the mechanical and physical properties of cement pastes, including shrinkage, creep, porosity, perviousness, can be modified to attain superior durability.

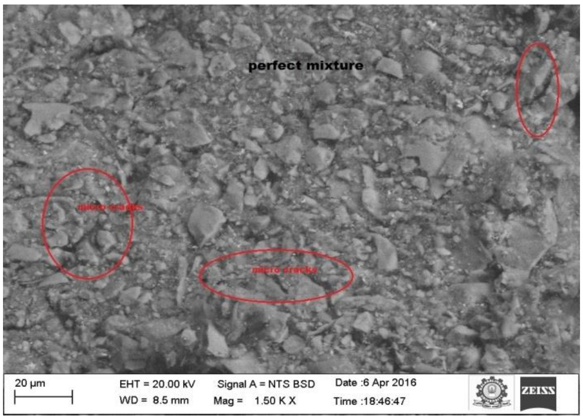

Figure-8: *SEM photograph of without nanoparticle in 1:2*

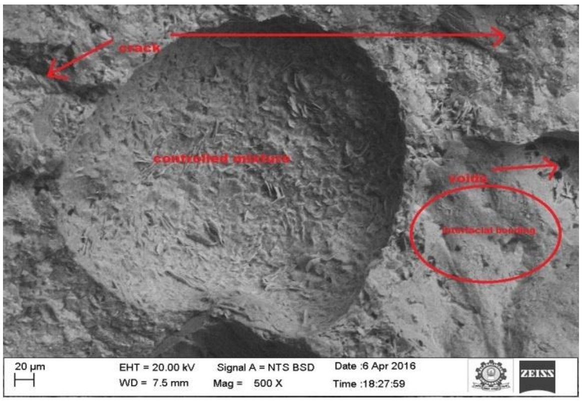

Figure-9: *SEM photograph of with nanoparticle in 1:2*

This structure can be improved by using nanoparticle. A 3% replacement of cement by zinc oxide nanoparticle appears to increase the structure of the calcium-silicate-hydrate which leads to improve the hydration of cement paste. Sample A3 having better microstructure compared to sample B3.

The fig 6 & 8 shows the conventional mixture, in that fig 6 shows some cracks as well as voids and fig 8 shows controlled mixture but not have good surface level with micro cracks. It seems that the conventional having low strength. Hence the fig 7 & 9 shows the bonding of cement mixture with ZnO. It shows good surface properties it indicates that the interfacial bonding of cement with the nano powder mixtures. In fig 7 we can see the small micro cracks as well as the voids its seems that the compressive strength of the mortar at the mean while fig 9 it seems high cracks, voids and large breakage in this we have concluded that the if we increase the mix proportion it lower down the compressive strength.

4. Conclusions

Zinc oxide nano particle filled cement composite was developed for a new combination of cement mortar with improved strength and durability. From the experimental studies, the following conclusions are made.

From the experimental results of mechanical strength it is expected that adding of ZnO nanoparticles up to 3% by weight of cement can act as filler, strengthening the microstructure of mortar. The experimental results of compressive and split tensile strength of mix ratio 1:1 are 23.88% and 39.93% respectively higher than conventional mix proportion 1:1. Based on the results of compressive and split tensile strength of mix ratio 1:2 is 23.05% and 61.35% respectively better than conventional mix proportion 1:2. In general, the mix proportion 1:1 is having better strength than mix proportion 1:2. After added up to 3 weight % of ZnO nano particle in mix proportion 1:2 is performed better than conventional mix 1:1. The water absorption and sorptivity of blended mortar is higher than plain mortar. Because, nano particles block the passageways connecting capillary pores and cracks in cement paste. Impact strength of mortar showed significant enhancement by using up to 3% of NZ particle and then it is slightly decreased. According to SEM observations shows that the nanoparticles were not only acting as a filler, and also activator to promote hydration proves and to improve the microstructure of cement mortar if nano particles were uniformly dispersed. Partial replacement of cement by nano-ZnO particles declined workability of fresh mortar; therefore use of superplasticizer is substantial. In summary, by comparing all the observed properties, NZ3 – CM 1:1 provided the best all-round performance.

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