International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 4 Issue 09, September-2015

Influence of Micro/Nano Sized Fly Ash on the Properties of Cement Mortar

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***Abstract*— The interest of research work is to prepare and study the effect of micro/ nano sized fly ash (or ground fly ash) on the mechanical properties of cement mortar. The grounded fly ash was obtained by grinding raw fly ash in a mechanical ball milling up to 5 hours, this ground fly ash (GFA) was in the range of micro to nano level. The mortar was prepared with adding of GFA as partially replaced ordinary Portland cement at the rate of 10%, 20%, 30% and 40% by weight of binder. This paper investigates the consistency and setting time of blended cement paste and compressive strength, water permeability of blended cement mortar samples. The partial replacement of GFA with cement mortar shows improvement in the microstructure and mechanical properties of mortar. Compressive strength of cement mortars blended with 10 % micro/nano-flyash particles showed results by additional to 10%, 24% & 34% than normal mortar samples after 28, 56 & 90 days of curing respectively and water absorption test proves that 10% micro/ nano blended mortar specimens absorbs 21% less water than that of other samples.**

Key Words: Ground Fly Ash (Micro/Nano Fly Ash), Mechanical Ball Milling, Compressive Strength, Water Absorption.

INTRODUCTION

Pozzolanic materials like fly ash is currently on of the major ingredients for high strength and most efficient construction materials. As the fly ash available from thermal power plans is much coarse in nature, grinding operation is essential to realise the high performance mortar and concrete in building and transport sectors.

Grinding of fly ash is one of the very attractive options to obtain higher compressive strength of the concrete, as has been reported extensively in the technical literatures like [11], [12], [13], [14], [15], [16] and [17]. Superior strengths of fly ash mortars are obtained by Paya et al. [17] using ground fly ash to finer sizes, where the water cement ratio was maintained constant. These literatures are however not mentioning the consistency related flow parameters for the mortars, in case grinding is changed.

Extensive studies were conducted by Kiattikomol et al. [18] for the enhanced strength of mortars using grounded fly ash of five different origins, to three different fineness. The consistency and the substitution rate were kept constant for the mortars in above study. water demand by some fine ash samples in raw form were more compared the control varieties of mortar. The irregular shape and porous nature of fly ash are the dominating attributes for the higher water requirements. Hence the conclusion was that workability and consistency of mortar do not improve by the blending of fly ash in all cases.

One of the major contributions of many significant works viz [2], [3], [4], [5], [6] states microstructure study, mechanical properties and durability properties of concrete are dependent on fine particles such as nano particles of SiO_2, TiO_2, Al_2O_3, Fe_2O_3, salg and fly ash as partial replacement with cement.

Partial distribution of micro/ nano in cement paste and mortar is studied in the current research work. This paper highlights the characterization of micro/ nano fly ash by mechanical ball milling process through XRD and SEM. This also assess the effects of micro/ nano-scaled fly ash on the consistency, setting time, compressive strength and water absorption properties of cement mortar.

EXPERIMENTAL PROCEDURE

Raw Materials:

Cement:

In this study, ordinary Portland cement (OPC) 43 grade with Blaine specific surface 300 m^2/Kg complying with IS:12269:1987 was used. The chemical and physical compositions of cement are mentioned in Table 1.

Fine Aggregate:

In the preparation of cement mortar mixes standard sand in accordance with IS: 650:1991, was used with cement and blended cement i.e. with micro/nano-fly ash in different proportions.

Water: Water used, is free from impurities and having pH-7 for the specimen preparation.

Fly ash:

The fine particle of fly ash was collected from thermal power plant and added as a partial replacement to cement. The chemical and physical compositions or properties of fly ash are mentioned in Table 1.

Micro/nano-fly ash or Ground fly ash (GFA):

An effort was taken to prepare micro/ nano sized fly ash from raw fly ash by mechanical ball milling. The morphology of nano sized fly ash was mentioned in XRD and SEM as shown in fig 1 & 2.

Table 1. Chemical and physical properties of cen

Materials	Chemical composition (%)								Physical properties		
	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	Na_2O	K_2O	MgO	LOI (%)	Density (g/cc)	Blain Area (m^2/kg)
Cement	63.5	20.3	2.2	1.8	2.1	0.45	0.35	1.1	1	3.15	300
Fly ash	1.3	54.41	30.40	8.44	0.1	1.0	1.98	1.53	-	2.1	350

Table 2. Effect of milling hours on particle size

0 hr grind fly-ash		5 hr grind fly-ash		10 hr grind fly-ash	
Cum. % Passing	sieve nm	Cum. % Passing	sieve nm	Cum. % Passing	sieve nm
100	3270	100	2312	100	2312
95	2778	95	1944	95	1944
90	2597	90	1635	90	1635
80	780	80	1375	80	1375
60	678	60	1156	60	1156
50	660	50	972	50	818
40	644	40	687	40	578
30	629	30	578	30	249.4
20	615	20	409	20	243
10	599	10	344	10	204
5	588	5	289	5	171.9
0	486	0	243	0	121.5

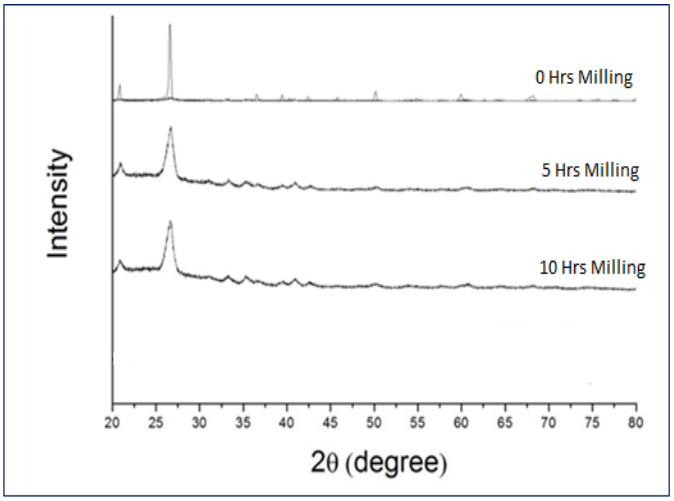

Fig. 1. X-ray diffraction of 0, 5 & 10 hr ground fly ash

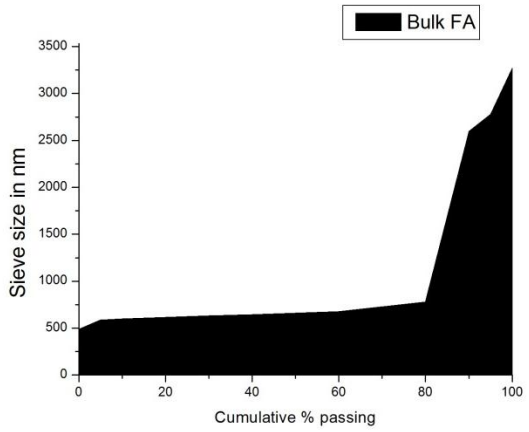

Fig.2 (a)

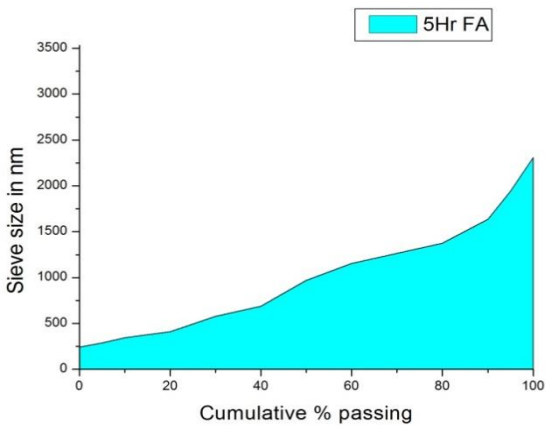

Fig 2 (b)

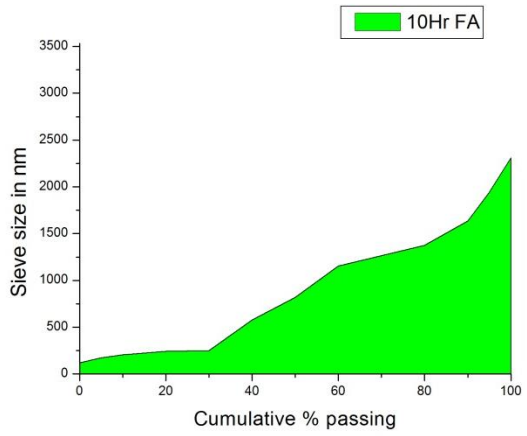

Fig 2 (c)

Fig 2. Cumulative % of particles passing of (a) Raw fly ash (b) 5 hr grinding & (c) 10 hr grinding FA

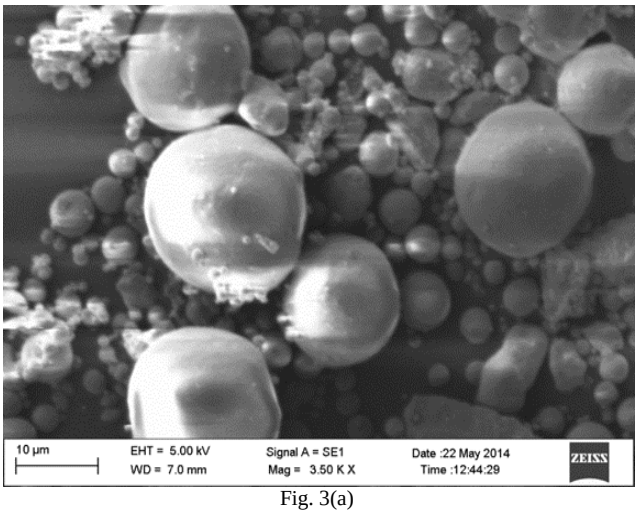

Fig. 3(a)

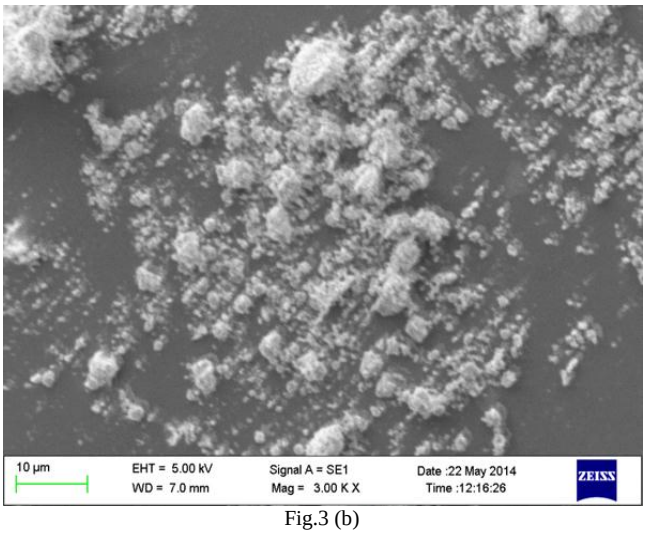

Fig.3 (b)

Fig. 3. SEM image of (a) raw fly ash (b) nano-fly ash at 10 h milling

Table. 3 Consistency, initial and final setting time of blended cement pastes

Mix	Cement %	GFA %	Consistency %	Setting Time in minutes	
				initial	final
N	100	0	30	153	230
N10	90	10	31	160	225
N20	80	20	32	166	230
N30	70	30	32	175	230
N40	60	40	33	190	255

Mortar specimen:

The cement was blended with micro/ nano fly ash in the proportions of 0%,10%, 20%, 30% and 40% by weight replacement of cement and the ratio was taken 1:3 (blended cement: standard sand). The nomenclature of the corresponding samples was for normal cement mortar (N) and for 10%, 20%, 30%, and 40% by weight replacements of cement mortar were named as N10, N20, N30 and N40 respectively. The mortar specimens are mentioned in the Table 3. The water was added to the blended cement as per IS code recommendation.

Consistency & Initial and final setting time tests:

The normal consistency, initial and final setting time tests were conducted with the Vicat's apparatus in accordance with IS: 4031:1988 part 4 and 5 and shown in Table no-2.

Compressive strength test:

Mortar cubic specimens of 70.6 x 70.6 x 70.6 mm were made for compressive strength in accordance with IS 4031 (Part 6) 1988. After 24 hours of casting, all specimens were inserted in water for curing at room temperature (25 ± 5^0C) for 3, 7, 28, 56 and 90 days as per the recommendation of IS 4031-1988. The cube specimens were taken for compressive strength test using a compression testing machine under load control at 0.2 MPa/s.

Water absorption test:

Three 70.6 mm cube samples of each mortar mix were taken for water absorption test. Saturated surface dry specimens were put in an dry oven at 100°C for 48 h. After measuring the initial weight, specimens were immersed in water for 48 h. After taking out from water, the specimen was kept outside for one hour and then the final weight was measured and the absorption was calculated and reported in the Fig.6 to assess the permeability of mortar.

RESULTS AND DISCUSSION

Consistency and Setting time:

Due to increase of ground fly ash percentages the water demand increases with respect to percentage addition of ground fly ash as shown in table 3.

Structure and microstructure analysis:
It was found from XRD for the 10 h milling, the crystallite size was reduced from 15 µm to 289 nm shown in Table 1 and Fig. 1. The size, shape and texture of the fresh as well as micro/nano structured fly ash were studied using Scanning Electron Microscopy (SEM). The high intensity peak of SiO_2 at angle 26.58 degree diffraction peak reduces at 10h, it shows that the crystalline size reduces while milling in to smaller scale and it also confirmed from the microstructure image shown in Fig. 1 & 3. Particle size distribution of different hours milling of fly ash was shown in Fig 2.

Compressive strength of mortar:
The compressive strength results of cube specimens at 3, 7, 28, 56 and 90 days were mentioned in the Table 3.

The results were mentioned in Table 3. The table shows the effect of ground fly ash addition with cement mortar on compressive strength of the hardened mortar.

It was observed that the strength was improved in 10% replacement of ground fly ash and as on increase in GFA the strength gets decreasing.

Table 4. Compressive strength of mortar specimens

Mix	Compressive strength in MPa				
	3D	7D	28D	56D	90D
N	21.56	29.43	45.88	45.78	48.24
N10	20.78	33.34	50.24	56.56	64.43
N20	21.62	30.79	44.67	49.43	54.67
N30	19.28	25.95	38.56	43.44	46.78
N40	17.89	20.35	33.67	36.78	39.44

Water Absorption Test:
The absorption values of mortars were shown in fig. 4. It was clear that presence of pozzolanic material in cement mortar decreased the water absorption value. Micro/nano-FA was more effective in reduction of permeability than that of normal cement mortar. The following two phenomena can be distinguished for decreasing of permeability caused by using nano-FA:
(i) Pore filling effect of micro/ nano particles to the pores of sand, cement and also it blocks the passages connecting capillary pores and water channels in cement paste
(ii) Produce a large number of C-S-H gel products on hydration of micro/ nano particles and make a more homogenous distribution of C-S-H gel and hence less pore structure.

Water absorption was reduced as on adding of Micro/nano fly ash, for mortar mix N10, the percentage reduction of water absorption was 21%. Similarly for other mixes the percentage reduction of water absorption were found to be 8%, for N20 specimen.

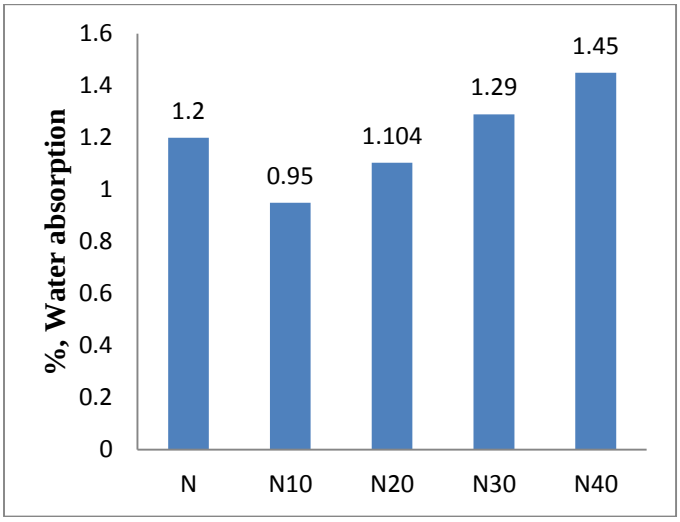

Fig. 4. percentage of water absorption of mortar mixes.

CONCLUSION:
From the experimental data, the following final points are to be derived :

- After 10 hr of grinding in mechanical milling the sample reaches nearly nano level as seen in XRD and SEM Fig. 1 and 3.
- As on adding of micro/ nano fly ash the water demand increased and similarly the water demand increases with respect to addition of nano fly ash.
- The compressive strength of the N10 was found to be highest with respect to other GFA blended cement samples as mentioned in Table 4. With the addition of GFA to cement the blended mortar samples improved their strength characteristics. At 10% GFA the strength was improved by 10%, 24% & 34% than normal mortar samples after 28, 56 & 90 days of curing respectively.
- Percentage of water absorption of N10 blended mortar specimen absorbs 21% less water than that of other samples.
  In higher percentage of GFA samples showed less refinement of the pore structure, which leads less strength.

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