Influence of Nano-Metakaolin Proportions on the Strength Performance of Fly Ash Based Geopolymer Mortar

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***Abstract*–Geopolymer mortar is a new binding material that acts as a substitute to cement mortar and is based on the geopolymerisation process which can convert silica and alumina rich waste materials into useful construction materials. With respect to utilization of waste materials and the environmental benefits, geopolymer binder is better as compared to cement mortar. Not only this, waste materials can be blended with nano materials for improving strength properties. Therefore, to find out the variations in compressive strength of fly ash based geopolymer mortar, this experimental study has been conducted using 12 M alkali activator solution and different proportions of nano-metakaolin (0%, 2%, 4%, 6%, 8% and 10% of fly ash). In this study, compressive strength of these samples was determined using Universal Testing Machine, whereas, composition of geopolymer mortar was checked using X-ray diffraction (XRD) test. It is found that geopolymer mortar containing 4% proportion of nano-metakaolin showed considerable improvement in compressive strength at 28 days curing period.**

Keyword: ***Geopolymer mortar, nano-metakaolin, compressive strength, environmental safety, X-ray diffraction (XRD) test***

I. Introduction

In current scenario, advancement in industrialization has resulted in the grave repercussions on the environment and it has become a grimmer issue for the sustainable growth of any nation. Considering the cement manufacturing industry, for instance, it has a big share towards the rising level of green house gases [1]. Therefore, it is the need of the hour to develop an environment-friendly and cost effective binder that could act as substitute to cement in mortar and concrete. The main focus of most of the research and development organizations is on the potential ways of using waste from industries as a replacement of cement in construction materials [2]. In this concern, geopolymer binder comes into picture. The term geopolymerisation was given by Davidovits [3] to define the alkali activation process of base materials which are rich in alumina - silica content and results into the formation of binders. In this process, alkaline solution activates the alumina and silica present in waste materials and results in the formation of 3-dimensional polymeric chain [4]. Physical appearance and mechanical properties of the construction materials- mortar and concrete- formed through geopolymerization is quite similar to Portland cement [5]. Various research studies have been conducted on the use of fly ash as a base material in geopolymer based construction materials. It has been investigated that the synthesis of green geopolymer mortar is a sustainable approach which has potentially better mechanical strength, minimum carbon emission and is cost effective [6]. Having high content of alumina oxide (Al_2O_3) and silica oxide (SiO_2), fly ash could be taken as base material and can combine with a solution to form geopolymer binders [7]. Vijai et al. [8] evaluated the effects on the development of geopolymer concrete with change in the source, thermal power station, of fly ash. Owing to the increase in curing age and sodium hydroxide molarity, an increase in the strength in the form of compressive strength, split tensile strength and flexural strength was observed. Properties of geopolymer mortar and concrete are directly dependent upon the variables such as alkaline activator quantity, fineness of raw material, curing methods, curing temperature [9]. In addition, the strength and microstructure properties of geopolymer matrices can be further developed by incorporating nanoparticles. Various contemporary research studies have focused on the production of geopolymer mortar and concrete reinforced with various nano-materials in order to improve the performance of various construction materials [10]. Numerous types of nano-materials such as nano–silica, nano-titanium dioxide, nano-metakaolin, nano-zinc oxide, nano-alumina, nano-clay et cetera can be used for improving the strength of concrete [11].

Adak et al [12] found that the nano-silica modified geopolymer concrete exhibits better structural performance in comparison to conventional geopolymer concrete. Use of nano-alumina and nano-silica in geopolymer acts as filler, and therefore, decline the voids in geopolymer matrices [13]. Reduction in porosity and enhancement in the both flexural as well as compressive

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strength of geopolymer based on fly ash is reported with the utilization of 2% nano-clay [14]

Therefore, this research study was proposed to explore the variations in compressive strength development of fly ash based geopolymer mortar in relation with the different proportions of nano-metakaolin.

II. Methodology

A. Materials

1. Fly ash

The source material, fly ash (Class F), was obtained from the Guru Nanak Dev thermal plant, Bathinda, Punjab. In fly ash, Silicon dioxide (SiO_2), Calcium Oxide (CaO), and Loss on ignition were respectively 52.66**%,** 1.64**%,** 2.35**%**, whereas Magnesium oxide (MgO) and Sodium oxide (Na_2O) proportion was respectively 0.84**%** and 0.02**%.**

2. Sand

River sand, which was locally available, screened through 2.36 mm sieve, was taken as fine aggregate.

3. Alkaline Activator Solution

The combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) of 12 molarity was utilized as alkaline activator solution. Liquid solution of sodium silicate was used, whereas pellet form of sodium hydroxide was supplied. For one litre 12 M molarity sodium hydroxide solution, 360gm of NaOH pellets was dissolved in water.

4. Nano-Metakaolin

The nano-metakaolin for this research study was procured from Sheetal industries, Gujarat, India with the chemical content of SiO_2 as 51-54%, Al_2O_3 as 41-44%, K_2O and LOI (loss on ignition) as 1-2%, however chemicals such as Fe_2O_3 and Na_2O were respectively 0.35% and 0.13%. Apart from these proportions, MgO and CaO chemical content were also present rather in quite small quantity which is 0.065% and 0.016%, respectively.

B. Mix Proportion

In this research study, alkali activation of fly ash-based geopolymer mortar was done by 12M of sodium hydroxide. To form alkaline activator solution, 12M molarity of sodium hydroxide (NaOH) combined with sodium silicate (Na_2SiO_3) solution in the ratio of 1:2.5. The ratio of alkaline activator to fly ash and fly ash to sand was 0.40 and 1:3 respectively. Six mixes of geopolymer mortar were prepared by replacing base material, fly ash, with nano-metakaolin (0% to 10% @ 2% increment). Mix proportion of these mixes is given in Table 1.

C. Sample Preparation and Testing

1. Alkaline Activator Solution (AAS) Preparation

NaOH solution was prepared a day before casting. Both the solutions (NaOH and Na_2SiO_3) were added in a beaker to prepare alkaline activator solution.

2. Mortar Preparation

To prepare geopolymer mortar, both the source materials-fly ash and nano-metakaolin- were mixed in dry condition for 5 minutes. After that, quantity of alkaline activator solution, which is calculated as per AAS/FA ratio, was added and mixed thoroughly to obtain homogeneous paste. Later on, dry sand was added to this paste and mixed further for 3 minutes. At last, the prepared mixture was poured into standard cubes moulds of size 70.6mm x 70.6mm x 70.6mm, and to remove voids from the moulds, vibrator was used.

D. Compressive Strength Testing

For testing the compressive strength of geopolymer mortar same procedure was followed as of cement mortar [15]. All the specimens of standard size were examined for compressive strength using the universal testing machine (UTM). Before testing the specimens, the specimens were initially cured at 70°C for 24 hour temperature curing, and then ambient cured for 28 days. To determine an average three cubes of each mix were casted and tested for compressive strength.

Table 1: Mix Proportion of Geopolymer Mortar

Sample identity	Fly ash (gm)	Nano-metakaolin (gm)	Alkaline activator/ fly ash ratio	Water/ solids ratio	Sand (gm)	Alkaline activator solution (ml)	Alkaline activator	
							NaOH solution (ml)	Na_2SiO_3 Solution (ml)
FN-1	2400	0	0.40	0.30	7200	960	274.2	685.8
FN-2	2352	48	0.40	0.30	7200	960	274.2	685.8
FN-3	2304	96	0.40	0.30	7200	960	274.2	685.8
FN-4	2256	144	0.40	0.30	7200	960	274.2	685.8
FN-5	2208	192	0.40	0.30	7200	960	274.2	685.8
FN-6	2160	240	0.40	0.30	7200	960	274.2	685.8

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E. X-ray Diffraction Test Method

X-Ray Diffraction (XRD) technique is non- destructive analytical technique which is most commonly used for analyzing the crystal structure, mineral composition and unit cell dimensions of naturally existing or manufactured crystalline materials. This technique is primarily based on the Bragg's Law ($n\lambda=2d \sin\theta$). According to this law, the wavelength of electromagnetic radiation or X-ray (λ) depends on the two parameters: diffraction angle of incident ray (θ); and lattice spacing between the planes (d) in a crystalline sample.

For this research study, XRD test was conducted to determine the impact of NMK content on the microstructure properties of fly ash based geopolymer mortar. The powdered mortar samples were taken from the cubes after performing 28 days compressive strength test. Only the samples which passed through 90 µm sieve were taken to perform the test.

III. Results and Discussion

A. Compressive Strength Testing

Table 2 depicts the compressive strength results of fly ash-based mortar samples alkali activated using 12 M alkaline activator solution. At 4% proportion of NMK, considerable increase in the compressive strength was noted; however, with 10% content of NMK, compressive strength of all the samples gets reduced in comparison to control mix (FN-0) as shown in Figure 1.

Moreover, fly ash based geopolymer mortar blended with nano-metakaolin acquire most of its 28 days compressive strength after 3 days of curing. This is because the silica and alumina oxide of NMK in the mortar intensify the geopolymerization reaction.

Table 2: Compressive Strength Test Results of Fly Ash Based Geopolymer Mortar

Mix	NMK %	NaOH Molarity (M)	Compressive strength of geopolymer mortar (N/mm^2)			
			Days			
			3	7	14	28
FN-1	0		29.27	35.09	37.29	38.63
FN-2	2		32.01	38.66	40.37	42.92
FN-3	4	12	37.84	43.57	46.05	48.21
FN-4	6		35.60	38.21	39.59	40.87
FN-5	8		33.52	35.18	37.83	39.69
FN-6	10		29.05	33.28	36.20	37.27

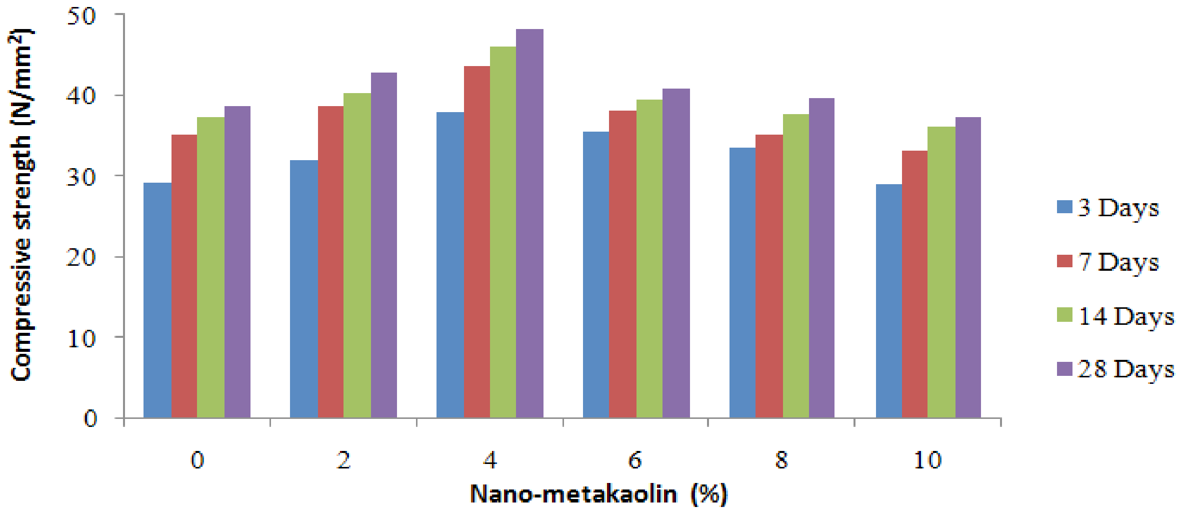

Fig.1: Effect of Nano-metakaolin on Compressive Strength of Geopolymer Mortar Alkali Activated with 12 Molar Concentration of NaOH at Different Curing Ages

B. *X-ray Diffraction Test Analysis*

To analyze the microstructure of the samples, which were initially tested for 28 days compressive strength, XRD test was used. For this, the powdered samples of FN-1 (0% NMK), FN-3 (4% NMK), and FN-6 (10% NMK), were used. The results of this test are represented as the peaks of major geopolymerization reaction products- quartz and mullite. The crystalline phases of the tested samples were indexed using Powder Diffraction Files (PDFs) on the basis of International centre of diffracted data (ICDD). The results of XRD test for mixes FN-1, FN-3 and FN-6 are shown in Fig.2, Fig.3 and Fig.4, respectively.

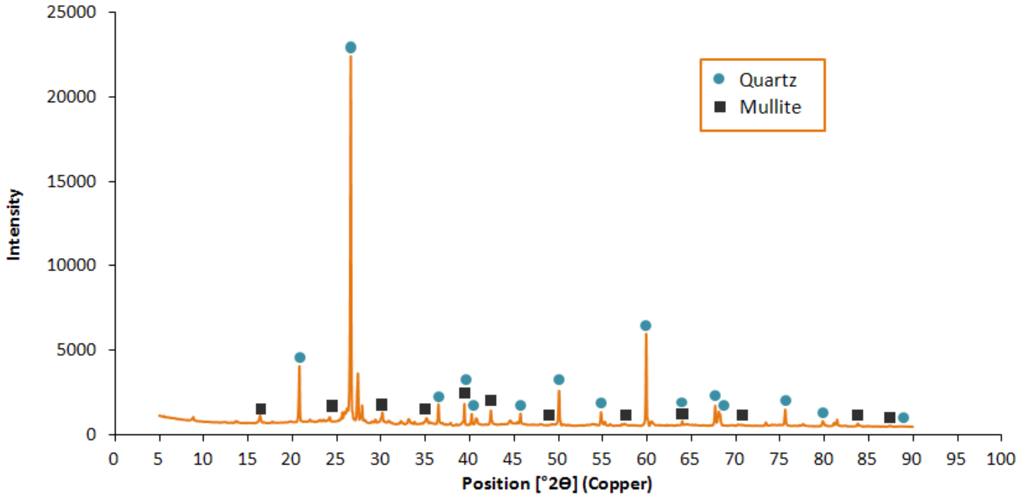

Fig. 2: XRD pattern of FN-1

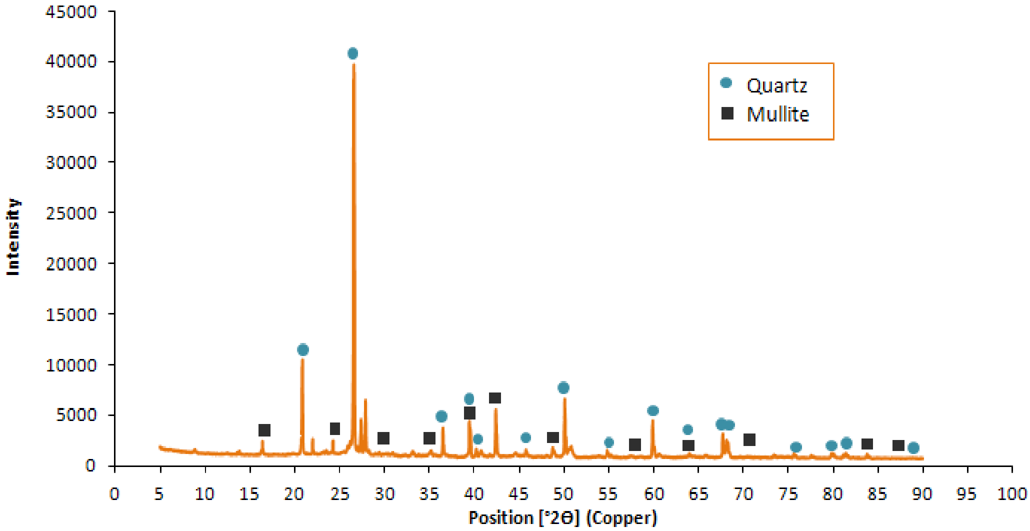

Fig. 3: XRD pattern of FN-3

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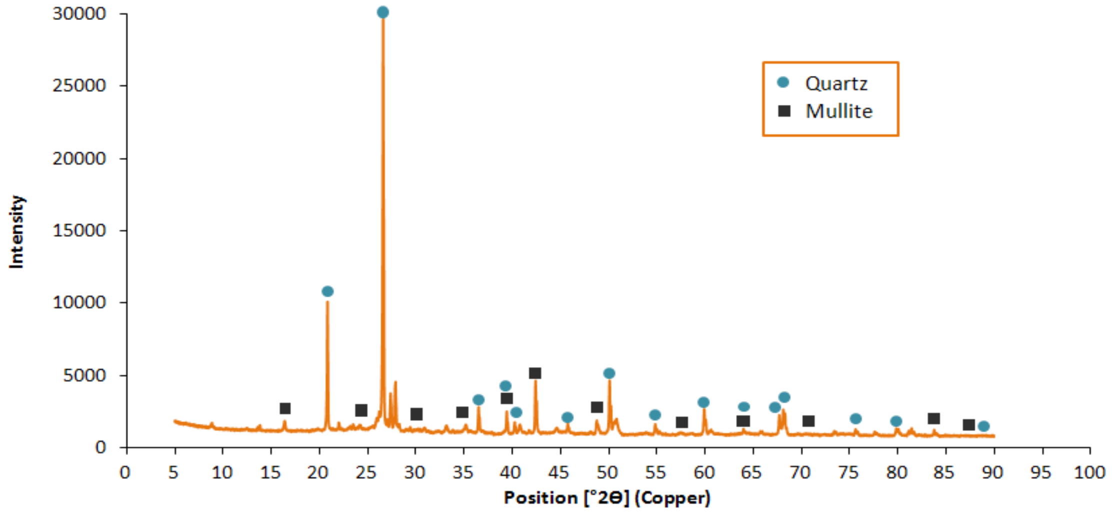

Fig. 4: XRD pattern of FN-6

The XRD test patterns of FN-1, FN-3 and FN-6 depict that quartz (SiO_2), which is the main phase in the tested samples, could be easily observed in the 2Θ range of 26º-32º. Along with this, mullite ($Al_6Si_2O_{13}$) phase is also present. The quartz peak intensities at 26.6° in the mixes FN-1, FN-3 and FN-6 are 22121.46, 39671.47 and 29671.47, respectively. Test result shows that 12M concentration of NaOH along with 4% NMK, extensively affects the filler component intensities as compared to other proportions of NMK. With this, geopolymerization reaction at 4% NMK increased significantly, and consequently, there is surge in compressive strength. With further increase in NMK proportion, intensity of peaks goes on decreasing and thereby, lowering the compressive strength.

IV. Conclusion

1. From the test results, it has been noticed that the optimum compressive strength of 48.21 N/mm^2 was obtained at 4% proportion of NMK.
2. It has also been observed that about 70-80% of 28 days compressive strength of nano-metakaolin reinforced geopolymer mortar is obtained after 3 days of ambient curing.
3. Considering the increase in the content of silica and alumina oxide for geopolymerization reaction due to increment in the percentage levels of NMK, compressive strength of mortar increases.
4. XRD test results shows that at 4% replacement of NMK, the intensities of filler components get increased, which stimulate the geopolymerization process. This results in the formation of more compact structure. However, addition of nano-materials in high quantity (beyond 4%) results in decline in the intensity of peaks and therefore, compressive strength starts decreasing.

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