

Metakaolin Based Geopolymer Mortar Reinforced with Multi-Walled Carbon Nanotubes - A Case Study

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Authors' contributions

This work was carried out in collaboration among all authors. Authors PK and JS designed the study. Author PK performed the experiments and wrote the first draft of the manuscript. Authors JS, MK and RJ managed the analyses of the study. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CJAST/2020/v39i4131119

Editor(s):

(1) Dr. Wen Shyang Chow, Universiti Sains Malaysia, Malaysia.

Reviewers:

(1) Mohammad Asadi, Islamic Azad University Tehran Science and Research Branch, Iran.

(2) Yusrina Mat Daud, Universiti Malaysia Perlis, Malaysia.

(3) Ghasan Fahim Huseien, National University of Singapore, Singapore.

Complete Peer review History: <http://www.sdiarticle4.com/review-history/63650>

Original Research Article

Received 28 September 2020

Accepted 04 December 2020

Published 21 December 2020

ABSTRACT

Metakaolin based geopolymer mortars are presently considered as a feasible substitute to Ordinary Portland Cement mortar due to its various benefits. The present experimental investigation is planned by varying the concentrations of sodium hydroxide as 8M, 10M and 12M along with the variation of multi-walled carbon nanotubes (0, 0.25%, 0.50%, 0.75% and 1% by weight of the binder). For each specimen, the compressive strength was determined at the curing ages of 3, 7 and 28 days. The results clearly indicate that the incorporation of multi walled carbon nanotubes (MWCNTs) in the geopolymer matrixes enhances the compressive strength. Transmission electron microscope (TEM) was used to depict the microstructure and morphology of MWCNTs. The ultimate compressive strength was obtained by employing 12M concentrated sodium hydroxide solution along with 0.5% of MWCNTs in geopolymer mortar. The values of integral absolute error were computed for all the curing ages. All the values lie within the acceptable range (0 to 10%).

Keywords: Compressive strength; geopolymer; metakaolin; multi-walled carbon nano tubes.

1. INTRODUCTION

For the production of cement, limestone along with the source of silica is heated in a kiln at very high temperatures (over 1350°C). The manufacturing of cement needs enormous energy and thereby releases carbon dioxide of approximately the same amount as cement. Therefore, it has become mandatory to mitigate the production of cement by replacing it with alternative materials such as fly ash, bottom ash, metakaolin, ground granulated blast furnace slag, rice husk ash, corn cob ash etc. which has the similar properties of alumina and silica (main ingredients of cement). In Ordinary Portland Cement (OPC), deterioration is common and hence it becomes essential to replace the cement with alkali activated materials [1]. Owing to these reasons, the new concept of geopolymer is introduced in which alkali activated materials are incorporated instead of cement. This concept of geopolymer was first introduced by Davidovits in 1978 to signify the wide range of materials which are the members of inorganic polymers family [2].

When geopolymers are used instead of cement, the emission of carbon dioxide reduces up to 80% [1,3]. For the geopolymerization process, geopolymer binders (fly ash, rice husk ash, ground granulated blast furnace slag, metakaolin etc. which are rich in alumina and silica) are used as a source material and sodium or potassium based alkaline solutions are used to activate the geopolymer binders. The mortar and concrete prepared by the geopolymerization process exhibit equivalent mechanical properties to those obtained from OPC. Metakaolin is essentially an anhydrous aluminosilicate material formed by the calcination of kaolin clays. Due to its disordered structure, it possesses a huge reactive potential when it is activated in alkali solution [4-7] or calcium hydroxide in presence of water [4]. Metakaolin based geopolymer exhibit excellent properties such as compressive strength [8] and it also improves the microstructure of geopolymer. However, some geopolymer do not have sufficient strength and sometimes also show brittle behavior. Therefore, they are inappropriate for the safe structural design applications [9,10]. As a result, it is essential to enhance the fracture properties of these geopolymer.

For the enhancement in the properties of geopolymer/cement/concrete, nano materials were used. These have the great potential to act

as filler in concrete which reduces the chances of permeability [11]. These are very promising for the production of high performance structural and multi functional nano composite materials [12]. Carbon nanotubes (CNTs) are one of the nano materials which have the cylindrical shell type shapes made up of carbon atoms arranged in episodic hexagonal arrangements. CNTs experience high electrical and mechanical properties therefore these are very advantageous for producing fiber reinforced concrete [13].

Multi-walled carbon nanotubes (MWCNTs) are used as reinforcement in high performance concrete. The enhancement in the properties of geopolymer using MWCNTs depends upon the uniform dispersion of MWCNTs. The alkaline solution used to process geopolymer can possibly upgrade the collaboration of MWCNTs with the geopolymer matrix; thereby affecting its dispersion within the geopolymeric network [9,14,15]. The surfactant is also used for the uniform dispersion of MWCNTs which helps in augmenting the properties of geopolymer. MWCNTs were used to delay the promulgation and development of cracks in mortar at the nano scale [13]. The effect of MWCNTs on the properties of fly ash based geopolymer was investigated by Safi et al. [14]. In their research, different concentrations of MWCNTs such as 0.0%, 0.1%, 0.5% and 1.0% by weight were utilized. According to the analysis, geopolymer matrix containing 0.1% and 0.5% of MWCNTs were uniformly distributed in the matrix while 1% of MWCNTs were poorly distributed and rigorously agglomerated. The experimental investigation also indicated that the flexural strength, Young's modulus and flexural toughness increased by 160%, 109% and 275%, respectively with the addition of MWCNTs [14]. The properties of cement matrix containing MWCNTs strongly depended upon the dispersion of MWCNTs and its bonding with the cement matrix [16]. Moreover, it was demonstrated by the previous researchers [17-19] that the addition of less than 1% of MWCNTs can greatly enhance the mechanical properties of the composites. Consequently, one of the main reasons of using MWCNTs in mortar and concrete is to improve the compressive strength and durability properties. The objective of this research was to study the compressive strength of metakaolin-based geopolymer mortar reinforced with MWCNTs (0%, 0.25%, 0.50%, 0.75% and 1%) by weight of the binder for three different molar concentrations (8M, 10M and 12M) of NaOH.

2. MATERIALS AND METHODS

2.1 Materials

Following are the materials used for the geopolymerization process:

2.1.1 Metakaolin

Metakaolin is an anhydrous calcined form of the kaolinite which is obtained by burning of kaolin clay at a temperature between 600°C to 800°C. In this investigation, metakaolin obtained from Kaolin Techniques Private Limited, Gujarat was used as source material for geopolymerization. The metakaolin used was in creamish pink colour and the chemical composition of metakaolin is given in Table 1.

2.1.2 Multi-walled carbon nanotubes (MWCNTs)

In this experimental study, MWCNTs produced by chemical vapor deposition method was used. They were obtained from Platonic Nanotech Private Limited, Jharkhand. The morphology and micro structure of MWCNTs was studied by Transmission electron microscope (TEM) at a scale of 100 nm is shown in Fig. 1, which shows that these consists of multi walls and the boundaries between the walls are clear [20]. The properties of MWCNTs are given in Table 2.

2.1.3 Superplasticizer

Superplasticizer was used for the homogeneous dispersion of MWCNTs in the geopolymer matrix. In this study, polycarboxylate based superplasticizer was used and the proportion of it used in this study was 5% by weight of the binder. It was obtained from the Asian Chemical

Industries, Ludhiana. The properties of superplasticizer are given in Table 3.

2.1.4 Alkaline activator solution

Alkaline activator Solution acts as a major constituent in the process of geopolymerization. Generally, different alkaline activators such as potassium hydroxide, potassium silicate, sodium hydroxide and sodium silicate were used for the geopolymerization process. In this study, sodium silicate and sodium hydroxide were used as alkaline activator. These were supplied by the local supplier of Ludhiana. Sodium hydroxide used for this study was available in pellets form while sodium silicate was available in liquid form which consists of Na₂O in the range of 7.5-10% and SiO₂ in the range of 25-28%. For this experimental work, 8 M, 10 M and 12 M solution of sodium hydroxide was prepared by dissolving respectively 320 grams, 400 grams and 480 grams sodium hydroxide pellets in water for the solution of one litre. The alkaline activator solution is used to be prepared one day prior the day of casting the cube specimens because during the mixing process of hydroxides and silicates, significant amount of heat is generated.

2.1.5 Fine aggregates

Fine aggregates conforming to zone II as per IS 383 – 1970 were used for the experimental work.

2.1.6 Water

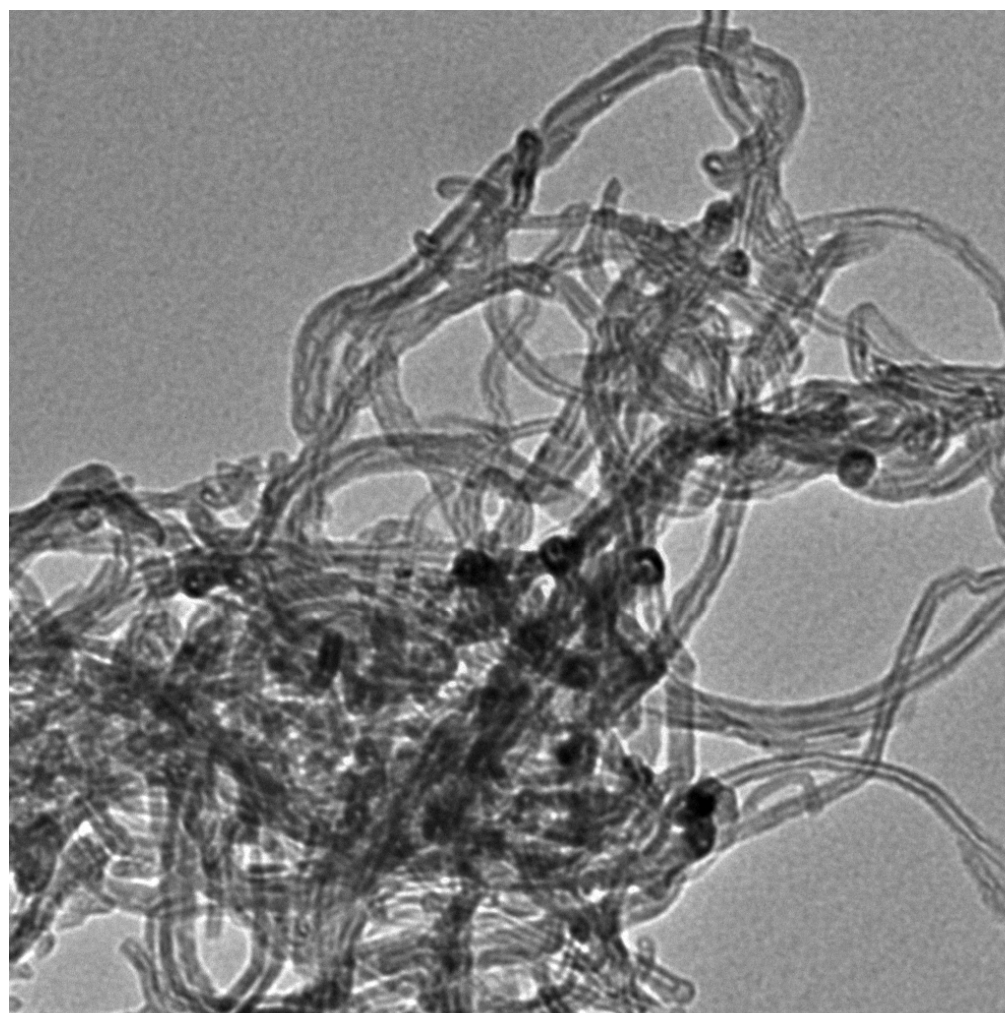
Water is a major constituent used for the casting of cube specimens. The fresh and clean tap water was used in this study for the preparation of geopolymer mortar. As per IS 456-2000, the water used was free from any organic matter, silt, oil and acidic material.

Table 1. Chemical composition of metakaolin

Sr. no.	Chemical composition (%)	Values
1.	SiO ₂	52 ±1%
2.	Al ₂ O ₃	42 ±1%
3.	TiO ₂	0.5 Max.
4.	Fe ₂ O ₃	< 1.3%
5.	CaO	< 0.5%
6.	MgO	< 0.5%
7.	Na ₂ O, K ₂ O	0.5 – 2.5%
8.	Loss on ignition	0.8 - 1.2

Table 2. Properties of MWCNTs

Sr. no.	Specification	Values
1.	Physical form	Fluffy, Very light powder
2.	Colour	Black
3.	Diameter	5~15 nm
4.	Length	10~15 microns
5.	Purity	97%
6.	Metal content	2%
7.	Ash	1%
8.	Specific Surface Area	220 m ² /g
9.	Bulk Density	0.06~0.09 g/cm ³



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 Spot number=5
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 Azim angle=0.0

Fig. 1. TEM image of MWCNTs

Table 3. Properties of superplasticizer

Sr. no.	Specification	Values
1.	Aspect	Light Brown Liquid
2.	Relative density	1.08±0.01
3.	pH	≥ 6
4.	Chloride ion content	< 0.2%

2.2 Dispersion of MWCNTs

The dispersion of MWCNTs was attained with the help of bath sonication which helps for overcoming the Van der Waals interactions between the CNTs. For the effective dispersion of MWCNTs, an aqueous solution of water and surfactant was prepared and then the measured amount of MWCNTs was added to the solution.

In this study, polycarboxylate based superplasticizer used as a surfactant for the homogeneous dispersion of MWCNTs. The solution was ultrasonicated in water bath for about 1 hour. Fifteen mixes of geopolymer mortar were prepared by employing different proportions of MWCNTs (0%, 0.25%, 0.50%, 0.75% and 1% by weight) for 8M, 10M and 12M. The sonicator apparatus used in this study is shown in Fig. 2.



(a) Top view



(b) Front View

Fig. 2. Sonicator apparatus

2.3 Experimental Procedure

The mixes of geopolymer mortar were prepared by using standard ratio of 1: 3 that is, one part of source material and three parts of fine aggregates. For all the samples, alkaline activator solution to binder ratio, sodium silicate to sodium hydroxide and water to solids ratio was fixed as 0.60, 2.5 and 0.60 respectively. The details of all the mixes are shown in Table 4.

2.3.1 Mixing, casting and curing of geopolymer mortar

In this investigation, metakaolin was firstly activated by employing alkaline solution and the mixing was continued for about 2-3 minutes to ensure the homogeneous mixing. After this, fine aggregates were added to the mix and again mixed it properly. Later on, the ultrasonicated solution in addition to extra water was added to the alkali activated mix and then the mixing was continued for about 4-5 minutes to ensure the homogeneity.

Then the freshly prepared geopolymer mortar was poured into cubes of standard size of 70.6 mm × 70.6 mm × 70.6 mm for the determination of compressive strength [21]. The mortar was poured in the cube specimens in two layers by tamping each layer for about 25 times with the usage of tamping rod. Consequently, all the cube specimens were placed on a vibrator for about 2 minutes to remove the excess air voids. After one hour of casting as shown in Fig. 3, the cube specimens were placed in an oven for 24 hours at 40°C for thermal curing. Then the cubes were removed and demoulded after 24 hours from thermal curing and then placed at room temperature for ambient curing until the day of testing.

The compressive strength testing of all the specimens was done using universal testing machine (UTM) at the age of 3, 7 and 28 days of curing. Three iterations of geopolymer mortar for different ages were made for different concentrations of MWCNTs and the mean of these iterations was considered as the compressive strength of that mix.

Table 4. Metakaolin based geopolymer mix proportions

Mix	M (M)	MK (g)	MWCNTs (%)	AAS		SP (g)	FA (g)	W (g)
				SH (g)	SS (g)			
1	8	1800	0	308.57	771.43	0	5400	597.89
2	8	1800	0.25	308.57	771.43	90	5400	597.89
3	8	1800	0.50	308.57	771.43	90	5400	597.89
4	8	1800	0.75	308.57	771.43	90	5400	597.89
5	8	1800	1.00	308.57	771.43	90	5400	597.89
6	10	1800	0	308.57	771.43	0	5400	622.08
7	10	1800	0.25	308.57	771.43	90	5400	622.08
8	10	1800	0.50	308.57	771.43	90	5400	622.08
9	10	1800	0.75	308.57	771.4	90	5400	622.08
10	10	1800	1.00	308.57	771.43	90	5400	622.08
11	12	1800	0	308.57	771.43	0	5400	647.06
12	12	1800	0.25	308.57	771.43	90	5400	647.06
13	12	1800	0.50	308.57	771.43	90	5400	647.06
14	12	1800	0.75	308.57	771.43	90	5400	647.06
15	12	1800	1.00	308.57	771.43	90	5400	647.06

Note: M = Molar concentration of NaOH; MK = Metakaolin; MWCNTs = Multi-walled carbon nanotubes; AAS = Alkaline activator solution; SH = Sodium hydroxide; SS = Sodium silicate; SP = Superplasticizer; FA = Fine aggregates; W = Water

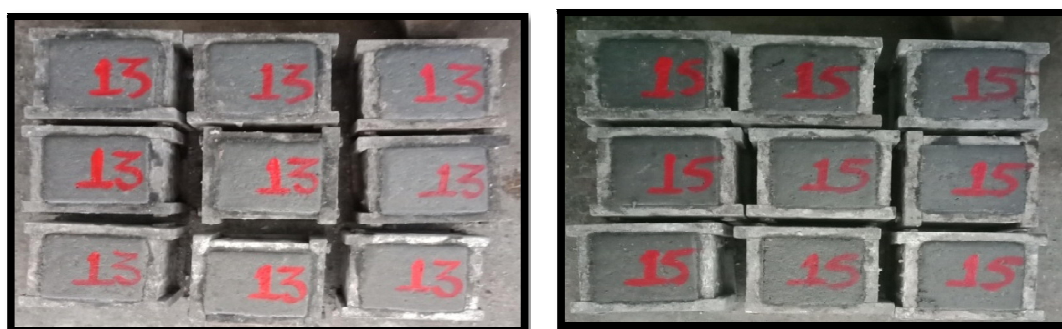


Fig. 3. Casting of samples

3. RESULTS AND DISCUSSION

Table 5 indicates the results obtained from the compressive strength testing of metakaolin based geopolymer mortar incorporating MWCNTs at the age of 3, 7 and 28 days of curing. The concentration of sodium hydroxide in the aqueous stage of the geopolymeric framework follows up on the dissolution process as well as on the bonding of solid particles in the structure [22]. The utilization of high molar concentration of NaOH prompts more noteworthy dissolution of the solid materials and enhances geopolymerization response and henceforth higher compressive strength is attained [23]. From the observations recorded, it was found that the compressive strength of metakaolin based geopolymer mortar increases with the increase in the molar concentration. But the compressive strength of metakaolin based geopolymer mortar in all the molarities showed an increasing trend up to 0.50% of MWCNTs and

then decreased. The optimum strength of geopolymer mortar for 8M, 10M and 12M corresponds to 0.50% of MWCNTs. The compressive strength of geopolymer mortar at 0.50 % of MWCNTs as shown in Figs. 4, 5 and 6 for 8 M, 10 M and 12 M was 54.97 MPa, 59.34 MPa and 63.34 MPa respectively for 28 days of curing. While the compressive strength of metakaolin based geopolymer mortar at 28 days with 1% MWCNTs was found to be 48.67 MPa, 52.53 MPa and 56.70 MPa in 8M, 10M and 12M which was slightly lower than 0.5% MWCNTs but it remained higher than the control mixes. Similar investigation was reported by Abbasi et al. [9] in which the compressive strength of metakaolin based geopolymer paste at 1% MWCNTs was lower than the 0.50% but higher than the reference mix. It is vital to note that the compressive strength of geopolymer mortar increase with the augmentation in molar concentration [2,24].

Table 5. Compressive strength of metakaolin based geopolymer mortar

Mix	Molar concentration	MWCNTs (%)	Compressive strength (MPa)		
			3 days	7days	28 days
1		0	31.88	36.95	41.98
2		0.25	37.60	42.35	48.45
3		0.50	43.43	48.35	54.97
4	8M	0.75	39.75	45.13	49.80
5		1.00	37.82	44.38	48.67
6		0	35.55	39.67	46.09
7		0.25	40.26	46.43	52.44
8	10M	0.50	46.78	53.57	59.34
9		0.75	42.85	47.80	53.95
10		1.00	40.52	46.70	52.53
11		0	39.37	44.72	50.01
12		0.25	44.25	49.23	56.36
13	12M	0.50	49.42	55.81	63.34
14		0.75	45.79	50.44	57.96
15		1.00	44.08	49.14	56.70

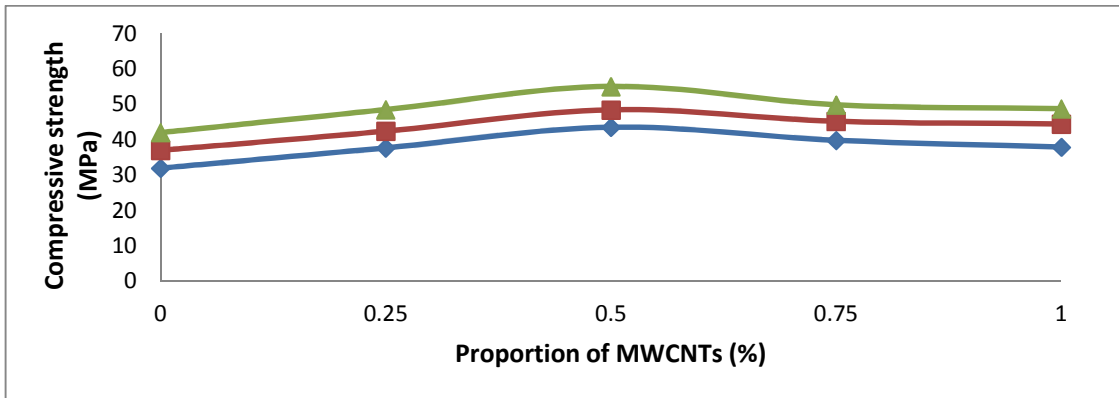


Fig. 4. Effect of MWCNTs on the compressive strength of geopolymer mortar at different ages with molar concentration of 8M

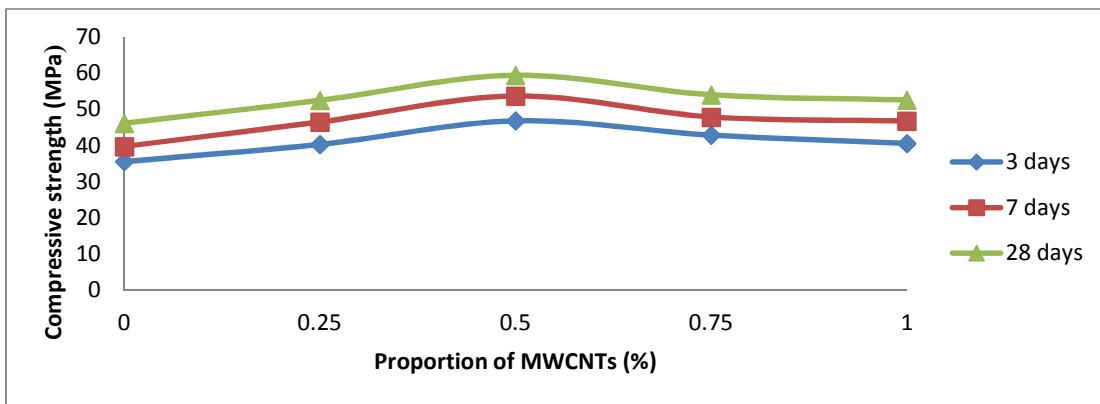


Fig. 5. Effect of MWCNTs on the compressive strength of geopolymer mortar at different ages with molar concentration of 10 M

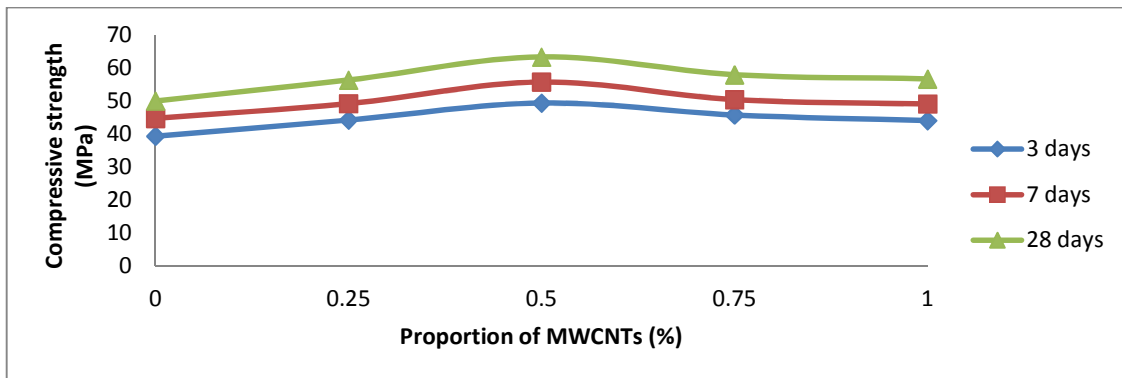


Fig. 6. Effect of MWCNTs on the compressive strength of geopolymer mortar at different ages with molar concentration of 12 M

3.1 Development of Prediction Equation for the Compressive Strength of Metakaolin Based Geopolymer Mortar

The experimental investigation clearly illustrates that the compressive strength of metakaolin

based geopolymer mortar is directly proportional to the molarity of NaOH as well as to the proportion of MWCNTs. Hence, an effort has been made to relate the different parameters with the compressive strength. Regression analysis when performed for the compressive strength of

metakaolin based geopolymer mortar gives the best fit equation for compressive strength of 3, 7 and 28 days as:

$$\sigma_c = 22.57 + 1.62 x_1 + 5.002 \quad (1)$$

$$\sigma_c = 27.75 + 1.61 x_1 + 5.75 \quad (2)$$

$$\sigma_c = 29.65 + 2.025 x_1 + 5.88 x_2 \quad (3)$$

Where,

σ_c = Compressive strength of metakaolin based geopolymer

x_1 = Molarity of NaOH

x_2 = Proportion of MWCNTs

3.2 Computation of Integral Absolute Error (IAE)

To find the expected value of the compressive strength of metakaolin based geopolymer mortar, equations (1), (2) and (3) can be used. Tables 6, 7 and 8 shows the observed and expected

values of compressive strength for different ages of curing.

The Integral Absolute Error (IAE) is used to ensure the dependability of the relationship resulting from the regression analysis. It is calculated by the following formula:

$$IAE = \left\{ \frac{\sum \sqrt{(O - E)^2}}{\sum O} \times 100 \right\}$$

Where, O_i is the experimentally observed value and E is the expected value determined from the analysis.

If the integral absolute error value is zero, then the experimentally observed values are equal to the expected values determined from the regression equation. In this analysis, the IAE values are 5.99%, 5.7031% and 5.6208% for the compressive strength of metakaolin based geopolymer mortar at 3,7 and 28 days which are in the acceptable range that is, from 0 to 10%.

Table 6. Integral Absolute Error (IAE) for the regression analysis of compressive strength at 3 days

Mix	Observed compressive strength (O) (N/mm ²)	Estimated compressive strength (E) (N/mm ²)	Residual (O - E)	$\sqrt{(O - E)^2}$	$\sqrt{(O - E)^2} / \sum O_i$
1	31.88	35.53	3.65	3.65	0.005893
2	37.6	36.78	-0.82	0.82	0.001324
3	43.43	38.03	-5.4	5.4	0.008719
4	39.75	39.28	-0.47	0.47	0.000759
5	37.82	40.53	2.71	2.71	0.004376
6	35.55	38.77	3.22	3.22	0.005199
7	40.26	40.02	-0.24	0.24	0.000388
8	46.78	41.27	-5.51	5.51	0.008896
9	42.85	42.52	-0.33	0.33	0.000533
10	40.52	43.77	3.25	3.25	0.005247
11	39.37	42.01	2.64	2.64	0.004263
12	44.25	43.26	-0.99	0.99	0.001598
13	49.42	44.51	-4.91	4.91	0.007928
14	45.79	45.76	-0.03	0.03	4.84E-05
15	44.08	47.01	2.93	2.93	0.004731
	$\sum O_i = 619.35$				$\frac{\sum \sqrt{(O-E)^2}}{\sum O_i} = 0.059902$

Hence IAE = 0.059902 × 100 = 5.99%

Table 7. Integral Absolute Error (IAE) for the regression analysis of compressive strength at 7 days

Mix	Observed compressive strength (O) (N/mm ²)	Estimated compressive strength (E) (N/mm ²)	Residual (O – E)	$\sqrt{(O - E)^2}$	$\sqrt{(O - E)^2} / \sum O_i$
1	36.95	40.63	3.68	3.68	0.005252
2	42.35	42.07	-0.28	0.28	0.0004
3	48.35	43.51	-4.84	4.84	0.006908
4	45.13	44.94	-0.19	0.19	0.000271
5	44.38	46.38	2	2	0.002854
6	39.67	43.85	4.18	4.18	0.005966
7	46.43	45.29	-1.14	1.14	0.001627
8	53.57	46.73	-6.84	6.84	0.009762
9	47.8	48.16	0.36	0.36	0.000514
10	46.7	49.6	2.9	2.9	0.004139
11	44.72	47.07	2.35	2.35	0.003354
12	49.23	48.51	-0.72	0.72	0.001028
13	55.81	49.95	-5.86	5.86	0.008363
14	50.44	51.38	0.94	0.94	0.001342
15	49.14	52.82	3.68	3.68	0.005252
$\sum O_i = 700.67$				$\frac{\sum \sqrt{(O-E)^2}}{\sum O_i} = 0.057031$	

Hence IAE = 0.057031 × 100 = 5.7031%

Table 8. Integral Absolute Error (IAE) for the regression analysis of compressive strength at 28 days

Mix	Observed Compressive strength (O) (N/mm ²)	Estimated Compressive strength (E) (N/mm ²)	Residual (O – E)	$\sqrt{(O - E)^2}$	$\sqrt{(O - E)^2} / \sum O_i$
1	41.98	45.85	-3.87	3.87	0.0049
2	48.45	47.32	1.13	1.13	0.0014
3	54.97	48.79	6.18	6.18	0.0078
4	49.80	50.26	-0.46	0.46	0.0006
5	48.67	51.73	-3.06	3.06	0.0039
6	46.09	49.9	-3.81	3.81	0.0048
7	52.44	51.37	1.07	1.07	0.0013
8	59.34	52.84	6.5	6.5	0.0082
9	53.95	54.31	-0.36	0.36	0.0005
10	52.53	55.78	-3.25	3.25	0.0041
11	50.01	53.95	-3.94	3.94	0.0050
12	56.36	55.42	0.94	0.94	0.0019
13	63.34	56.89	6.45	6.45	0.0081
14	57.96	58.36	-0.4	0.4	0.0005
15	56.70	59.83	-3.13	3.13	0.0039
$\sum O_i = 792.59$				$\frac{\sum \sqrt{(O-E)^2}}{\sum O_i} = 0.056208$	

Hence IAE = 0.056208 × 100 = 5.6208%

4. CONCLUSION

The experimental investigation depicted the change in the compressive strength of metakaolin based geopolymer mortar with the increase in molarities and MWCNTs respectively.

From the observation recorded, following conclusions are drawn as:

- The results indicated that the incorporation of MWCNTs improves the mechanical performance of the composites as

compared to the plain metakaolin based geopolymer mortar.

- The mechanical strength increased with the increase in proportion of MWCNTs up to 0.50% thereafter it started decreasing. The extent of increase/decrease in strength depends upon dispersion characteristics of MWCNTs in the matrix. If these are properly dispersed in the matrix, the strength increases or vice versa.
- The molar concentration of NaOH is directly proportional to the compressive strength as the compressive strength increases with the increase in concentration of NaOH. So, a combination of 0.50% MWCNTs and 12M NaOH is recommended to be optimum.
- The prediction equations developed for the compressive strength of metakaolin based geopolymer mortar are the best fit equations because the IAE values obtained with reference to experimental values are in the acceptable range (0 to 10%) corresponding to 3,7 and 28 days of curing.

DISCLAIMER

The products used for this research are commonly and predominantly used products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. The research was not funded by the producing company. This paper is the outcome of research carried out by the first author for her Master's Degree.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the Punjab Agricultural University, Ludhiana for providing the facilities. The statements made herein are solely the responsibility of the author(s).

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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