Piezo-resistive Properties of Polymer based cement nano composites

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Abstract. The nano technology has potential applications in development self-sensing smart materials. These nano materials is used in cement matrix or polymer base to improve the mechanical and piezo electrical properties of composites. In the present study, the graphene and carbon nano tube are added in matric, which acts as the conductive fibre into polymer cement composites. Cement based polymer composites are prepared by adding carbon fibre, epoxy resin, carbon nano tube and graphene as conductive filler in matrix to prepare the distinctive specimens for experimental investigations. The electro mechanical test are carried out to assess the piezo electrical properties of these composites subjected gradual increase of mechanical loading such as flexural and compression. The present study describes the self-sensing piezo-resistive capability of composite to monitor the strain in structures. The experimental test elucidates the addition of epoxy in cement matrix enhance the mechanical properties while carbon nano tube and graphene proves to be promising smart self-sensing materials in composites by measuring the variation in stress at critical regions. The SEM with EDX analysis shows that epoxy resin act as binding agent which hinder the cracks propagations and improves the mechanical properties of composites.
Key words *: Epoxy cement nano composite, carbon nano tube, graphene, piezo electrical properties , mechanical properties*

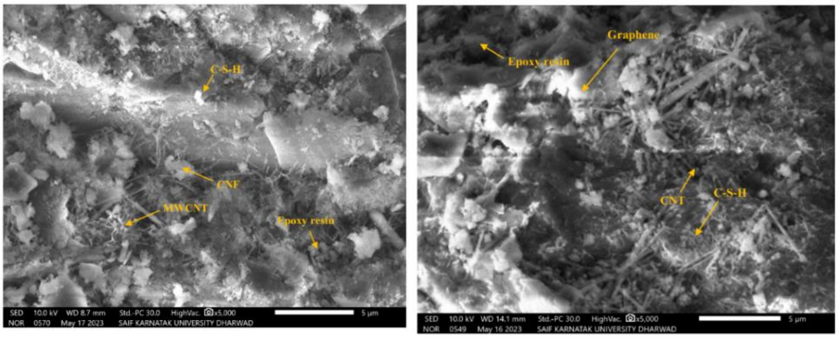
1 Introduction

The structure's overall performance and service life are extremely significant and vital in terms infrastructures integrity. It is important to find the earliest fractures and failures in the structure since other unexpected damage that may occur during the lifespan of structures such as environmental impact, inadequate maintenance, and excessive loading causing the failure of structures. Hence it become essential to identify the damage and cracks at initial level to improve the service life and ensure continuous monitoring system. The advancement of nano technology enables the development self-sensing a structural material to detect its own state, including damage, temperature, damage-related strain, stress and stress[1-3]. The many researchers made an attempt to convert cement as self-sensing materials with the applications of nano materials such Carbon nano tube (CNT), Graphene (Gr), carbon black etc.[4-5] The size, shape and quantity of these nano materials influence the conductivity of cement composites. These composites also referred as piezoresistive composites as electrical resistance is corelated with respect to applied mechanical stress[6-8].
Initially Schulte and Baron in 1988 reported that self-sensing property of construction materials is accomplished by including the carbon fiber due to their intrinsic property of electrical resistivity [9]. The recent studies have focused on novel approaches of developing smart composites using epoxy resin as a concrete binder to form the good bonding agent in matrix. For examples, thermoplastic polymer consisting of petroleum and epoxy resin is employed in the production of coating resistance [10-13]. Epoxy polymer composite exhibits

the greater strain and chemical resistance, wear resistance, electrical insulation, and waterproofing capabilities. Hence, Epoxy polymers found be various application in manufacturing construction materials to improve their mechanical properties[14-15]. Epoxy has a wide deformation range, great chemical resistance, wear resistance, electrical insulation, and waterproofing capabilities[16-18]. The Epoxy used conductive particles in cement matrix to minor the compressive strain in concrete structures. Such kind of composites eliminate the effects of humidity and polarization. The epoxy resins with hardener effectively binds in concrete which proves them to be better than other polymers. The epoxy resin cement composites improve the compressive and flexural strength[19-21]. Djabali explained the effect of the water absorption and temperature variation causes the changes of electrical resistance of the carbon black (CB) and CNTs filled epoxy composites under compression loading.[22] According to studies conducted by Park et al., it had revealed that CNF-containing composites with high aspect ratios shown self-sensing abilities to those with low aspect ratios[23]. In the present work carbon fibers, carbon nanotube and graphene are used as conductive fiber and their influence on piezoelectrical properties of matrix composite are investigated. This research is also extended to know the effect of epoxy resin as filler in cement composite on mechanical and electrical properties. Hence various composition cement samples are prepared with above materials and experiment tests are performed.

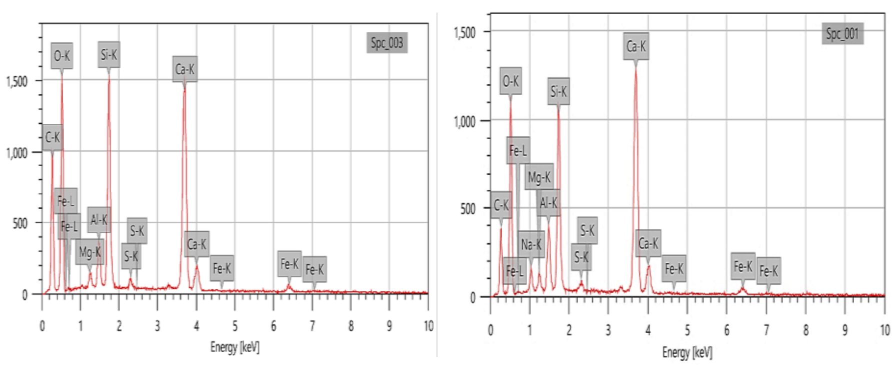
2 Microstructural Interaction of Composites

Microstructural characterization is carried out using scanning electron microscopy and energy dispersive X-ray spectroscopy technique to study the morphology and chemical composition of samples. Microstructural analysis explains the blend of CNT and epoxy into cement matrix and their influence on the properties of composites. Figure 1, Indicates the dispersion of MWCNT and Graphene at nanoscale. Nano particles clumps is observed at some place after ultrasonication. The interfacial area is reduced by the development of CNT clusters, which might decrease the chemical and physical bonding between epoxy and MWCNT. Also, at some places agglomerations of nano materials were noticed due to interparticle van der Waal force. It is also observed that MWCNT and Graphene nano particles are uniformly distributed within epoxy. The numerous interactions between epoxy and MWCNTs can improve the transfer of applied load which preventing further crack development within composites [24]. The uniform distribution of these nano particles forms conductive path hence improves the self-sensing capabilities [25]. Figure 2 it indicates that higher peaks were observed for calcium (Ca), alumina (Al), oxides (O) and Carbon(C). The presence of calcium, alumina is responsible increasing the compressive strength of composite. The presence of Carbon atoms is due to the addition of nano materials which is responsible for piezo electrical properties [26].



a. Cement with graphene and CNF with epoxy resin composite b. Cement with MWCNT and CNF with epoxy resin composite

Fig. 1. SEM Analysis of composite.



a. Cement with graphene and CNF with epoxy resin composite b. Cement with MWCNT and CNF with epoxy resin composite

Fig. 2. EDS test result of composite sample containing graphene, CNF and Epoxy

3 Experimental details

3.1 Materials

The materials employed in this investigation are Ordinary Portland Cement (OPC) 43 grade, confirming to IS 269:2015, river sand- Zone III are used to prepare the cement mortar matrix. The MWCNT or graphene, CNF are procured from manufacture and their physicochemical properties are shown in Table 1. Table 2 and 3 presents the properties of epoxy resin L12 and hardener K6 which used as polymer. Aluminium mesh and copper wires were used to measure the electrical resistance of the sample using digital multimeter. Since nanoparticles possess high degree conductivity, greater strength and stiffness, hence MWCNT or graphene and carbon nano-fiber are used as conducting fillers ad fibers in the composite.

Table 1. Properties of Nano Composites

Properties	MWCNT	CNF	Graphene
Carbon Purity	>95%	Up to 95%	>98%
Diameter	10μ	8μm	5-10μ
Density	0.14 g/cc	-	0.24 g/cc
Conductivity	106-107 S/m	-	1500 S/m

Table 2. Properties of epoxy resin

Parameters	Value
Modulus of elasticity E	5 GPa
Flexural strength	60 MPa
Tensile strength	73 MPa
Maximum elongation	4 %
Viscosity at 250 C	(12000-13000) cP
Density (g/cm3)	1.16

Table 3. Properties of Hardener K6

Parameters	Value
Appearance	Pale yellow liquid
Density at 250 C	0.95-1.1 (g/cm3)
Viscosity	5-15 (mpa. s at 250 C)

3.2 Preparation of Samples

In this research work, two distinctive cement matrix base are prepared namely cement mortar and cement mortar with epoxy resins with cement mortar ratio 1:1. The various composition samples 0.05% of carbon fibre, graphene and MWCNT by cement is added in cement mortar which act as conductive fibre in earlier case and in later case 0.5% by weight of cement, epoxy and K6 hardener is added in cement mortar along with above conductive fiber is used to prepare the various composition of cube and beam specimens. The Table 4. describes the details different specimens. The nano materials are subjected to repulsive Vander wall forces between them; hence dispersion is essential to reduce this force and ensure the uniform mixing into cement matrix. The dispersion of CF, MWCNT and graphene is carried out by probe sonicator for 60 min with the required amount of water along with epoxy resin. The sonicated nano materials are mixed homogeneously with cement mortar to prepare the specimens. The cubes are casted with having size of 70 mm X 70 mm X 70 mm as per IS code 4031 (part VI) to carry out the compression test and beam specimens are casted having size 80 mm X 20 mm X 20mm as per ASTM (C 293) Code to conduct the flexural test[27]. The 2mm thickness aluminium mesh inserted with 1mm diameter copper wire is used as electrodes to measure the electrical resistance of specimen under applied load. The Figure 3 represents the schematic diagram of electrode and specimens. The Figure 4 explain the procedure involved in the preparation of samples. These specimens are cured for ,14 and 28 days to conduct the electro mechanical test subjected compression and flexural loading.

Table 4. Specimen Composition details

Sample type	Sample	Proportions
Cube Composites	S1	Cement +0.05% wt. of Graphene* +0.05% wt. of CNT *
	S2	Cement +0.05% wt. of Graphene* +0.05% wt. of CNT*+ 0.05% wt. of Epoxy*
	S3	Cement +0.05% wt. of MWCNT* +0.05% wt. of CNT*
	S4	Cement +0.05% wt. of MWCNT* +0.05% wt. of CNT*+ 0.05% wt. of Epoxy*
Beam Composites	S5	Cement +0.05% wt. of Graphene* +0.05% wt. of CNT*
	S6	Cement +0.05% wt. of Graphene* +0.05% wt. of CNT+ 0.05% wt. of Epoxy*
	S7	Cement +0.05% wt. of MWCNT* +0.05% wt. of CNT*
	S8	Cement +0.05% wt. of MWCNT* +0.05% wt. of CNT+ 0.05% wt. of Epoxy*

Note: * Wt. % w.r.t to cement.

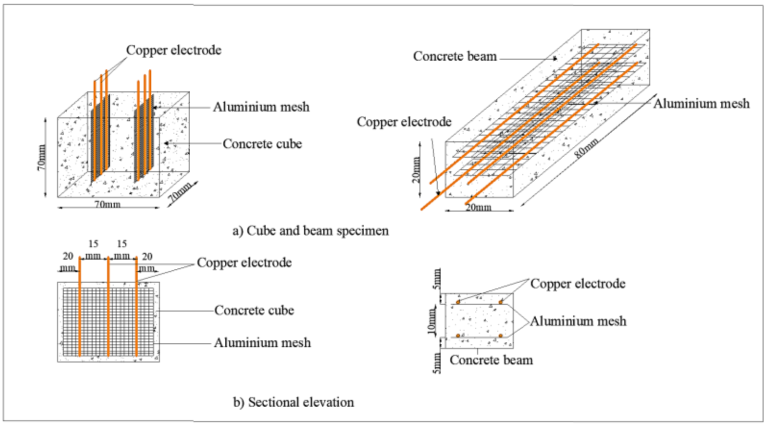


Fig. 3. Details of Specimen embedded with electrode

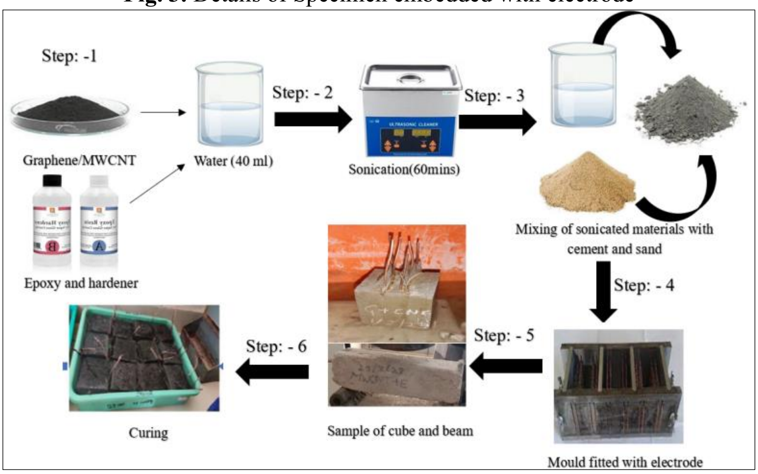


Fig. 4. Preparation process for cement epoxy nano composites

3.3 . Experimental setup

The experimental setup consists of electro-mechanical test on beam and cube specimens subjected to flexural and compression loading to evaluate the self-sensing properties. Compression load is applied on cube specimen using compression testing machine of capacity with constant loading rate of 10KN. Similarly flexural load is applied on beam specimen with the help of Loading frame of 10-ton capacity with a loading rate of 1 mm/min test to perform three-point load bending test. Figure 5 presents the experimental setup for specimens. The self-sensing properties are evaluated by correlating electrical resistance with respect to applied load. The change in electrical resistance is measured with the help of FLUKE 87V digital multimeter due to gradual increase of loads. The probes of digital multimeter is connected to the electrode of specimens to serve the above purpose[28]. The represents the experimental setup for beam and column specimens. These tests are repeated specimens cured for 7 days,14 days and 28 days to assess the effect of CF, MWCNT and graphene on electrical properties and mechanical properties.

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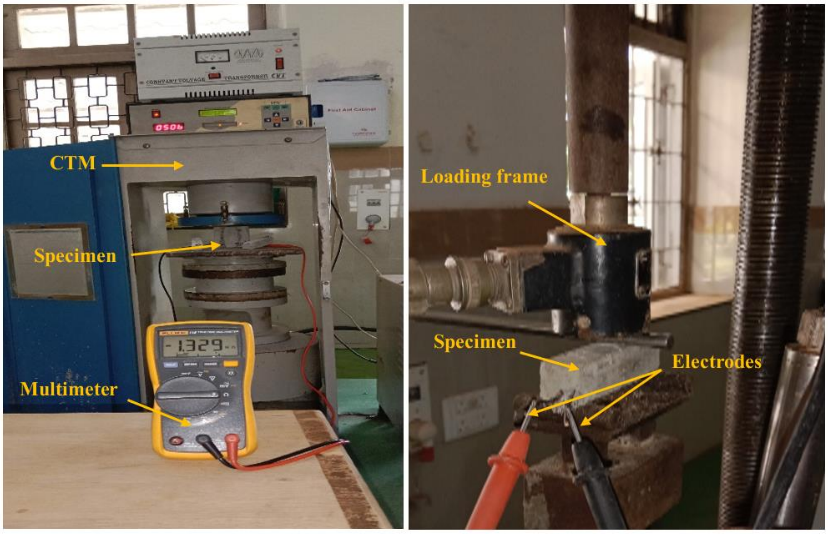

a: Compression test on cube composites b. Flexural test for beam composites

Fig. 5. Experimental setup for electromechanical test.

4 Results and discussion

From the experimental result the, variation of resistance with respect to stress due to gradual increases of load is plotted to evaluate the performance of cement epoxy nano composites in terms of electrical and machinal properties.

4.1 Piezo Electrical Properties

The self-sensing property of specimens are assessed by in terms of electrical properties. The piezo electrical property can be defined by ability of materials to shown the change in electrical resistance due to applied load. The Figure 6 indicates the change in resistance value to due applied compression load on Sample S2,S2,S3 and S4. It is observed that decrease in electrical resistance as load increases. At the failure load, the lowest resistance is observed. Due to compression loading, the distances between the nearby nano particles gets reduces leads to the decreasing in the resistance. As per tunnelling conduction theory, decrease in the tunnelling gap leads to a clear reduction in electrical resistance. Similarly, when beam samples subjected to flexural loading, the increasing in the resistance due to applied load as shown in Figure 7. The variation in resistance found to be relatively less. When composite subjected to flexural loading the, fibres get expand which increase distances between them, hence resistance is increases. Furthermore, as cracks develop and widen due to increase in load causes the less interactions between nano materials and cement epoxy matrix leads to the reduction in the number of conduction pathways[29-30]. It is observed that , the greater resistance value is found to be at 28 days of curing. The moisture content influences the electrical conductivity of samples. This is due pores in composites lose moisture content as hydration takes places which lowers the electron movement between the conductive phase and the matrix. From the Fig.S1,S3,5, and S7, it indicates that addition of epoxy makes them more conduction with addition of nano particles. The Electrons tunnel through a polymer due to lesser distance between the adjacent nano particles[30-32].

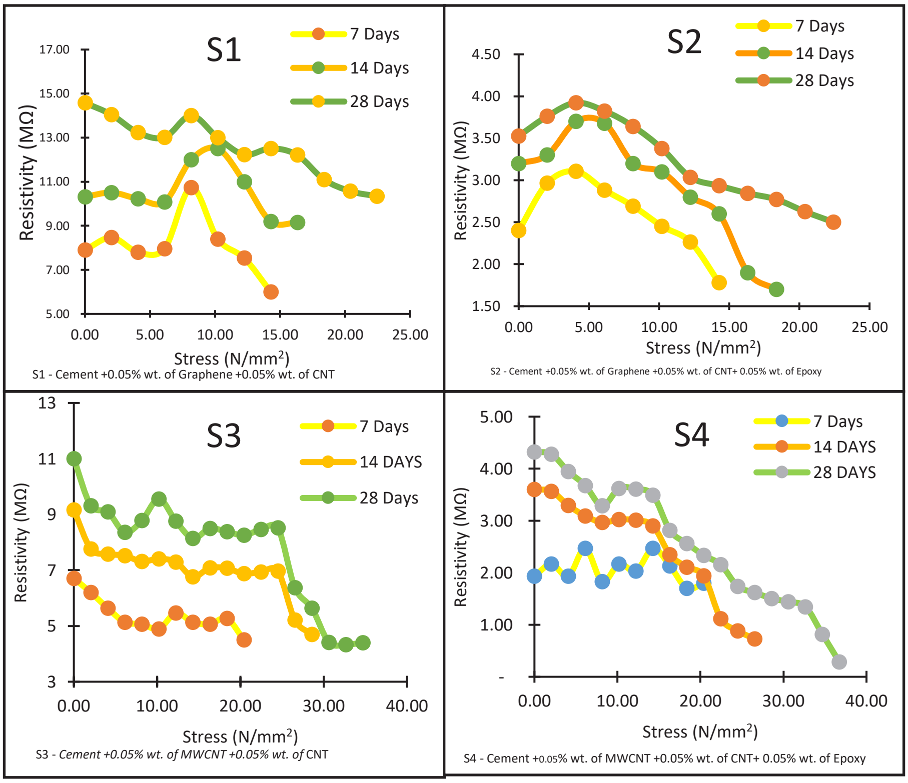

Fig. 6. Piezo -resistivity properties Cement epoxy nano composite

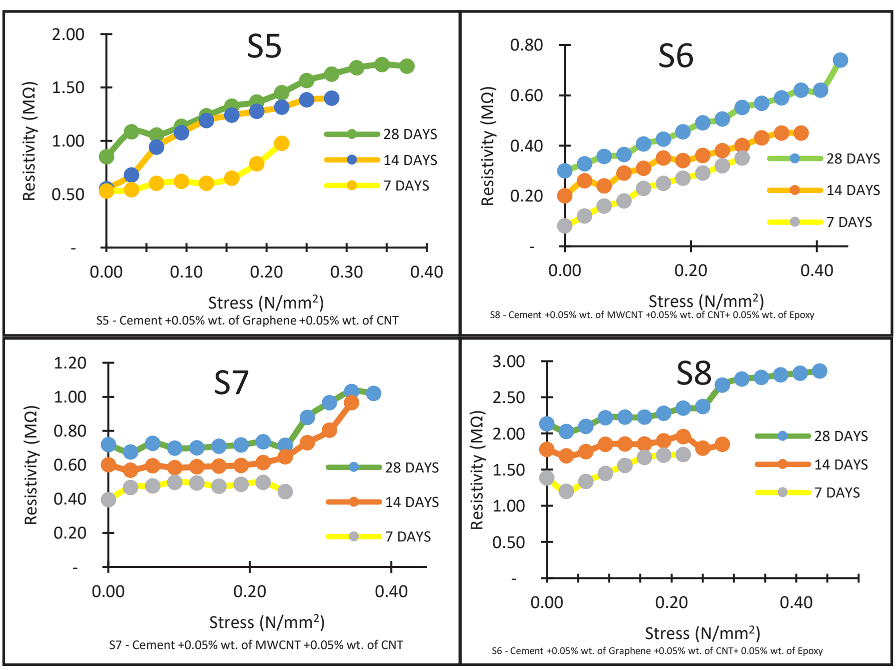

Fig. 7. Electromechanical test results on beams

4.2 Mechanical Properties

The Mechanical properties such as compressive strength, flexural strength are presented in Figure 8 and 9 respectively. The Specimens S6 and S8 have grater compressive and flexural strength compared to S5 and S7 due to addition of epoxy in cement matrix. The increasing in the compressive and flexural strength found to be about 15% in the cement epoxy composite compared to other composites. The inclusions of Graphene and MWCNT fill up the cement matrix pores causing the reduction in cracks and increase the load carrying capacity of composites. The interfacial bond between the nano particles and the epoxy cement matrix enables the effective load transfer these interfaces, which enhances the mechanical properties of the composite[34]. The epoxy and nano particles have greater strength and clastic modulus compared to plain cement. The epoxy and nano particles supports load acting on composite and reduced the lateral deformation in composite. Meanwhile, the pulling effect of the MWCNR and Graphene in the cement epoxy matrix also constrained the transverse deformation of the composites, resulting in the increases of mechanical properties[35].

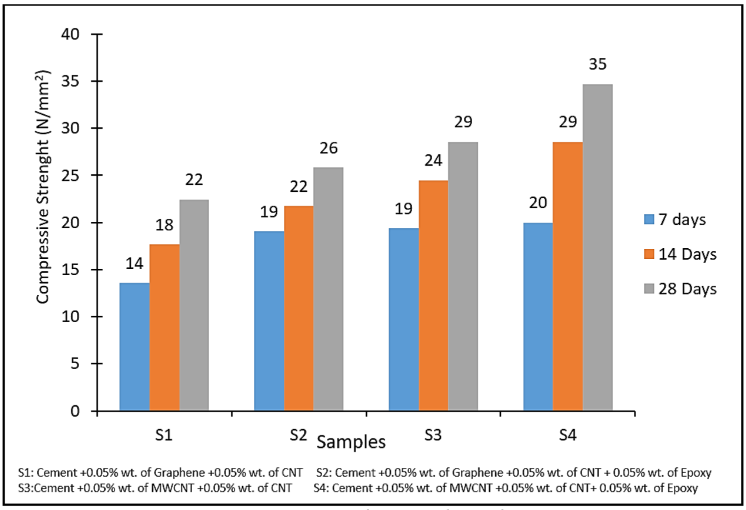

Fig. 8. Compressive Strength on cubes

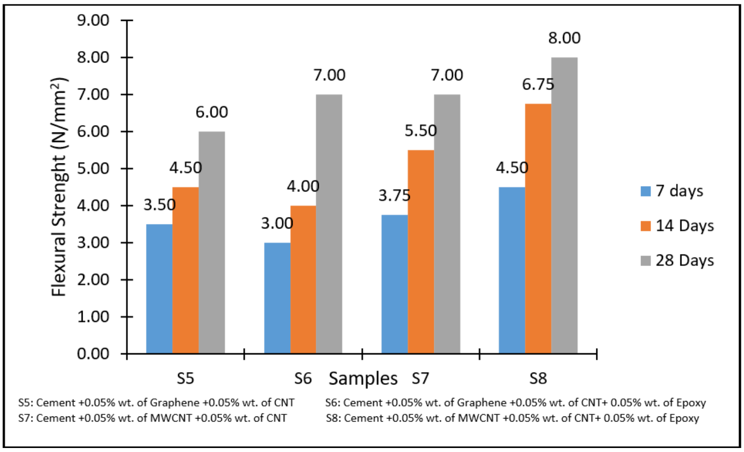

Fig. 9. Flexural Strength of beam

5 Conclusion

The major goal of the research is to develop the smart self sensing cement based polymer nano composite. The experimental results show the variation in electrical resistance due to change in applied stress which indicates the piezo resistivity properties of composites. It is also observed that epoxy-based nano-composites shows improvement in strength as compared to cement-based nano-composites. Nano materials such as MWCNT, Graphene acts as conductive materials in cement composites. These nanofillers improves electrical properties along with the mechanical properties such as compressive and flexural strength. The epoxy has crucial role in increasing strength. The most important component in improving the mechanical properties of the nanocomposites is the interfacial bond between the nano materials and the epoxy matrix, which helps in load transfer from the CFs through the interface. It is observed that when compression load is applied on specimen resistivity decreases and during application of flexural load there is increment in resistivity. This fluctuation of resistivity with respect to load helps in monitoring health of structure. In summary, Cement composites with Carbon fibres, nano materials and Epoxy improves the mechanical and electrical properties composites which enables them as smart multifunction composite to develop the self sensing sensors for structural health monitoring application.

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