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Study of Effect of Sol-Gel Synthesized TiO_2/Fe_2O_3 Nanocomposites on Compressive Strength of Concrete

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Abstract: In the present study, TiO_2/Fe_2O_3 nanocomposites are synthesized by using sol-gel method and applied in preparation of concrete, to study the compressive strength property. Portland cement is replaced by a mineral admixture namely fly ash (5 wt%) and TiO_2/Fe_2O_3 nanocomposites (0, 0.5, 1.0, 1.5 and 2.0 wt% of fly ash). Sol-gel method is very effective and low cost. Also, the size, shape and structure of final product are greatly influenced by reaction parameters. The characterization of TiO_2/Fe_2O_3 nanocomposites is done by X-ray powder diffraction (XRD), UV-Visible spectroscopy, Scanning electronic microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The synthesized TiO_2/Fe_2O_3 nanocomposites are of average crystallite size 6.59 ± 2.6 nm. The compressive strength of concrete cubes increased by 40.9% after adding TiO_2/Fe_2O_3 nanocomposites in concrete replacing up to 2 wt% of fly ash present.
Keywords: TiO_2, Fe_2O_3, nanoparticles, nanocomposites, compressive strength, concrete, fly ash, cement.

I. INTRODUCTION

Currently, nanotechnology is finding wide applications in every field of engineering, especially in construction field. Construction industry ranks eighth out of ten most significant areas of applications of nanotechnology. Although nano products are costly, research in nanotechnology is growing because of immediate return of profit from high value products. Concrete is the mostly used material and the popularity of concrete is due to the fact that common ingredients, the properties of concrete are tailored to meet the demand of any particular application. Concrete is good in compressive strength, durability and stiffness. The main disadvantage is that it is weak in tension and a brittle material. Due to increase in population, high raised buildings are constructed and for that concrete is very cheapest material for construction. So, regarding its strength it should be strong enough to bear the load. Many researches are done to increase the strength of concrete. Strength of concrete depends on the composition and quality of ingredients. Engineered concrete consists of six components i.e. fine aggregate, coarse aggregate, cement, water, chemical admixture and mineral admixture that results in high performance and ultrahigh performance concrete. The main active constituent of concrete is cement and water that are reactive in nature that binds the fine and coarse aggregate. Concrete consist of 1-2% of voids that leads to decrement of compressive strength. The voids of mortar in concrete can be filled by using nanoparticles viz. nano-SiO_2, nano-ZnO, nano-Al_2O_3, nano-ZrO_2, nano clay, nano-Fe_2O_3 and nano-TiO_2. The nanoparticles have proven to be very effective for increasing the compressive strength[1].The TiO_2 nanoparticles have several properties like self-cleaning, photo-catalytic activity and non-toxicity. The Fe_2O_3 nanoparticles have self-sensing, corrosion resistance and fire resistance properties. Several investigations have been undertaken on application of individual TiO_2 and Fe_2O_3 nanoparticles in cementitious building materials. Some studies on like photo-catalytic activity revealed that the TiO_2/Fe_2O_3 nanocomposites gave better results compared with individual TiO_2 and Fe_2O_3 nanoparticles[2].In the present study, it is expected that the performance of cement-based materials can be enhanced by replacement with nanocomposites, performance in terms of self-cleaning, self-sensing and strength properties. Hence, TiO_2/Fe_2O_3 nanocomposites were synthesized in the laboratory and same were applied in the preparation of concrete. Tests were conducted to check for improvement in strength properties.

II. EXPERIMENT

A. Materials

The chemicals used for synthesis of nanocomposites were Titanium tetra isopropoxide ($Ti\{OCH(CH_3)_2\}_4$), Iron (III) nitrate nonahydrate($Fe(NO_3)_3.9H_2O$), Ethanol(CH_3CH_2OH) and Ammonia solution 25%(NH_3) were purchased from Sigma Aldrich Chemicals (India) Ltd and are AR grade. The materials used for the preparation of concrete were Ordinary Portland Cement (ACC

53 grade), fine aggregate (sand), coarse aggregate (maximum size of 20 mm crushed stone) and fly ash from Rayalaseema Thermal Power Plant (RTPP), Muddanuru, YSR Kadapa (Dist.), Andhra Pradesh with following characteristics (Table 1).

Table 1 Test results of fly ash*

S. No.	Property	Test results
1	Specific gravity	2.7
2	Bulk density in loosest state	780-860 kg/m^3
	Bulk density in densest state	920-980 kg/m^3
3	Fineness modulus	4 %

*Laboratory reports of Rayalaseema Thermal Power Plant (RTPP), Muddanuru, YSR Kadapa (Dist.).

Double distilled water was used throughout the experiments for synthesis. All chemicals were used without any further purification. The water used for casting and curing of concrete specimens is free from acids, impurities and suspended solids. If the above contaminants are present in the water, strength and durability of concrete will be affected. The local drinking water, which is free from such contaminants, have been used in this experimental investigation.

B. Synthesis of TiO_2/Fe_2O_3 nanocomposites by using Sol-Gel technique

To synthesize TiO_2/Fe_2O_3 nanocomposites (Fig. 1), 60 mL of ethanol (CH_3CH_2OH) was taken in a beaker and kept it for stirring on a magnetic stirrer. 5.18 mL titanium (IV) isopropoxide ($Ti\{OCH(CH_3)_2\}_4$) and 1.52 g iron (III) nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$) were added to it as precursors for Ti^{4+} and Fe^{3+} respectively. The mixture was stirred for 2 hours. 36 mL of distilled water was added to the mixture. Ammonia solution (NH_3) was added drop wise using dropper in order to maintain the pH around 10. The stirring was continued for another 24 hours. The resulting mixture was kept in dark overnight for nucleation. The precipitated product was centrifuged at 1000 rpm for 1 hour to remove the template present in the solution of ethanol-water mixture. The solid material was collected into the petri dishes then dried in hot air oven at 100 ℃. This material was calcined in muffle furnace at 400℃ for 6 h to remove the occluded template. The obtained particles were converted to fine powder by using mortar and pestle (Fig. 2) [3].

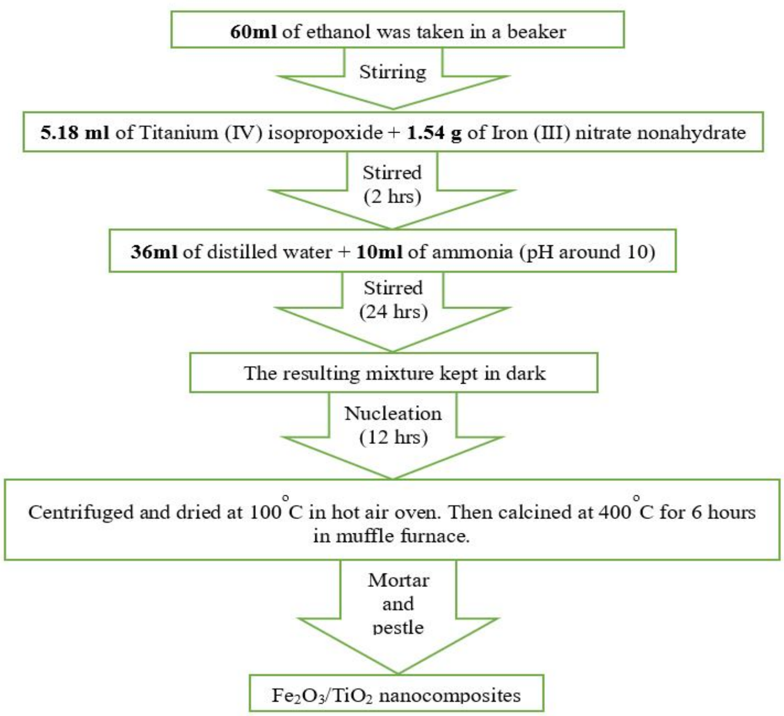

Fig. 1Flow chart for Synthesis of TiO_2/Fe_2O_3 nanocomposites

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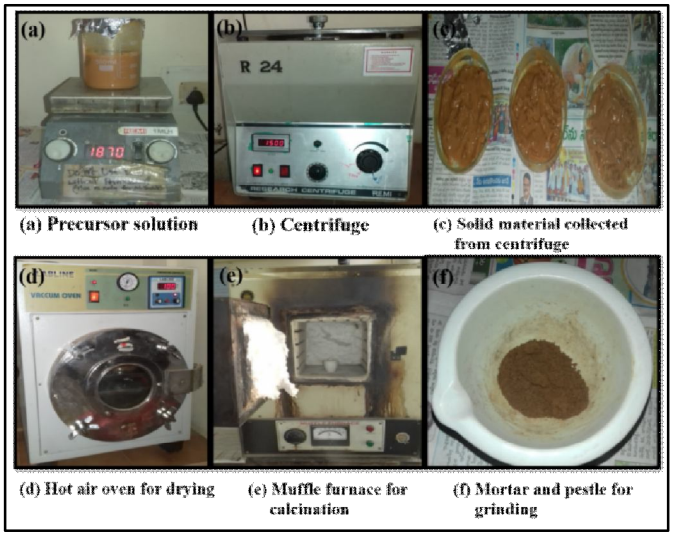

Fig.2 Synthesis of TiO_2/Fe_2O_3 nanocomposites by Sol-Gel method

C. Characterizations

X-ray diffraction (XRD) analysis of TiO_2/Fe_2O_3 nanocomposites was recorded on a WRD PANalytical "XPERT 3 PRO" X-Ray diffractometer using Cu-Kα (λ = 1.54060 nm) as the radiation source in the 2θ range of 5° - 70° with a step size 0.01 and a step time of 5s.Scanning electron microscopy (SEM) was performed at 15.0 kV with magnification 30.0k for 1μm and 12.0k for 4 μm scale bars respectively. A Systronics PC-based double beam UV-Vis spectrophotometer (type: 2201) was used to generate the spectrum for the scan range 190-1000 nm. Fourier Transform Infrared Spectroscopy (FTIR) was conducted to identify the functional groups and the spectrum was recorded in the range 4000-500 cm^{-1}.

D. Tests for Cement

The Vicat mould test was used for finding out normal consistency as per IS 5513: 1976 [4], initial setting time as per IS 4031 (Part 4): 1988 [5] and final setting time as per IS 4031 (Part 5): 1988 [6]. The normal consistency of a cement paste is defined as that consistency which will permit a Vicat plunger having 10 mm diameter and 50 mm length to penetrate to a depth of 33-35 mm from the top of the mould. Vicat apparatus conforming to IS 5513: 1976 [4]. The fineness of cement has an important bearing on the rate of hydration and hence on the rate of gain of strength and also on the rate of evolution of heat. Finer cement offers a greater surface area for hydration and hence faster the development of strength. The particle size fraction below 3 microns has been found to have the predominant effect on the strength at one day while 3-25-micron fraction has a major influence on the 28 days strength. Increase in fineness of cement is also found to increase the drying shrinkage of concrete. The fineness modulus of cement sample was determined by sieving method using 90 microns sieve as per IS 4031 (Part 1): 1988 [7]. The specific gravity of hydraulic cement was found as per IS 4031 (Part 11): 1988 [8] by using Le-Chatelier flask. This specific gravity result for cement was used in the mix design calculations of concrete.

E. Tests for aggregates

The specific gravity of the fine aggregate and coarse aggregate were found using pycnometer (500 ml) having limit of 2.6 - 2.8 as per IS 2386 (Part 3): 1963 [9]. This specific gravity results were used in the mix design calculations of concrete.
The fineness modulus of fine aggregates and coarse aggregate have been calculated as per IS 383: 1970 [10]. Here fine aggregate (sand) zone was designated from Table 4 of IS 383: 1970 whether it belongs to Zone I/ Zone II/ Zone III/ Zone IV (i.e., coarser to finer). The fineness modulus limits may be taken for Fine sand (2.2 - 2.6), Medium sand (2.6 - 2.9), Coarse sand (2.9 - 3.2). Sand having a fineness modulus more than 3.2 will be unsuitable for making satisfactory concrete.
Moreover, in coarse aggregate (crushed stone) zone was designated from Table 2 of IS 383: 1970whether it belongs to single-sized aggregates of nominal size/ graded aggregates of nominal size [10]. The upcoming results were also used in the mix design calculations of concrete.

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F. Mix design of M20 grade concrete

The M20 concrete mix was designed using IS 10262: 2009and IS 456: 2000 that gave a mix proportion of 1:1.65:2.99with water cement ratio of 0.50 [11, 12]. It is proposed that the replacement of cement with mineral admixture i.e., fly ash on 5% of weight of cement and combination of Fe_2O_3/TiO_2 nanocomposites with varying percentages (0%, 0.5%, 1%, 1.5% and 2.0%) on 5% weight of fly ash. For each mix three normal 150mm*150mm*150mm cube specimens were cast to know the compressive strength of cubes after 7 days of curing.

G. Mixing, casting and curing

The concrete materials are mixed properly for producing the homogenous mass of concrete. The method of mixing is decided by considering the terms such as requirement, quality, quantity etc. Hand mixing was preferred and done manually (Fig. 3). The required amounts of ingredients were weighed individually using weighing balance. All the ingredients were poured layer by layer and mixed by adding water till the mixture got uniform colour. During mixing of concrete the cube mould plates were removed, properly cleaned and assembled by means of fitting the bolts tightly. A thin layer of oil was applied on all the inner faces of the moulds. The concrete sample was filled into the cube moulds of size 150 mm*150 mm*150 mm in 3 layers, each layer was compacted by tamping rod. The strokes were penetrated with respect to tamping throughout its depth. The casted cubes were stored under shed at a place free from the vibration for 24 hours and covered them with gunny sacking. The specimens were marked during this period. After 24 hours the cubes were de-moulded (removed from moulds) and immersed for about 7 days in clean fresh water for curing. Mixing, casting and curing are very much essential for ensuring the strength enhancement and water permeability.

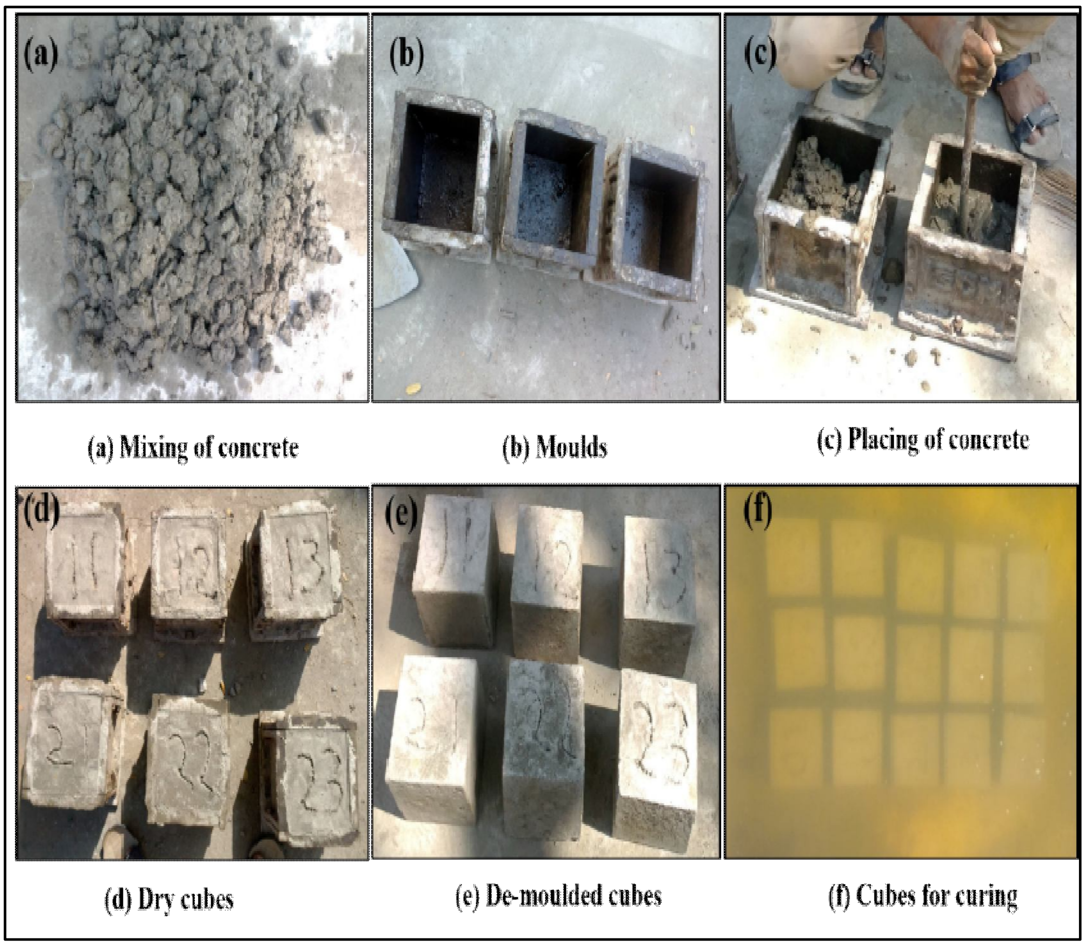

Fig. 3 Mixing, Casting and Curing

H. Determination of compressive strength

The specimens were removed from water after 7 days of curing and excess water was wiped out from the surface. The bearing surface of the testing machine was cleaned. Each specimen was placed in the machine in such a manner that the load applied to the opposite side of the cube cast. The load was applied gradually without shock and continuously at the rate of 140 kg/cm^2/min till the specimen failed. The maximum load (kN) was recorded which appeared in the display (Fig. 4).

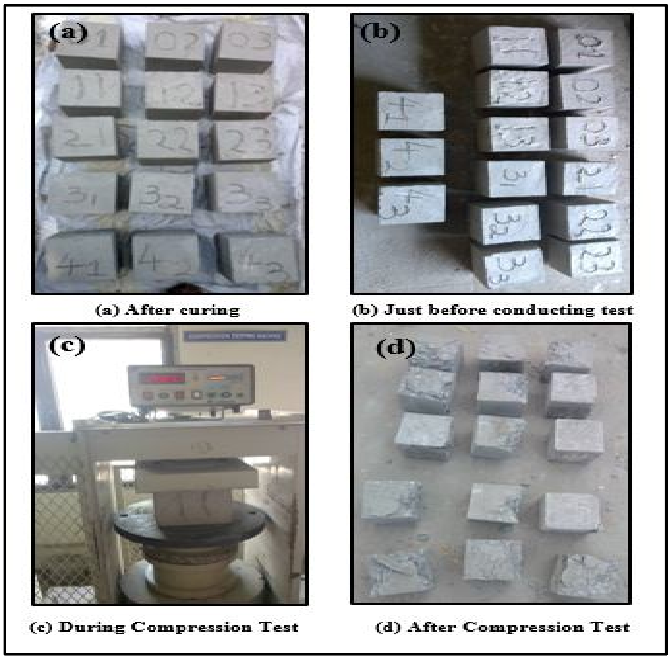

Fig. 4 Compression test for cubes

III. RESULTS & DISCUSSIONS

A. Synthesis results of TiO_2/Fe_2O_3 nanocomposites

The yield of synthesized TiO_2/Fe_2O_3 nanocomposites was 12.07 grams by taking the precursors 30.92 ml of Titanium (IV) isopropoxide (TTIP) and 9.12 g of Iron (III) Nitrate nonahydrate.

B. XRD Analysis

The crystalline nature of synthesized nanocomposites was identified using X-ray diffractogram (XRD) pattern. The diffractogram pattern was indexed properly for all crystalline peaks and compared with JCPDS data file (Fig. 5). The major peaks at 2θ values of 25.28° and 40.08° corresponds to the planes of (101) and (004) of tetragonal anatase TiO_2 (JCPDS Card No. 21-1272) [13, 14]. The peaks at 2θ values of 35.98°, 50.03°, and 63.22° which corresponds to the planes of (104), (024), and (214) corresponds to the α-phase of Fe_2O_3 (JCPDS card no. 03-0800) [15].The average crystallite size of TiO_2/Fe_2O_3 nanocomposites was estimated to be 6.59 ± 2.6 nm from Debye-Scherer formula. The composite shows crystalline phase peaks of both TiO_2 & Fe_2O_3 and hence confirms the composite formation.

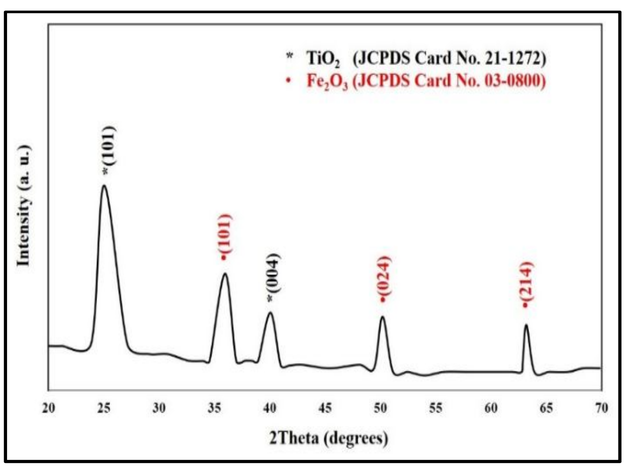

Fig.5XRD analysis of TiO_2/Fe_2O_3 nanocomposites

C. FTIR Spectroscopy

The broad peak observed at 3256.58 cm^{-1} was assigned to stretching vibrations of (O-H) of Fe_2O_3, and the peak at 578.30 cm^{-1} represents to Fe-O stretching mode [16]. The peak at 1620.76 cm^{-1} corresponds to deformative vibration of Ti-OH stretching modes and the peak at 823.78 cm^{-1} corresponds to the Ti-O bending mode of TiO_2 (Fig. 6) [17].

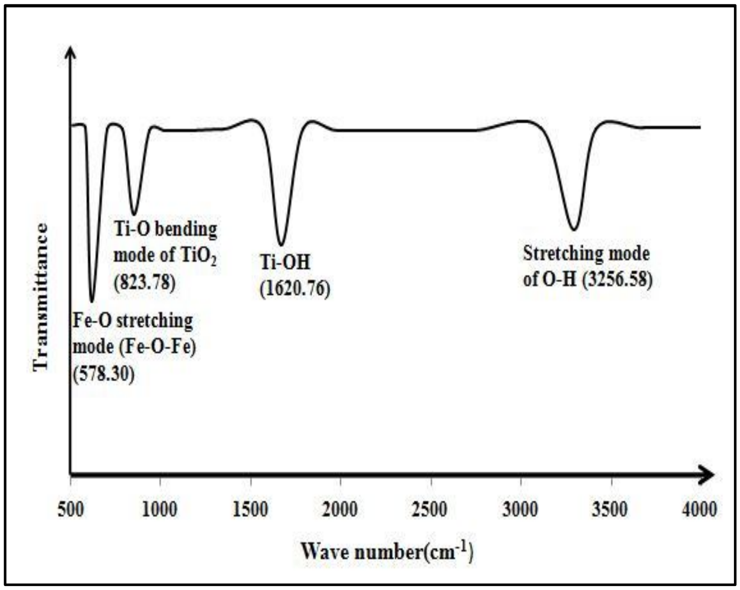

Fig. 6FTIR analysis of TiO_2/Fe_2O_3 nanocomposites

D. SEM Analysis

The surface morphologies of TiO_2, Fe_2O_3 and TiO_2/Fe_2O_3 nanoparticles assembly were examined by Field Emission Scanning Electron Microscopy (Fig. 7). The SEM image shows the spherical shape of the TiO_2 nanoparticles. However, it was found that α-Fe_2O_3 nanoparticles were agglomerated and did not possess any definite shape.Theaverage particle size is around 75 nm.

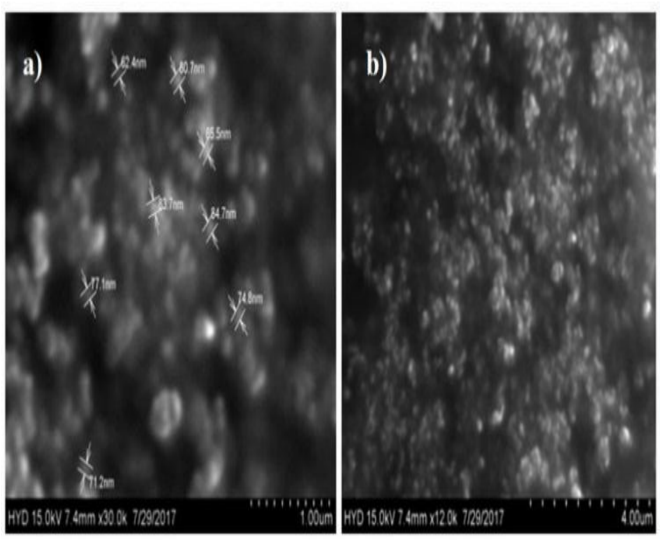

Fig.7SEM analysis of TiO_2/Fe_2O_3 nanocomposites

E. UV-Vis Spectroscopy

UV-Vis absorption spectroscopy is the measurement of the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface. The UV-Visible spectrum of TiO_2/Fe_2O_3 nanocomposites (Fig. 8) shows absorbance at 390 nm and 560 nm, which indicates the wavelength of TiO_2, Fe_2O_3 nanoparticles respectively [18].

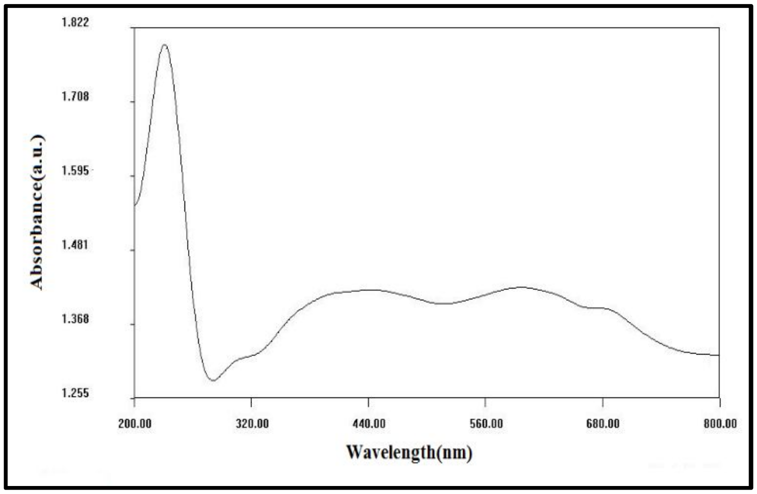

Fig.8UV-Vis Spectrum analysis of TiO_2/Fe_2O_3 nanocomposites

F. Basic test results of concrete materials

1) *Test results of cement:* Locally available Ordinary Portland cement of ACC-53 grade conforming to IS 12269: 2013[19] was procured, and following results obtained according to the appropriate tests conducted in the laboratory (Table 2).

Table2Test results of cement

S. No.	Property	Test results
1	Normal consistency	30%
2	Initial setting time	50 minutes
	Final setting time	460 minutes
3	Fineness modulus	5%
4	Specific gravity	3.15

2) *Test results of fine aggregate or sand:* ocally available natural river sand conforming to IS 383: 1970 was used as fine aggregate [10]. It was tested for specific gravity (Table 3) and fineness modulus (Table 4).

Table3Specific gravity of fine aggregate

S. No.	Description	Weight (g)
1	Empty pycnometer (W_1)	620
2	Pycnometer + 3/4th fine aggregate (W_2)	1449
3	W_2 + 1/4th water (W_3)	1990
4	Pycnometer + water (W_4)	1470

Specific gravity of fine aggregate $=\frac{(W_2-W_1)}{((W_4-W_1)-(W_3-W_2))}$= 2.68.As per IS 2386 (Part 3): 1963 the specific gravity of fine aggregate is having a limit of 2.6 - 2.8 [9]. Hence the selected fine aggregate (sand) was safe for the production of satisfactory concrete.

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Table 4 Fineness modulus of fine aggregate by sieve analysis

LS Sieve Designation	Weights			% finer	Zone-III limits as per IS 383: 1970 (Table 4)
	Retained wt. of Fine aggregate (grams)	% wt. retained	Cumulative % wt. retained		
10 mm	0	0	0	100	100
4.75 mm	24	0.96	0.96	99.04	90 – 100
2.36 mm	78	3.12	4.08	95.92	85 – 100
1.18 mm	232	9.28	13.36	86.64	75 – 100
600 µm	474	18.96	32.32	67.68	60 – 79
300 µm	1244	49.76	82.08	17.92	12 – 40
150 µm	440	17.6	99.68	0.32	0 – 10
Pan	8	0.32	100	0	-

Fineness modulus = (sum of cumulative % weight retained from sieve 4.75 mm -150 µm)/100= 232.48/100 = 2.32. The fine aggregate (sand) belongs to Zone-III from Table 4 ofIS 383: 1970 and the sand is 'Fine Sand' (Fineness modulus: 2.2 - 2.6) [10].

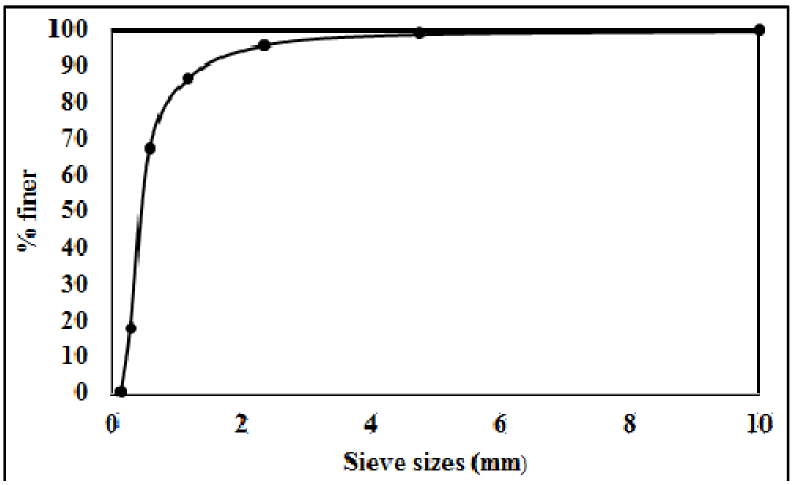

Fig. 9 Grading curve of fine aggregate

3) Test results of coarse aggregate: The machine crushed gravel conforming to IS 383: 1970 [10], obtained from the local quarry was used as coarse aggregate. It was tested for specific gravity (Table 5) and fineness modulus (Table 6).

Table 5 Specific gravity of coarse aggregate

S. No.	Description	Weight (g)
1	Empty pycnometer (W_1)	620
2	Pycnometer + 2/3rd coarse aggregate (W_2)	2160
3	W_2 + 1/3rd water (W_3)	2445
4	Pycnometer + water (W_4)	1470

Specific gravity of coarse aggregate = $\frac{(W_2-W_1)}{((W_4-W_1)-(W_3-W_2))}$ = 2.72. As per IS 2386 (Part 3): 1963, the specific gravity of coarse aggregate has limit of 2.6 - 2.8 [9]. Hence the selected coarse aggregate (crushed stone) was safe for the production of satisfactory concrete.

Table 6 Particle size distribution of coarse aggregate by sieve analysis

I.S Sieve	Weights			% finer	Grade limits as per IS: 383-1970 (Table 2)
	Weight retained (g)	% weight retained	Cumulative % weight retained		
63 mm	0.0	0.0	0.0	100.00	-
40 mm	0.0	0.0	0.0	100.00	-
37.5 mm	0.0	0.0	0.0	100.00	-
25 mm	0.0	0.0	0.0	100.00	100
20 mm	520	10.40	10.40	89.60	85 – 100
16 mm	635	12.70	23.10	76.90	-
12.5 mm	420	8.40	31.50	68.50	-
10 mm	2580	51.60	83.10	16.90	0 – 20
6.3 mm	815	16.30	99.40	0.60	0 – 5
Pan	30	0.60	100.00	0.0	-

The coarse aggregate (crushed stone) belongs to single sized aggregate of nominal size of 20 mm from Table 2 of IS 383: 1970[10].

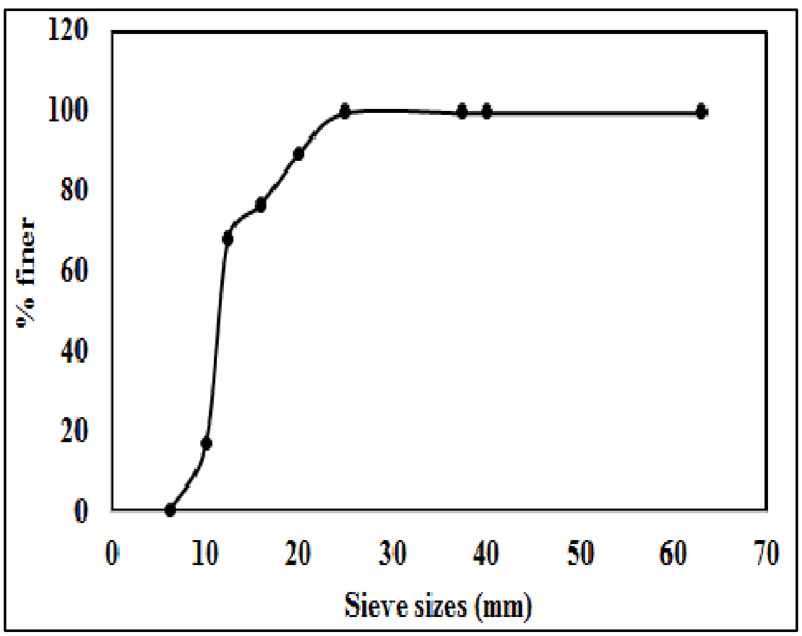

Fig. 10: Grading curve of coarse aggregate

G. Mix design of M20 grade concrete

For studying the effect of TiO_2/Fe_2O_3 nanocomposites on compressive strength, M20 grade concrete mix was considered. The M20 Mix design was carried out using ISI methods IS 10262: 2009 [11] and IS 456: 2000 [12]. The terms obtained from mix calculations are tabulated (Table 7).

Table 7 Results of mix calculation

Water cement ratio	0.5
Water content	197 litres
Cement content	394 kg/m^3
Volume of all in aggregate	0.6779 m^3
Mass of coarse aggregate	1180.08 kg
Mass of fine aggregate	654.037 kg

The actual quantities of mix proportions of concrete materials obtained from mix design are as follows:

Table 8 Actual quantities of concrete materials

S. No.	Material	Ratio	Density
1	Cement	1	394 kg/m^3
2	Fine aggregate	1.65	654.037 kg/m^3
3	Coarse aggregate	2.99	1180.08 kg/m^3
4	Water	0.5	197 kg/m^3

By these calculations the required amount of nanocomposites (0.5, 1.0, 1.5 and 2.0 wt%) for 5% fly ash replacement by nanocomposites for 3 cubes of each specimen is 10.97 grams. Since synthesized nanocomposites weighed 12.07 grams and 5% replacement of fly ash was considered for preparation of M20 grade concrete. The constituents of mix proportions for M20 grade concrete (Table 9) and the composition of five different mixes were designated (Table 10).

Table 9 Mix Proportions

Mix proportion	Water	Cement	Fine aggregate	Coarse aggregate	Fly ash *(5 wt% of cement)*
By Density (kg/m^3)	197	374.3	654.037	1180.08	19.7
By weight (kg) *(For 3 cubes of size 150*150*150 mm)*	2.194	4.168	7.284	13.143	0.2194

Table 10 Composition of different specimen showing replacement of fly ash by nanocomposites

S. No.	Name of the mix specimen	Replacement of fly ash with TiO_2/Fe_2O_3 nanocomposites (wt%)	Mix compositions (wt%)			Number of cubes casted
			Cement	Fly ash	Nano composites	
1	FA-0	0	95	5.0	Nil	3
2	FA-1	0.5	95	4.975	0.025	3
3	FA-2	1	95	4.950	0.050	3
4	FA-3	1.5	95	4.925	0.075	3
5	FA-4	2.0	95	4.900	0.100	3

H. Compressive strength of concrete

The compressive strength of cubes (Table 11) was determined by the formula, compressive strength = $\frac{Load\ (N)}{Area\ (mm^2)}$. Here, the area is surface area of the cube = 150 mm*150 mm = 22,500 mm^2.

Table 11 Compressive Strength of Cubes

Name of the Mix	Cube number	Weight of cube (kg)	Load on cube (kN)	Average load (kN)	Compressive strength (N/mm^2)	% increase or decrease of compressive strength
FA-0	01	8.440	665	651.66	28.96	0
	02	8.630	630			
	03	8.445	660			
FA-1	11	8.570	736	715.33	31.79	9.7
	12	8.470	690			
	13	8.480	720			
FA-2	21	8.605	780	740.66	32.92	13.6
	22	8.830	740			
	23	8.435	702			
FA-3	31	8.590	790	791.33	35.17	21.4
	32	8.315	801			
	33	8.415	783			
FA-4	41	8.830	868	918.66	40.82	40.9
	42	8.335	920			
	43	8.480	968			

Compressive strength of the cubes is found to be incremented, after fly ash replacement by nanocomposites at different proportions (Fig. 11).

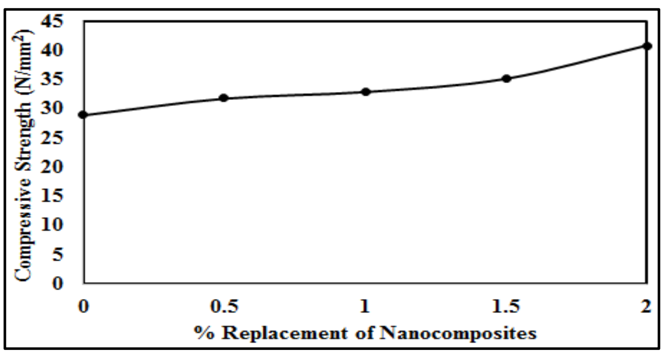

Fig. 11 Compressive Strength vs % replacement of nanocomposites

The compressive strength of concrete cubes increased after gradually increasing TiO_2/Fe_2O_3 nanocomposites up to 2.0 wt% of fly ash. (Fig. 12).

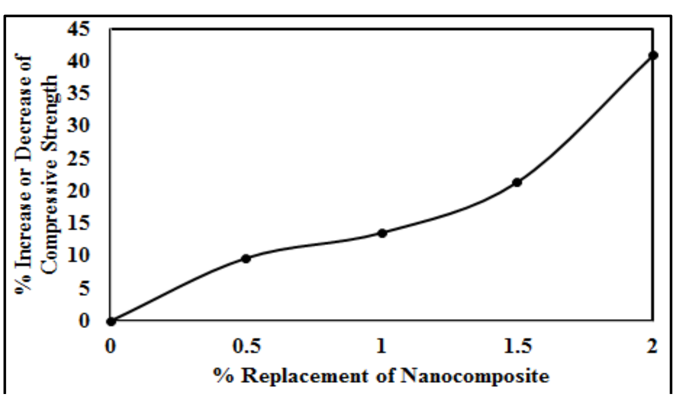

Fig. 12 % change of compressive strength vs % replacement of nanocomposites

High reactivity of TiO_2/Fe_2O_3 nanocomposites accelerated C-S-H gel formation resulting in increased crystalline $Ca(OH)_2$ at an early age of hydration [20]. The rapid formation of the gel results in a more compact microstructure. Further nanocomposites act as filler, reducing the porosity and making the micro structure denser [21]. Thus, increasing the compressive strength of the concrete.

IV. CONCLUSIONS

The TiO_2/Fe_2O_3 nanocomposites were successfully synthesized by using sol-gel method. UV-Visible spectrum displayed peaks at 390 nm for TiO_2 and at 560 nm for Fe_2O_3.From XRD analysis, the average crystallite size of anatase TiO_2 and Fe_2O_3 was estimated to be 6.59 ± 2.6 nm using *Debye-Scherer* formula. FTIR analysis revealed both Fe and Ti functional groups. SEM analysis indicated that aggregated Fe_2O_3 nanoparticles were surrounded by the TiO_2 nanoparticles and average particle size is around 75 nm. The concrete cubes made of TiO_2/Fe_2O_3 nanocomposites gave improved compressive strength than normal concrete cubes. Strength enhancement of 40.9% was achieved by gradually increasing TiO_2/Fe_2O_3 nanocomposites up to 2.0 wt% of fly ash.

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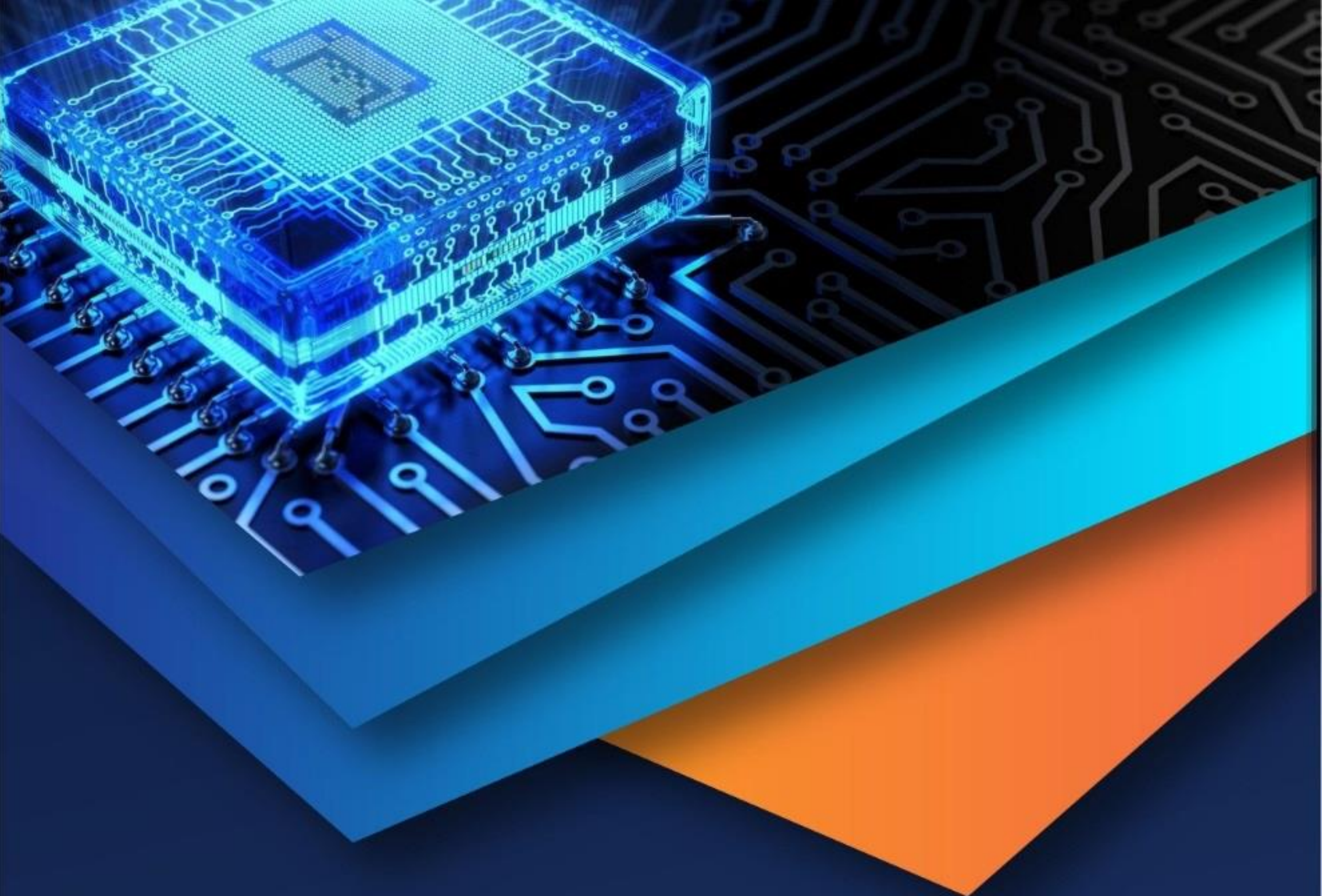

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