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Study Effect of Sol Gel Synthesized Tio2/Fe2O3 Nano Composites on Compressive Strength of Concrete

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Abstract: In this paper the TiO_2/Fe_2O_3 nano composites has to be synthesized. From UV-Visible spectra the peaks has to observe. From XRD analysis, the averagecrystallite size of anatase TiO_2 and Fe_2O_3 has to estimate. From SEM analysis, it is observed that aggregated Fe_2O_3 nano particles were surrounded by the TiO_2 nano particles and average particle size is around 75 nm. From compression test, the concrete cubes made of TiO_2/Fe_2O_3 nano composites gave better compressive strength than normal concrete cubes. Finally Strength enhancement has to achieve by gradually increasing TiO_2/Fe_2O_3 nano composites.

Keywords: XRD, Fe_2O_3 Nano Particles.

I. INTRODUCTION

Nanotechnology offers with the small systems or small sized substances. the everyday dimension spans from sub-nanometer to numerous hundred nano-meters. A nanometer (nm) is a one billionth of a meter (i.e., 10-9m). substances in this length variety show off a few tremendous specific houses and functions, a transition from atoms or molecules to bulk shape takes location in this length range [1]. Nanotechnology allows in generating substances with prospective properties, for every area of technology consisting of physics, chemistry, bioscience, engineering and so on. The application of nanotechnology in civil engineering and construction is immensely useful to the field. Several techniques of nanotechnology were used to increase the life of concrete, change its physical, thermal, mechanical and durability properties [2]. Change in these properties are due to the incorporation of nanoparticles in the cementitious materials. Nanoparticles have excessive floor vicinity to volume ratio, which provides a top notch driving force for diffusion. Sintering can take region over shorter time scales than for large particles. In principle, this doesn't affect the density of the final product, even though waft problems and the tendency of nanoparticles to agglomerate complicates matters. moreover, nanoparticles had been found to impart a few greater houses to various daily products. as an instance, the presence of titanium dioxide nanoparticles imparts the self-cleaning belongings and presents extra advantage of supporting to clean the surroundings [3].

A. Nanocomposites

Nanocomposite materials have gained much attention and hobby of scientists in current years because of their progressed houses than the unmarried steel nanoparticles. Nanocomposite is aggregate or matrix, wherein extraordinary materials combine to develop new residences of the substances making sure that one of the substances have length in variety of one-100 nm. There are two components on nanocomposites i.e. non-stop phase and discontinuous reinforcing section. Nanocomposites may be organized from any mixture of materials that may be classified into 3 basic building blocks i.e. metals, ceramics and polymers [4].

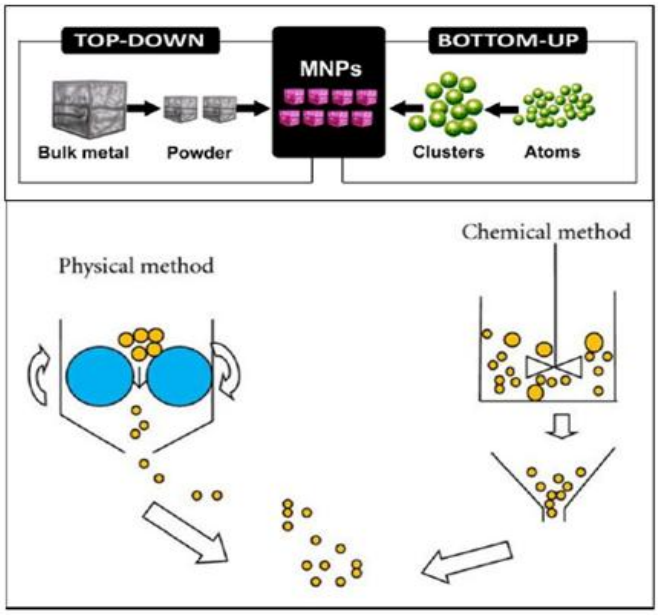

Fig 1: Schematic Representation of Top-Down and Bottom-Up Approaches

The synthesis of uniform sized nanocomposites is very vital because their optical, magnetic, electrical and biological houses depend upon their length and dimensions. The synthetic methods are regularly classified in to 3 classes i.e. answer primarily based synthesis, vapor phase synthesis and gasoline section synthesis. any other approach is to divide

these synthetic strategies into extensive categories i.e., top-down technique which includes physical techniques and backside up approach which encompasses moist methods (Fig. 1)[5]. the choice of synthetic method depends on the desired traits required in the nanoparticles composites e.g. size, morphology, crystal shape and so on., [6]. The gain of physical strategies is the production of massive quantities of nanocomposites however synthesis of equal sized nanocomposites debris isn't always without difficulty doable. In evaluation, moist chemical strategies provide uniformity in size of nanocomposites such that managed particle size can be completed without problems. similarly by using varying conditions of reaction, specific shapes (nano- rods, nanowires, nanotubes and many others.) of nanocomposites also can be synthesized [7].

B. Different Methods to SynthesizeNanocomposites

There are specific wet strategies for synthesis of metal nanocomposites but co-precipitation,sol gel and hydrothermal techniques are cost powerful compared to different strategies so these are mentioned in element.

1. Sol-Gel Method

Sol-gel method gained attention as a promising method for the synthesis of nanomaterials because of their slight response conditions and building up the materials from molecular precursors leading to variant in materials and homes. The ensuing made from sol-gel method is either movies [8] or colloidal powder [9].The sol-gel approach has functionality of manufacturing micro and nanostructures. The dimensions, form and structure of final product are significantly stimulated by the response parameters [10, 11]. The system entails the simple wet chemical reaction primarily based on hydrolysis and condensation main to formation of sol and the system of getting older results in formation of an incorporated network as gel (Fig. 2).

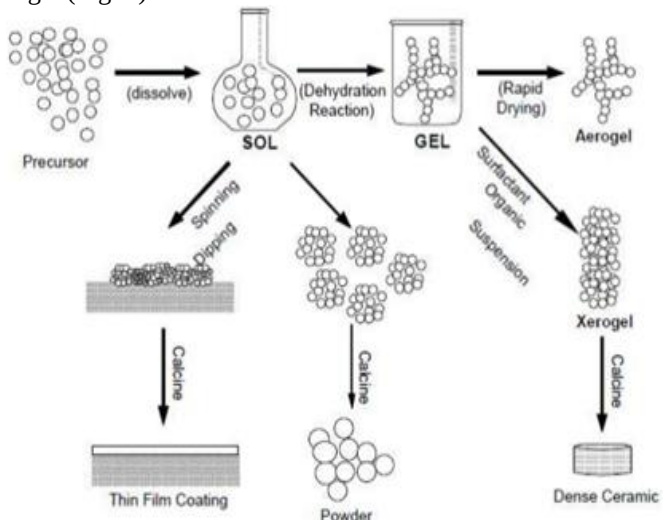

Fig2. Schematic Representation of Sol-Gel Method

The sol–gel technique is also very appealing for the synthesis of nanostructures containing multiple factor, because the gradual response kinetics permits exact structural engineering of the final product. Some other advantage is that the reactions are conducted at low temperatures or at room temperature. The sol-gel manner includes inorganic precursors that undergo diverse chemical reactions, resulting in the formation of a three-dimensional molecular network. One of the most commonplace routes is via hydrolysis and condensation of steel alkoxides to form large metallic oxide molecules that polymerize to shape the coating. The sol–gel technique permits coating of substrates with complicated shapes at the nanometer to micrometer scale. The substrates consist of colloidal particles, organic/inorganic crystals, or even fibers and nanotubes [12, 13].

2. Co-precipitation Method

The co-precipitation approach, used for the synthesis of metallic oxides nanoparticles, blended metal or metallic ceramics nano-composites, produces precipitates that are separated from answer. Inorganic salts are used as precursors, dissolved in water and different solvents to obtain homogenous solution of ions, and then those salts begin precipitating as hydroxides or oxalates whilst the vital awareness of species is attained observed through nucleation and boom levels. The size and shape of particles is significantly stimulated by using answer pH, temperature and awareness of salt. After precipitation, filtration and washing is finished followed by means of calcinations to transform hydroxide into oxides with a specific crystalline structure [14]. Distinct varieties of metallic nanocomposites (steel-steel oxide, oxide-oxide, and oxide-matrix) materials are synthesized using this method. The use of surfactants is likewise a commonplace practice to avoid agglomeration, which also tampers the particle size of the composites acquired by means of this technique [15]. The method offers the benefit of being low fee, simple, water based totally reaction, flexibility, slight response conditions and length control.

3. Hydrothermal Method

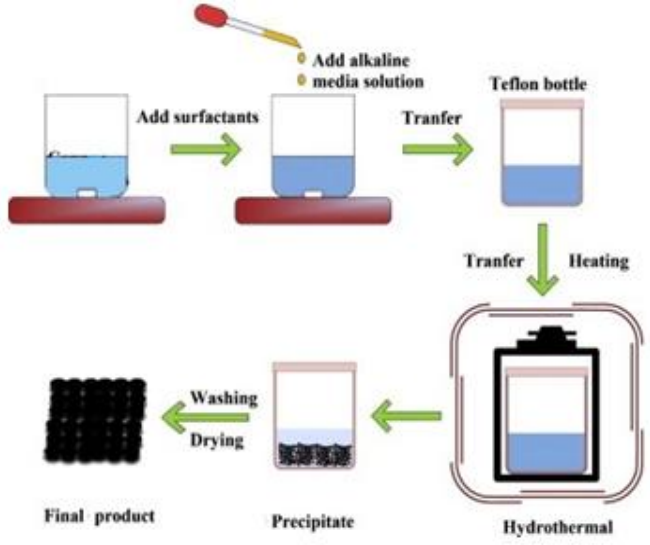

Fig3.Schematic Representation of Hydrothermal Method.

The hydrothermal method involves heterogeneous chemical reaction in a solvent (aqueous or non-aqueous) occurring

above room temperature and at pressure more than 1atm in a closed system (Fig. 3). To modify the size and properties, the surfactants [16], capping agents [17], mineralizers [18] are used. The new trend is to use this technique in combination with microwave[19], sol-gel [20]that can, not only vary the physiochemical and structural properties of the materials but also result in formation of single phased materials with enhanced stability.

C. Difference between Nanoparticles and Nanocomposites

Nanoparticles are small unit, possessing nano-metric dimensions in all the three dimensions. The diameters of nanoparticles can vary from 1 to 100 nm, which behaves as a whole unit in terms of its properties. Bulk materials have constant physical properties regardless of its size. However, at nanoscale based on the size of particles, properties will change. Size-dependent properties observed such as super-paramagnetism in magnetic materials [21], quantum confinement in semiconductor particles [22], and surface Plasmon resonance in some metal particles [23]. The nanocomposites is a multiphase solid material where one of the phases has one, two, or three dimensions of less than 100 nm. The solid combination of a bulk material and nano-dimensional phases differ in properties due to dissimilarities in structure. The mechanical, thermal, optical [24], electrochemical [25], catalytic properties [26] of the nanocomposites will differ from that of the nanoparticles. In general, nanocomposites synthesized by doping or deposition to any material help to change their electronic structure and transition probabilities and subsequently enhance the properties of the material or help to achieve newborn properties. Doping or deposition to nanoparticles makes the original band gap of the material under control and helps to tune their stabilization energy and substrate charge transfer, etc., [27].

II. PRESENT WORK

Present work focuses on increasing compressive strength of concrete, made of Ordinary Portland Cement (OPC) and partially replaced with fly ash and different amounts of TiO_2/Fe_2O_3nanocomposites (0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%). In the civil engineering field, most of the researchers investigated the effect of nanoparticles such as nano-SiO_2, nano-TiO_2, nano-Al_2O_3, nano-CuO, nano-TiO_2, nano-Fe_2O_3 and nano-ZnOon the physical and mechanical properties of cement based materials [28]. No published work is available on use of nanocomposites for application of concrete. This work aims to fill this gap.

A. Nano-TiO2

Nano-TiO2 has tested very powerful for the self-cleaning of concrete and presents the extra benefit of helping to clean the environment. Nano-TiO2 containing concrete acts by way of triggering a photograph-catalytic degradation of pollution, which include NOx, carbon monoxide, volatile organic Compounds (VOCs), chlorophenols, and aldehydes from vehicle and commercial emissions [29]. “Self-cleaning” and “de-polluting” concrete merchandise are already being produced by several companies to be used inside the facades of homes and in paving substances for roads and have been utilized in Europe and Japan. Similarly to imparting picture-catalytic houses, nano-TiO2 can boost up the early-age hydration and the trade in microstructure of cement-based totally substances, which influences the bodily and mechanical residences like compressive power of the mortars [30].

B. Nano-Fe2O3

Nano-Fe2O3 has been located to provide concrete with self-sensing capabilities in addition to enhance its compressive and flexural strengths. The extent electric powered resistance of cement mortar with nano-Fe2O3 was located to exchange with the applied load, demonstrating that mortar with nano-Fe2O3 ought to feel its personal compressive strain. Such sensing abilities are worthwhile for real-time, structural health tracking and for the development of smart structures, as they do no longer contain the use of embedded or attached sensors [31].

C. TiO2/Fe2O3nanocomposites

TiO2/Fe2O3nanocomposites form hetero structure assembly because of their different band gap energies. This has several advantages compared to individual nanoparticles [32, 33]. These nanocomposites are useful for photo-catalytic activity, electrochemical activity, photo voltaic studies etc.[34,35 and 36].Especially, the photo-catalytic activity for the degradation of 4-chlorophenol the Fe2O3/TiO2nanocomposites acts as an active photo-catalyst and gives better results compared with individualTiO2 and Fe2O3nanoparticles [37]. Based on the above results, 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, TiO2/ Fe2O3 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.

D. Concrete

Concrete is an artificial material in which the aggregates (i.e., both fine and coarse) are bonded together by the cement when mixed with water. The concrete become so popular and indispensable because of its inherent properties brought a revolution in the field of construction [38]. Its great versatility and relative economy in filling wide range of needs has made it very competitive building material. It has unlimited opportunities for innovative applications, design and construction techniques[39, 1].Based on these criteria several investigations have been moving on this construction material (concrete) by means of adding or replacement (Fully/ Partially) of admixtures, super plasticizers, nanomaterials, artificial aggregates etc., with concrete materials (i.e., Cement, Fine aggregate, Coarse aggregate and water) for making of different types of concretes like Self-Compacting Concrete (SCC), High Strength Concrete (HSC), High Performance Concrete (HPC), Ultra High Performance Concrete (UHPC), High Strength/Performance Self-Compacting Concrete (HSSCC/HPSCC) etc.,[40, 41 and 42].

In traditional construction sites, each of these concrete materials is procured separately and mixed in specified proportions at site to make concrete.

1. Cement

Cement is a tremendous concrete material, which can be used for the purpose of binding other concrete materials like aggregates, steel etc., and this cement is the combination of two raw materials called calcareous and argillaceous materials. As per Indian Standards, there are different types of cements produced for various purposes as follows:

TABLE I: TYPES OF CEMENT AS PER IS CODES

S.No.	Type of cement	Indian Standards (IS)
1	Ordinary Portland Cement a) 33 Grade b) 43 Grade c) 53 Grade	 IS269 IS8112 IS12269
2	PortlandPozzolana Cement	IS 1489
3	Portland Slag Cement	IS 455
4	Rapid Hardening Cement	IS 8041
5	Sulphate Resisting Cement	IS 12330
6	Low Heat Cement	IS 12600
7	Colored Cement/ White Cement	IS 8042
8	High Alumina Cement	IS 6452

2. Aggregates

Aggregate is a type of material used in construction, including sand, gravel, crushed stones etc., the purpose of aggregates is to increase the strength and weight. These are used in the construction of roads, railways, buildings etc. These aggregates are confirmed by IS 383: 1970. Types of aggregates areas follows

1. Classification according to the formation of aggregates
   - Natural aggregates
   - Artificial aggregates

2. Classification according to size of aggregates
   - Fine aggregates
   - Coarse aggregates

2.1 Classification according to formation

Natural aggregates: Natural aggregates consist of rock fragments which can be used in their natural kingdom, or are used after mechanical processing along with crushing, washing, and sizing.

Artificial aggregates: For making unique form of concrete, we should make special sort of synthetic aggregates. In those aggregates, most are lightweight aggregates and heavy weight aggregates.

2.2 Classification according to size

Fine aggregates: "Fine aggregate" defined as material that will passes through 4.75 mm sieve and will retained in the lesser (< 4.75 mm) sieves.

Types of Fine aggregates:

Sand: The first type of fine aggregate is Sand. Sand is little particles of silica. Larger particles categorized as gravel; smaller particles categorized as silt or clay. Below are different Types of sand:

Pit Sand: Sand from a pit, as wonderful from river or sea sand. When washed and screened, it is ideal sand for preferred purposes.

River Sand: River Sand obtained from the banks and beds of rivers is likewise coarse and high-quality. Coarse sand is exceptional to be used in creation works.

Sea Sand: This sand acquired from the shore of sea. The form of this sand is rounded. Because of water of sea, it mixes with different chemical substances.

Stone Dust: This is the 2ndtype of nice mixture. Stone dirt is a finely overwhelmed fabric used to fill within the spaces between gravel.

Cinder: Cinder is the slag from a steel furnace. before use this slag we beaten this in order that its length stays in exceptional mixture.

Surkhi: Surkhi is finely powdered burnt clay and commonly made from barely below burnt bricks.

Coarse Aggregate

Coarse aggregate was a basic material of the concrete. Crushed stone or gravel used in concrete are called coarse aggregate and it will not pass through a sieve with ¼-inch(i.e., 4.75 mm) diameter.

Types of coarse aggregates

- Stone Ballast
- Gravel
- Brick Ballast
- Clinker

Stone Ballast: Stone ballast produced by using mining a appropriate rock deposit and breaking down the eliminated rock to the preferred size the usage of crushers.

Gravel: Gravel is a mix of rock portions or small rocks. those are also known as rounded rocks.

Brick Ballast: For unimportant works, we are able to use brick ballast in concrete as coarse aggregates. For making this brick ballast,the well-burned bricks selected.

Clinker: Clinker usedin manufacturing of Portland cement. it is able to also be used as coarse mixture combined with other active elements or chemical admixtures to provide concrete.

E. Admixture

Admixture is natural or manufactured chemical compounds, which introduced to the concrete earlier than or at some point

of combination. The most usually used admixtures are fly ash, silica fume, air-entraining marketers, and water-reducing retarders. Admixtures used to provide unique residences to fresh or hardened concrete. it may beautify the durability, workability or energy traits of a concrete combination. these admixtures used to conquer difficult production conditions, which includes cool or warm climate placements, pumping requirements, early electricity necessities, and very low water content ratio specifications. Admixtures normally categorized into two types:

- Chemical Admixtures
- Mineral Admixtures

1. Chemical Admixtures

Chemical admixtures usually added to concrete in very small amounts mainly for the entertainment of air, reduction of water or cement content, plasticization of fresh concrete mixtures, or control of setting time.

2. Mineral Admixtures

Mineral admixture are commonly added to concrete in massive quantities to enhance the workability of the clean concrete to enhance resistance of concrete to thermal cracking, alkali-aggregates enlargement and sulphate attack and to allow a reduction in cement content. Fly ash, silica fume, ground granulated blast furnace slag, metakaolin and rice husk ash utilized in concrete [44].

F. Fly Ash

Fly ash is finely divided residue as a result of the combustion of powdered coal, transported through the flue gases, and amassed with the aid of the electrostatic precipitator. In U. ok, it's miles referred as pulverized fuel ash (PFA). Fly ash is the most broadly used mineral admixture all over the international. In recent times, the importance and use of fly ash in concrete, particularly for excessive power and high-performance concrete. Full-size studies performed everywhere in the world on the advantages that could executed with the aid of usage of fly ash as supplementary cementitious material. High volume fly ash concrete is a subject of contemporary interest all around the world. The usage of fly ash as concrete admixture now not most effective extends technical blessings to the properties of concrete however also contributes to the environmental pollutants manipulate. In India by myself, we produce approximately seventy five million tons of fly ash in keeping with 12 months but utilization of fly ash is handiest about 5% of the manufacturing. The first-rate of fly ash generated from one-of-a-kind plants range from one another to a large extent and as a result they're now not geared up to be used in concrete further processing is necessarily carried out [44].

G. Effect of Fly Ash on Concrete

Use of right great of fly ash, consequences in discount of water demand for favored slump. With the reduction of unit water content material, bleeding and drying shrinkage will reduced. Due to the fact fly ash isn't quite reactive, the warmth of hydration can be decreased thru the substitute of cement with fly ash, ordinary dosage of fly ash for excessive energy concrete is about 15% with the aid of weight of content material.

H. Objectives

- To synthesize TiO_2-Fe_2O_3nanocomposites by using Sol-Gel method.
- To characterize TiO_2-Fe_2O_3nanocomposites using UV-Vis Spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared Spectroscopy(FTIR).
- To check the compressive strength of concrete, which is made of ordinary portland cement and partially replaced with fly ash (5 wt%) and different amounts of TiO_2/Fe_2O_3nanocomposites (0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt% of fly ash).

III. MATERIALS & METHODS

A. Materials

1. Chemicals

The following chemicals are used in the synthesis of TiO_2/Fe_2O_3 nanocomposites using Sol-Gel method.

- Titanium (IV) isopropoxide (TTIP) – $Ti\{OCH(CH_3)_2\}_4$
- Iron (III) Nitrate nonahydrate – $Fe(NO_3)_3.9H_2O$
- Ethanol – CH_3CH_2OH
- Ammonia solution 25% - NH_3
- All above mentioned chemicals were purchased from Sigma Aldrich Chemicals (India) Ltd. Double distilled water was used throughout the experiments.

2. Materials for Concrete

The following materials are used in the preparation of concrete.

- Ordinary Portland Cement (ACC 53 grade)
- Fine aggregate (sand)
- Coarse aggregate (maximum size of 20mm crushed stone)
- Fly ash from Rayalaseema Thermal Power Plant (RTPP), Muddanuru, YSR Kadapa (Dist.), Andhra Pradesh with the following characteristics.

TABLE IV: 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 %

The water used for casting and curing of concrete specimens are 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 are free from such contaminants, have been used in this experimental investigation.

3. Glassware

- Beaker (500 ml)
- Measuring cylinders (100 ml &10 ml)
- Funnel
- Petri dishes

4. Equipment

- Electronic weighing balance
- Sonicator
- Centrifuge
- Hot air oven
- Muffle furnace
- X-Ray Diffractometer
- Ultraviolet-visible spectrophotometer
- Fourier transform infrared spectrophotometer
- Scanning Electron Microscope
- Vicat mould setup
- Le-Chatelier flask
- Pycnometer
- Compression Testing Machine
- Mortar & pestle
- Magnetic stirrer
- Sieves

5. Miscellaneous Materials

- Droppers (1 ml)
- Aluminum foil
- Spatula
- Tissue rolls
- Butter paper
- Gloves
- Goggles

B. Methods

1. Glassware Cleaning Process

The following process was used to clean all the glassware. First, the glasswarewas washed with detergent to remove dust contaminants on glass surfaces. During washing, the glasswarewas scrubbed with a brush to remove contaminants. After wash, the glasswarewas rinsed with distilled water. Then sonicated for 30 min to complete removal of adhesive dust particles.

2. Synthesis Of Tio_2/Fe_2o_3 Nanocomposites By Using Sol-Gel Technique

To synthesize TiO_2-Fe_2O_3 nanocomposites (Fig. 4), 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.

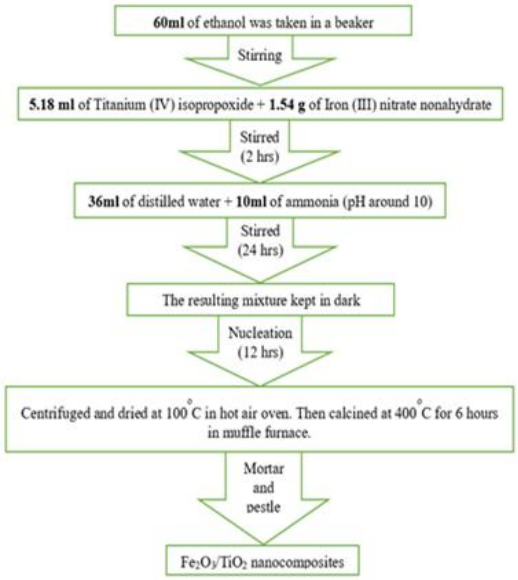

Fig4. Flow Chart For Synthesis Of Tio_2/Fe_2o_3 Nanocomposites.

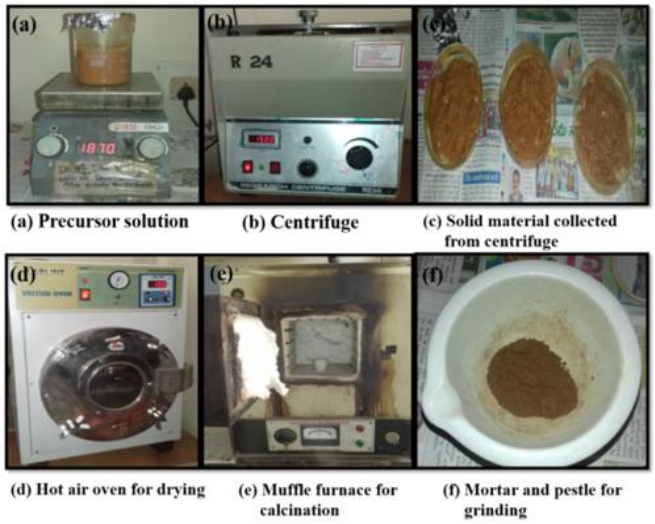

Fig 5: Synthesis Of Tio_2/Fe_2o_3 Nanocomposites By Sol-Gel Method

Ammonia solution (NH3) 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 100oC. This material was calcined in muffle furnace at 400ºC for 6 h to remove the occluded template. The obtained particles were converted to fine powder by using mortar and pestle (Fig. 5)[69].

3. Characterizations

The following characterizations are used for analysis of synthesized sample.

X-Ray Diffraction (XRD): In order to identify the crystalline nature of nanoparticles, X-ray diffractogram (XRD) is used. Using the XRD analysis, the lattice arrangement of the crystallites atoms and the size of crystals were obtained by using Braggs law and Debye- Scherrer equation respectively.XRD diffraction patternsof nanoparticles were recorded on a WRD PANalytical "XPERT 3 PRO"X-Ray diffractometer using CuKα (λ = 1.54060 nm) as the radiation source in the 2θ range of 5º - 70º with a step size 0.01 and a step time of 5s.

Scanning Electron Microscopy (Sem): The surface morphologies of nanoparticles assembly were examined by Field Emission Scanning Electron Microscopy. Using SEM analysis, we can view and understand the topographic images of synthesized sample. The picture showed a topographic image of nanocomposites that was captured from SEM having accelerating voltage of 15.0kV withmagnification 30.0k for 1 µm and 12.0k for 4µm scale bars respectively.

Fourier Transform Infrared Spectroscopy (FTIR): FTIR analysis was performed to identify functional groups on the surface of nanomaterials.It is one of the infrared spectroscopy techniques, where in "molecular vibrational resonance" principle is used to find the bonding structure of the functional groups. The spectrum was recorded in the range 4000-500 cm^{-1} on an FTIR spectrometer.

Ultra Violet-Visible (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 spectra of the samples were recorded using Systronics Double Beam UV Spectrophotometer-2201 model having range 190-1000 nm with ethanol as the reference.

4. Tests for cement

Vicat mould test: This testwas used for locating out normal consistencyas consistent with IS 5513: 1976 [82], preliminary settingtimeas in step with IS 4031 (part 4): 1988 [80] and very last setting time IS 4031 (component 5): 1988 [81]. The ordinary consistency of a cement paste is defined as that consistency to be able to allow a Vicat plunger having 10 mm diameter and 50 mm period to penetrate to a intensity of 33-35 mm from the top of the mould. Vicat equipment conforming to IS 5513: 1976 [82].

Fineness modulus test for cement: The fineness of cement has an crucial bearing on the charge of hydration and hence at the rate of benefit of power and also at the price of evolution of heat. Finer cement gives a extra surface location for hydration and consequently faster the improvement of electricity.The particle size fraction underneath three microns has been discovered to have the principal impact at the power at in the future while three-25-micron fraction has a main have an impact on at the 28 days strength. increase in fineness of cement is also determined to boom the drying shrinkage of concrete.The fineness modulus of cement pattern become decided via sieving approach the use of 90 microns sieveas per IS 4031 (Part 1): 1988 [79].

Specific gravity test for cement: The specific gravity of hydraulic cement has been found as per IS 4031 (part 11): 1988 [83] by using Le-Chatelier flask. This specific gravity result for cement was used in the mix design calculations of concrete.

5. Tests For Aggregates

Specific Gravity tests for aggregates: The specific gravity of the fine aggregate and coarse aggregate were found using pycnometer (500 ml) having limit of 2.6 - 2.8as per IS 2386 (part 3): 1963 [76]. This specific gravity results were used in the mix design calculations of concrete.

Sieve analysis for aggregates: The fineness modulus of fine aggregates and coarse aggregate has been calculated as per IS 383: 1970 [75].Here fine aggregate (sand) zone was designated from Table4 of IS 383: 1970 (Appendix A5) 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 Table2 of IS 383: 1970 (Appendix A5) whether it belongs to single-sized aggregates of nominal size/ graded aggregates of nominal size. The upcoming results were also used in the mix design calculations of concrete.

6. Mix Design of M20 Grade Concrete

The M20 concrete mix was designed using IS 10262: 2009 and IS 456: 2000that gave a mix proportion of 1:1.65:2.99 with water cement ratio of 0.50.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.

7. 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. In this project hand mixing was preferred and can be done manually. 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 (Fig. 6). 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.

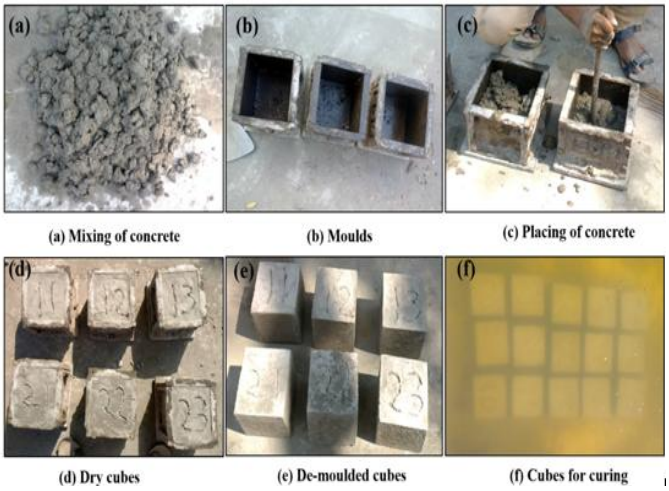

Fig 6: Mixing, Casting and Curing.

8. Determination of Compressive Strength

The specimens were removed from water after 7 days of curing and wipeout excess water from the surface. The bearing surface of the testing machine(Fig.7) 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. The compressive strength of concrete cubes determined by the following formula:

$$\text{Compressive strength} = \frac{\text{LOAD (N)}}{\text{AREA (mm}^2\text{)}} \quad (1)$$

Here, the area is the surface area of the cube = 150 mm * 150 mm. = 22500 mm^2

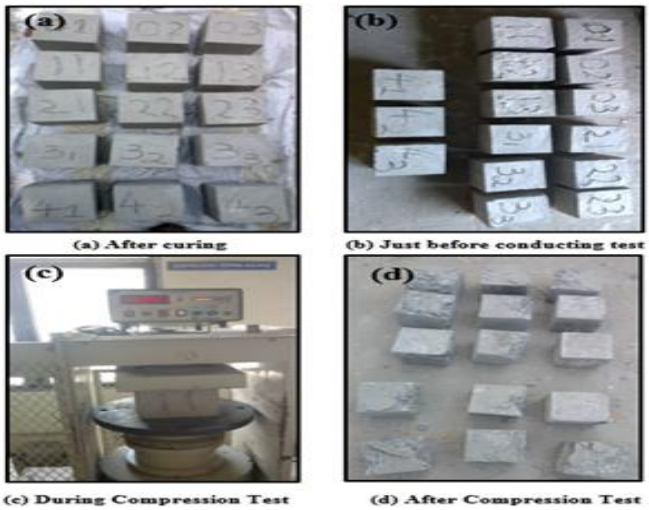

Fig 7: Compression test for cubes.

V. RESULTS & DISCUSSION

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) and9.12 g of Iron (III) Nitrate nonahydrate.

B. Characterizations of TiO2/Fe2O3 nanocomposites

1. X-ray Diffraction Analysis

In order to identify the crystalline nature of synthesized nanocomposites, X-ray diffractogram (XRD) is used.

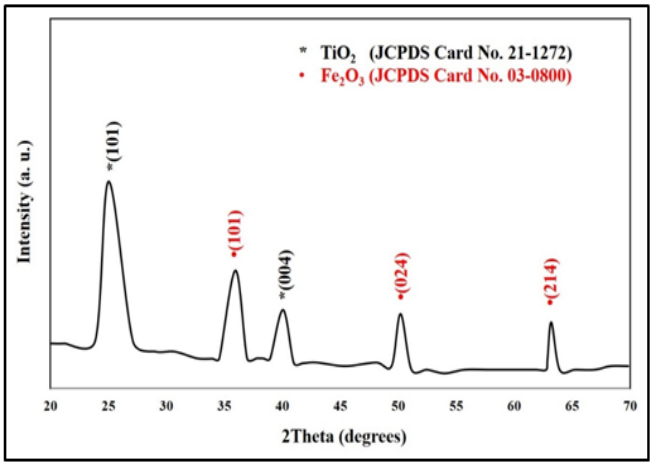

Fig 8: XRD analysis of TiO2/Fe2O3 nanocomposites.

The diffractogram pattern was indexed properly for all crystalline peaks and compared with JCPDS data file (Fig 8).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) [46, 70]. 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) [71]. From the "Debye- Scherrer" relation, Crystallite size,

$$\mathbf{t = \frac{0.9\,\lambda}{ß\cos\theta}}, \quad (2)$$

where, t = Crystallite Size,

β = Full Width at Half Maxima,

θ = Bragg angle and

λ =Wave length of X- Rays (for copper target 1.54 Å or 0.154 nm).

TABLE V: CALCULATION OF CRYSTALLITE SIZE OF THE NANOCOMPOSITES

S.No.	2 θ°	h k l°	ß (degrees)	ß (radians)	particles	t (nm)
1	25.04	101	2.38	0.041	Anatase TiO_2	3.462
2	35.98	111	0.004	0.006	Fe_2O_3	4.362
3	40.08	004	1.08	0.018	Anatase TiO_2	8.196
4	50.03	200	0.84	0.014	Fe_2O_3	10.924
5	63.22	200	1.6	0.027	Fe_2O_3	6.027

Average crystallite size of TiO_2/Fe_2O_3 nanocomposites = 6.59 ± 2.6 nm. The composite shows crystalline phase peaks of both TiO_2 & Fe_2O_3 and hence confirms the composite formation.

$$t = \frac{0.9\,\lambda}{ß\,\cos\theta}$$

$$t = \frac{0.9 * 0.154}{(0.041) * \cos(12.52)}$$

$$t = 3.462 \text{ nm}$$

$$\text{Mean } t = 6.59 \text{ nm}$$

$$\text{Variance} = 7.285$$

Standard deviation = √Variance = 2.6

2. 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.9). The SEM image shows the spherical shape of the TiO_2 nanoparticles. However, α-Fe_2O_3 nanoparticles were agglomerated and did not possess any definite shape.

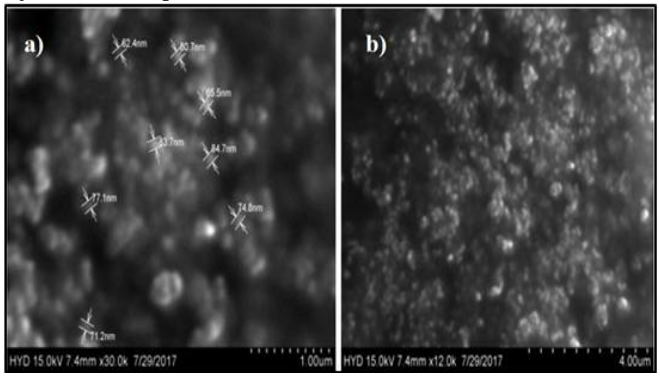

Fig 9: SEM analysis of TiO2/Fe2O3 nanocomposites.

3. FTIR Analysis

FTIR analysis has performed to identify functional groups on the surface of nanomaterials.

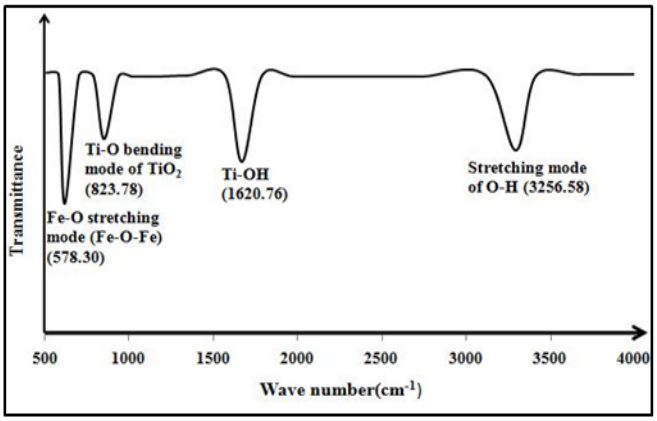

Fig 10: FTIR analysis of TiO2/Fe2O3 nanocomposites.

The broad band observed at 3256.58 cm^{-1} was assigned to stretching vibrations of (O-H) of Fe_2O_3, and the peak at 583.54 cm^{-1} represents to Fe-O stretching mode [72]. The band at 1620.76 cm^{-1} was corresponds to deformative vibration of Ti-OH stretching modes and the band at 823.78 cm^{-1} corresponds to the Ti-O bending mode of TiO_2(Fig. 10)[73].

4. Ultra Violet-Visible (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 spectra of TiO_2-Fe_2O_3 nanocomposites. Here absorbance is observed at 390nm and 560nm, which indicates the wavelength of TiO_2, Fe_2O_3 nanoparticles respectively (Fig.11)[45].

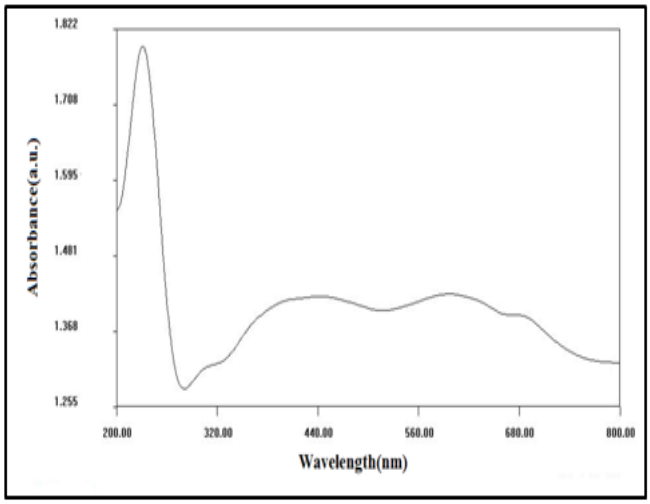

Fig 11: Uv-Vis Spectrum Analysis of Tio2/Fe2o3 nanocomposites.

C. 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 [77] was procured, and following results obtained according to the appropriate tests conducted in laboratory.

TABLE VI: TEST 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

Locally available natural river sand conforming to IS 383: 1970 [75] has been used as fine aggregate. It has tested for physical and mechanical properties results as follows:

TABLE VII: SPECIFIC GRAVITY OF FINE AGGREGATE

S.No.	Description	Weight (grams)
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

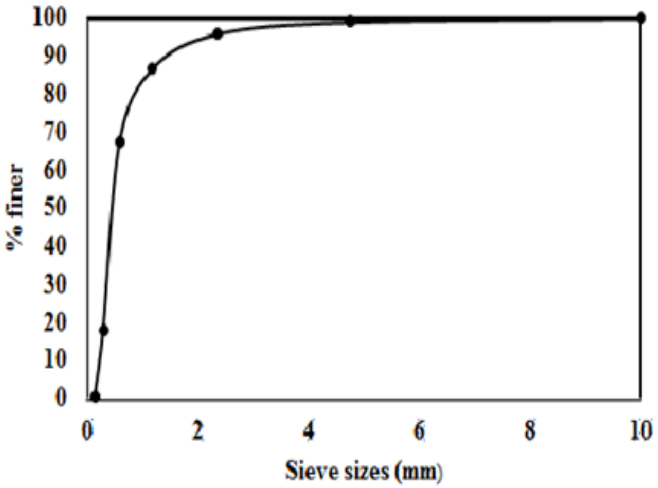

Fig 12: Grading curve of fine aggregate.

TABLE VIII: FINENESS MODULUS OF FINE AGGREGATE BY SIEVE ANALYSIS

LS Sieve Designation	Weights			% finer	Zone-III limits as per IS 383: 1970 (Table4)
	Retained weight of Fine aggregate (grams)	Percentage weight retained	Cumulative % weight retained		
10mm	0	0	0	100	100
4.75mm	24	0.96	0.96	99.04	90 – 100
2.36mm	78	3.12	4.08	95.92	85 – 100
1.18mm	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	-

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 having limit of 2.6 - 2.8 [76]. Hence the selected fine aggregate (sand) was safe for the production of satisfactory concrete.

Fineness modulus = (sum of cumulative % weight retained from sieve 4.75 mm -150 µm)/100

$= 232.48/100 = 2.32$

From Table VIII the fine aggregate (sand) belongs to Zone-III from Table4 of IS 383: 1970 (Appendix A5) and the sand is Fine sand (Fineness modulus: 2.2 - 2.6).

3. Test results of coarse aggregate

The Machine crushed gravel conforming to IS 383: 1970 has obtained from the local quarry has used as coarse aggregate [75]. It has tested for physical and mechanical properties results as follows:

TABLE IX: SPECIFIC GRAVITY OF COARSE AGGREGATE

S.No.	Description	Weight (grams)
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 $= \frac{(W_2-W_1)}{((W_4-W_1)-(W_3-W_2))}$

$= 2.72$

TABLE X: PARTICLE SIZE DISTRIBUTION OF COARSE AGGREGATE BY SIEVE ANALYSIS

LS Sieve	Weights			% finer	Grade limits as per IS: 383-1970 (Table2)
	Weight Retained(grams)	% 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	-

As per IS2386 (part 3): 1963 the specific gravity of coarse aggregate having limit of 2.6 - 2.8 [76]. Hence the selected coarse aggregate (crushed stone) was safe for the production of satisfactory concrete. From Table X the coarse aggregate (crushed stone) belongs to single sized aggregate of nominal size of 20 mm fromTable2 of IS 383: 1970 (Appendix A5).

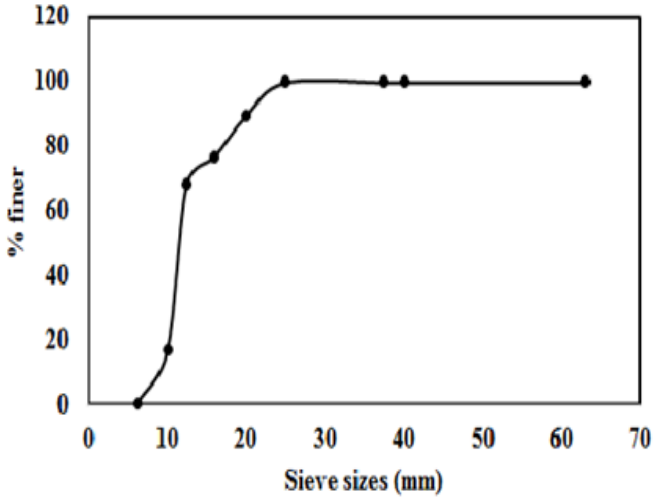

Fig 13: Grading curve of coarse aggregate.

D. Mix design of M20 grade concrete

For studying the effect of TiO_2/Fe_2O_3 nanocomposites on compressive strength of M20 grade concrete mix was considered. The M20 Mix design had carried out using ISI methods i.e., IS 10262: 2009 [78] and IS 456: 2000 [74]. The mix proportion obtained is 1:1.65:2.99 with constant water cement ratio 0.5. The design procedure as follows:

Grade designation : M20
Type of cement : ACC 53 grade
Maximum nominal size of aggregate: 20 mm
Exposure conditions : Severe
Minimum cement content : 320 kg/m^3
Maximum water cement ratio : 0.5
Type of aggregate : Crushed stone (gravel)
Maximum cement content : 450 kg/m^3
Degree of supervision : good

1. Test data for materials

Cement Specific gravity : 3.15
fine aggregate specific gravity : 2.72
grade of zone : Zone-III
coarse aggregate (gravel - 20 mm)
specific gravity : 2.68

2. Target mean strength for mix proportioning

$$F_{ck} = f_{ck} + 1.65 * S \quad (3)$$

Where, F_{ck}= Target mean compressive strength at 28 days(MPa)
f_{ck} = Characteristic compressive strength at 28 days (MPa) = 20 MPa
S = Standard deviation

From Table 1 of IS 10262: 2009 (Appendix A5), standard deviation, S = 4 N/mm^2. Therefore, target mean strength F_{ck}= 20 + 1.65*4 = 26.6 MPa

3. Selection of water-cement ratio

From see note 4.1 of IS 10262: 2009 [82], water-cement ratio = 0.45
From Table 5 of IS 456: 2000 (Appendix A5), maximum water-cement ratio = 0.5
Therefore, adopt water-cement ratio = 0.5

4. Selection of water content

From the Table 2 of IS 10262: 2009 (Appendix A5), the maximum water content for 20 mm aggregate = 186 liters (25 to 50 mm slump range)
Estimated water content for 100 mm slump = 186+ [(6*186)/100] = 197 litres

5. Calculation of cement content

Water-cement ratio= 0.5
Cement content = (197/0.5) =394 kg/m^3
From Table 5 of IS 456: 2000 (Appendix A5), minimum cement content for moderate condition and M20 grade of concrete is 320 kg/m^3.
Therefore, adopt cement content =394 kg/m^3.

6. Proportion of coarse aggregate and fine aggregate content

From Table 3 of IS 10262: 2009 (Appendix A5), the volume of coarse aggregate corresponding to 20 mm size coarse aggregate and fine aggregate (zone III) is taken as below:

Therefore, Volume of proportion of coarse aggregate = 0.64.
Volume of fine aggregate = 1-0.64 =0.36.

7. Mix calculations

The mix calculations per unit volume of concrete as described as follows.

Volume of concrete = 1 m^3
Volume of cement= (394/3.15)*(1/1000) =0.1251m^3
Volume of water = (197/1000) = 0.197m^3
Volume of all in aggregate =(1-0.1251-0.197)=0.6779m^3
Mass of coarse aggregate =0.6779*2.72*0.64*1000 =1180.08 kg
Mass of fine aggregate =0.6822*2.68*0.36*1000= 654.037 kg

8. Actual quantities of mix proportions

Cement: Fine aggregate` : Coarse aggregate : Water
1 : 1.65 : 2.99 : 0.5

Mix proportions of cement, fine aggregate and coarse aggregate (by density) is given by

Cement	394 kg/m^3
Fine aggregate	654.037 kg/m^3
Coarse aggregate	1180.08 kg/m^3
Water	197 /m^3

9. Calculations of nanocomposites requirement in grams

- 10% fly ash replacement by nanocomposites up to 2 wt% for 2 cubes of each specimen as follows:
  Density of fly ash = 394*(10/100) = 39.4 kg/m^3

Modified density of cement =394 - 39.4 = 354.6 kg/m^3
Volume of 2 cubes = 0.15*0.15*0.15*2*1.1
= 0.007425 m^3
Weight of cement = 0.007425*354.6 = 2.632 kg
Weight of fly ash = 0.007425*39.4= 0.2925 kg

Required amount of nanocomposites (0.5, 1.0, 1.5 and 2.0 wt%) for replacementof fly ash is 14.623 grams.

- 5% fly ash replacement by nanocomposites up to 2 wt% for 3 cubes of each specimen as follows:
  Density of fly ash = 394*(5/100) = 19.7kg/m^3
  Modified density of cement = 394–19.7=374.3kg/m^3
  Volume of 3 cubes = 0.15*0.15*0.15*3*1.1
  = 0.0111375 m^3
  Weight of cement = 0.0111375*374.3 = 4.168 kg
  Weight of fly ash = 0.0111375*19.7 = 0.2194 kg

Required amount of nanocomposites (0.5, 1.0, 1.5 and 2.0 wt%) for replacementof fly ash 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.

10. Mix proportions

The constituents of mix proportions for M20 grade concrete (Table XI) and the composition of five different mixes were designated (Table XII).

TABLE XI: 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 XII: 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

E. Compressive Strength of Concrete

The compressive strength of cubes was determined by following formula:

$$\text{Compressive strength} = \frac{\text{Load (N)}}{\text{Area (mm}^2\text{)}} \quad (4)$$

Here, the area is the surface area of the cube
= 150 mm * 150 mm.
= 22500 mm^2

TABLE XIII: 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			

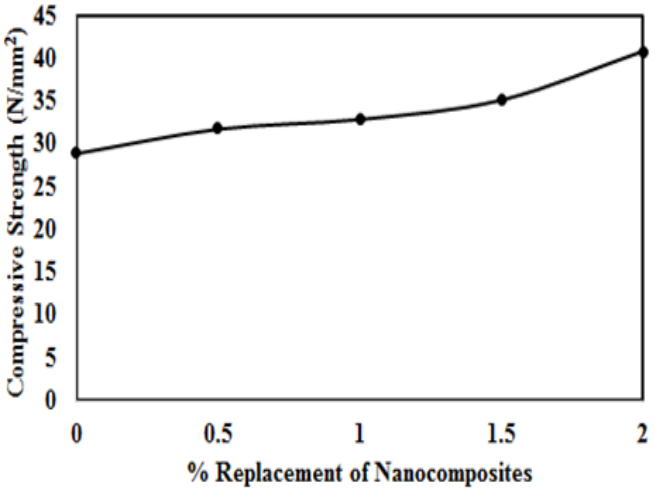

Fig 14: Compressive Strength vs % replacement of nanocomposites.

The compressive strength of concrete cubes was increased by gradually increasing TiO_2/Fe_2O_3 nanocomposites up to 2.0 wt% of fly ash. 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 [1]. 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 [28]. Thus, increasing the compressive strength of the concrete.

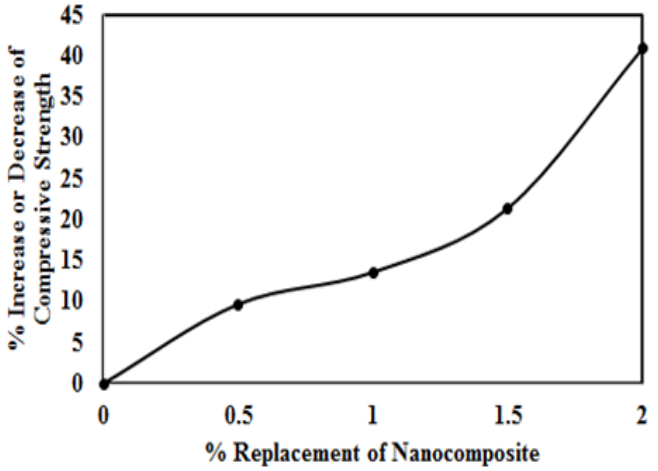

Fig 15: Percentage change of compressive strengthvs % replacement of nanocomposites.

VI. CONCLUSIONS

The following conclusions are carried out from the present study:

- The TiO_2/Fe_2O_3 nanocomposites were successfully synthesized by using sol-gel method.
- From UV-Visible spectra the peaks were observed at 390 nm for TiO_2 and at 560 nm for Fe_2O_3.
- From XRD analysis, the averagecrystallite size of anatase TiO_2 and Fe_2O_3 was estimated to be 6.59±2.6 nm using Debye-Scherer formula.
- From FTIR analysis both Fe and Ti functional groups were observed.
- From SEM analysis, it is observed that aggregated Fe_2O_3 nanoparticles were surrounded by the TiO_2 nanoparticles and average particle size is around 75 nm.
- From compression test, the concrete cubes made of TiO_2/Fe_2O_3 nanocompositesgave better compressive strength than normal concrete cubes.Strength enhancement of 40.9% was achieved by gradually increasingTiO_2/Fe_2O_3 nanocomposites up to 2.0 wt% of fly ash.

VII. APPENDICES

A. Characterization Techniques

1. Ultraviolet-Visible (UV) Spectroscopy

Ultraviolet-visible Spectroscopy is used for quantitative analysis and structural elucidation. The wavelength of UV radiation starts from 200 to 400 nm. The wavelength of visible radiation starts from 400 to 800 nm. UV-Vis spectroscopy is classified as a single beam and double beam instruments. In single beam instruments, there is an interchange of sample and reference solutions for each wavelength. The most flexible, general purpose spectrophotometers are double beam automatically recording types. The double beam design provides two equivalent paths for radiation, both originating from the same source. One of these beams travels across the sample, while the other passes through an identical cuvette containing reference material. The two beams are measured separately by detectors.

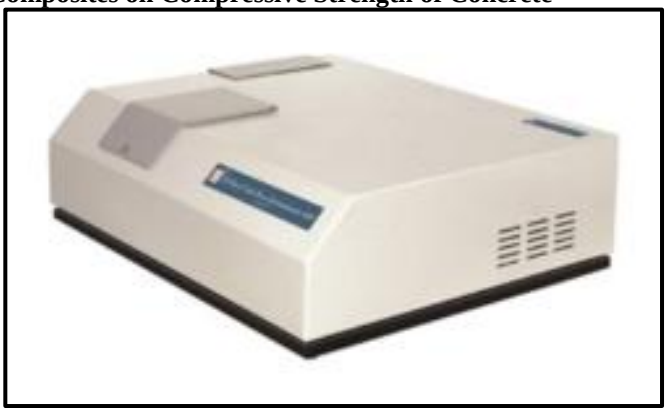

Fig 17: Systronics Double Beam UV Spectrophotometer - 2201

The recorder plots the wavelength of the entire region versus the absorption (A) of light at each wavelength. In the spectrum of nanoparticles, the adsorption peak's width strongly depends on the chemical composition and the particle size. As a result, their spectrum changes from their bulk ones.

2. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy is a famous characterization approach in which a sample is placed in among the direction of an IR radiation supply and its absorption of various IR frequencies is measured. IR photon energies, in a selection among 1 and 15 kcal/mol, are insufficient to excite electrons to better digital strength states but transitions in vibrational electricity states. these states are related to a molecule's bond. every molecule has its own specific identity. In FTIR spectrometry, sample absorbs all of the unique wavelengths characteristic of its spectrum and this subtracts particular wavelength from the interferogram. The detector now reviews variant in energy as opposed to (vs) time for all wavelengths concurrently. A laser beam is superimposed to provide a reference for the device operation.

Fig 18: Bruker - FTIR device - ALPHA Eco – ATR.

A mathematical function – Fourier transform allows us to convert intensity (vs) time spectrum to intensity (vs) frequency spectrum. FTIR spectroscopy may be used to investigate a huge range of materials in solid, liquid and gaseous stages. It produces now not handiest qualitative evaluation but with applicable requirements may be used for quantitative analysis. FTIR can be used to analyze the pattern with a diameter up to ~11mm and measured either in bulk or the top ~1 micrometer layer thick. FTIR spectra of natural compounds are normally so precise that they're like a fingerprint for the identification of a molecule.

3. X-Ray Diffraction (XRD) Method

XRD is a completely essential experimental technique that has lengthy been used to address all issues associated with the crystal shape, lattice constants, identification of unknown materials, orientation of unmarried crystals, and many others. In XRD, a collimated beam of X-rays, with a wavelength generally ranging from zero.7 to two Å, is incident on a specimen and is diffracted through the crystalline stages in the specimen according to Bragg's regulation: $\lambda = 2d\sin\theta$, in which d is the spacing between atomic planes within the crystalline section and λ is the X-ray wavelength. The depth of the diffracted X-rays is measured as a characteristic of the diffraction attitude 2θ and the specimen's orientation.

Fig 19: WRD PANalytical "XPERT 3 PRO".

This diffraction pattern is used to identify the specimen's crystalline levels and to measure its structural houses. X-ray diffraction best provides the collective information of the particle sizes and usually requires a massive quantity of powder. It must be cited that since the estimation might paintings simplest for terribly small particles, this approach may be very beneficial in characterizing nanoparticles.

4. Scanning Electron Microscope (SEM)

SEM is one of the maximum widely used techniques used withine characterization of nanomaterials and nanostructures. The decision of the SEM tactics some nanometers, and the units can function at magnifications that are without difficulty adjusted from ~10 to over three hundred,000.

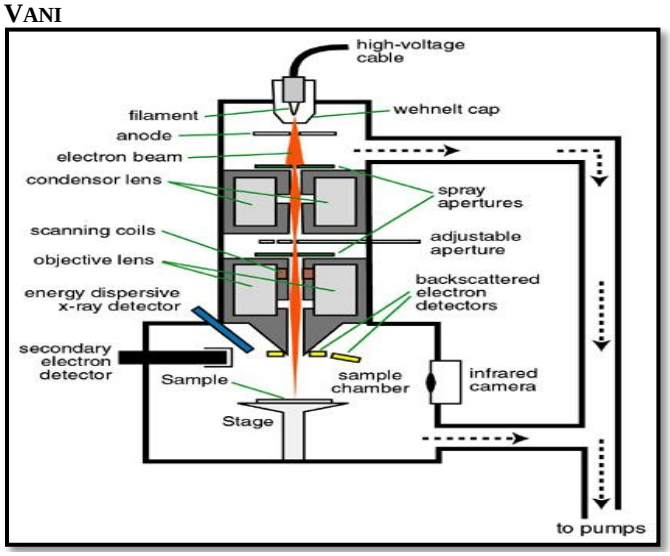

Fig20. Schematic diagram of Scanning Electron Microscope.

In a typical SEM, a supply of electrons is focused into a beam, with a totally quality spot length of ~five nm and having energy ranging from a few hundred eV to 50 keV this is rastered over the floor of the specimen via deflection coils. As the electrons strike and penetrate the floor, some of interplay arise that bring about the emission of electrons and photons from the sample, and SEM photograph is produced by gathering the emitted electrons on the cathode ray tube (CRT).

B. Basic Tests for Cement

1. Normal consistency test

The standard consistency of a cement paste is defined as that consistency in an effort to permit the Vicat plunger to penetrate to some extent five to 7 mm from the bottom of the Vicat mildew. to start with a cement sample of about three hundred g is taken in a tray and is mixed with a recognised percent of water by means of weight of cement, say beginning from 26% and then it's far accelerated by way of every 2% until the normal consistency is completed.

Fig 21: Vicat mould setup for 53-grade cement

2. Setting time test

The Vicat mildew is full of three hundred g of cement paste is ready via gauging the cement with 0.eighty five times the water required to provide a paste of standard consistency by resting it on a nonporous plate. it's far located beneath the rod bearing the needle and permitting it to penetrate into the test block. The preliminary putting time will be recorded by measuring fails to pierce the block past 5.0 ± 0.five mm from the bottom of the mould. similarly, the very last putting time shall be recorded by changing the needle of the Vicat equipment with an annular attachment. The length elapsing among the time whilst water is added to the cement and the time at which the needle makes an affect at the surface of check block even as the attachment fails to achieve this shall be the final placing time.

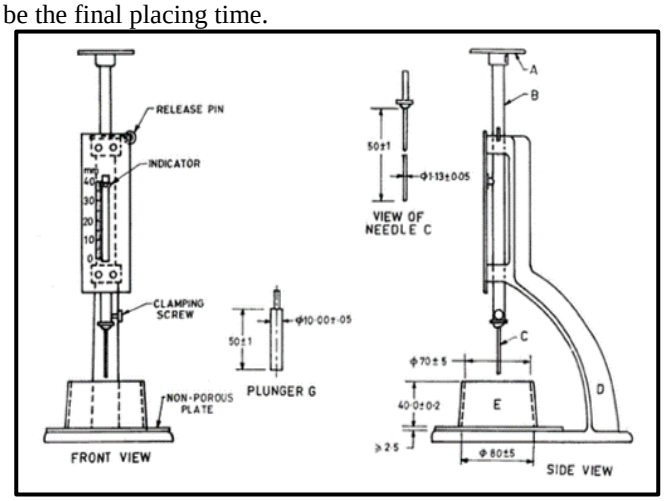

Fig 22: Vicat Mould Apparatus.

3. Fineness modulus test

The fineness of grinding has multiplied through the years. however now it has got nearly stabilized. extraordinary cements are floor to different fineness. Fineness of cement is examined by means of sieving. The ninety-micron sieve is used for sieving the cement by taking 10 g of cement sample. The tray and lid are arranged to the sieve to avoid the lack of cement. The sieve is agitated by swirling, planetary and linear movement till no greater fine cloth is exceeded via it. right here the residue gathered inside the tray is weighed. Then the fineness modulus is calculated with the aid of weight percent of retained pattern of cement inside the 90 micron sieve.

Fig 23: Sieve of 90 micron

4. Specific gravity test for cement

The Le-Chatelierflask is packed with kerosene free of water having a specific gravity no longer much less than 0.7313 to a point at the stem between zero and 1 ml mark. The flask is immersed in a regular temperature water tub and the studying is recorded.

Fig 24: Le-Chatelier Flask.

A weighed quantity of cement (about 64 g of Portland cement) is then brought in small quantities at the equal temperature as that of the liquid. After introducing all the cement, the stopper is placed in the flask and the flask rolled in an inclined function, or lightly whirled in a horizontal circle, so one can free the cement from air till no similarly air bubbles upward push to the surface of the liquid. The flask is again immersed in the water-tub and the final reading is recorded. The distinction among the first and the very last analyzing represents the volume of liquid displaced by the load of the cement used in the test.

B. Basic Tests for Aggregates

1. Specific gravity test for fine aggregate

The empty pycnometer of 500 ml extent is cleaned and weighed (weight W1). The floor-dry pleasant aggregate (sand) is taken in pycnometer approximately 3/4th extent and weighed (weight W2). The last 1/4th quantity is filled with distilled water and weighed (weight W3). finally, the wiped clean pycnometer is packed with distilled water and weighed (weight W4). Calculations - Specific gravity was calculated as follows:

$$\text{Specific gravity} = \frac{(W_2 - W_1)}{((W_4 - W_1) - (W_3 - W_2))} \quad (5)$$

Fig 25: Pycnometer.

2. Sieve Analysis (Fineness Modulus) Test for Fine Aggregate

Fig 26: sieves For Fine Aggregate.

The amount of surface-dry fine aggregates (sand) about 2.5 kg is taken and passed through sieves of 10 mm, 4.75 mm, 2.36 mm, 1.18 mm, 600 microns, 300 microns, 150 microns and pan. The fineness modulus is calculated as the percentage of the sum of cumulative weight of fine aggregate retained in the sieves.

3. Specific Gravity Test for Coarse Aggregate

The empty pycnometer of 500 ml volumeis cleaned and weighed (weight W_1). The surface-dry coarse aggregate (crushed stone) is taken in pycnometer about 2/3rd volume and weighed (weight W_2). The remaining 1/3rd volume is filled with distilled water and weighed (weight W_3). Finally, the cleaned pycnometer was filled with distilled water and weighed (weight W_4). Calculations - Specific gravity was calculated as follows:

$$\text{Specific gravity} = \frac{(W_2 - W_1)}{((W_4 - W_1) - (W_3 - W_2))}$$

4. Sieve Analysis Test for Coarse Aggregate

The particle size distribution of coarse aggregates has been calculated by using sieve analysis.

Fig 27: sieves For Coarse Aggregate.

The amount of coarse aggregates (crushed stone) about 5 kg was taken and passed through sieves of 60 mm, 40 mm, 37.5 mm, 25 mm, 20 mm, 16 mm, 12.5 mm, 10 mm, 6.3 mm and pan.

C. Gallery

1. Synthesis of TiO_2/Fe_2O_3 Nanocomposites using Sol-Gel method

(a) (b)

Fig 28: (a) Magnetic Stirring (b) Centrifugation

(a) (b)

Fig 29: (a) After Centrifugation (b) Vacuum Oven.

(a) (b)

Fig30: (a) Muffle Furnace (b) Fine powder of Nano-composites.

2. Basic Tests for Concrete Materials


(a) (b)

Fig 31: (a) Vicat mould setup (b) Sieve of 90 microns.


(a) (b)

Fig 32: (a) Le-Chatelier Flask (b) Pycnometer.


(a) (b)

Fig 33: (a) Sieves for Fine Aggregate (b) Sieves for Coarse Aggregate

3. Application of nanocomposites in concrete


(a) (b)

Fig 34: (a) Weighing Fly ash (b) Fly ash with different amounts of nanocomposites


(a) (b)

Fig 35: (a) Sieving sand with 4.75 mm sieve (b) Weighing Sand.


(a) (b)

Fig 36: (a) Weighing Crushed Stone/Gravel (b) Weighing Cement.


(a) (b)

Fig37(a) While mixing concrete (b) After mixing concrete.


(a) (b)

Fig 38: (a)Applying oil on moulds (b) Compacting concrete in moulds

Fig 39: Vibration for proper mixing.

Fig 40: After Casting.

Fig 41: After de-moulding the cubes.

Fig 42: Curing the cubes in water After curing.

Fig 43: Before testing the cubes.

4. Compression Test for Concrete Cubes

Fig 44: Compression Testing Machine (CTM) Testing the cube in CTM.

Fig 45: After Compression testing.

D. Material Safety Data of Chemicals

1. Titanium (IV) isopropoxide–Ti{OCH(CH$_3$)$_2$}$_4$

Precautions for safe handling

- Additional hazards when processed: Handle empty containers with care because residual vapors are flammable.
- Precautions for safe handling: Avoid all eye and skin contact and do not breathe vapor and mist. Provide good ventilation in process area to prevent accumulation of vapors. Take precautionary measures against static discharge. Containers and transfer lines require grounding during use. Use only non-sparking tools.
- Hygiene measures: Wash hands and other exposed areas with mild soap and water before eating, drinking or smoking and when leaving work. Wash contaminated clothing before reuse.

Conditions for safe storage,including any incompatibilities

Technical measures: Ground/bond container and receiving equipment. Proper grounding procedures to avoid static electricity should be followed. Use explosion-proof electrical equipment.

Storage conditions: Keep container tightly closed.

Incompatible materials: Moist air, Oxidizing agent, Water.

Storage area: Store in a well-ventilated place. Store away from heat.

2. Iron (III) nitrate nonahydrate – Fe(NO$_3$)$_3$.9H$_2$O

Precautions: Keep away from heat. Keep away from sources of ignition. Keep away from combustible material. Do not ingest. Do not breathe dust. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes.

Storage: Oxidizing materials should be stored in a separate safety storage cabinet or room.

3. Ethanol– CH$_3$CH$_2$OH

Precautions: Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, acids, alkalis, moisture.

Storage: Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame). Do not store above 23°C (73.4°F).

4. Ammonia solution 25% – NH$_3$

Precautions: Keep locked up. Keep container dry. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as metals, acids.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area. Do not store above 25°C (77°F).

E. IS Code Tables

TABLE XIV: IS 456:2000 (Table5)

Table 5 Minimum Cement Content, Maximum Water-Cement Ratio and Minimum Grade of Concrete for Different Exposures with Normal Weight Aggregates of 20 mm Nominal Maximum Size

(Clauses 6.1.2, 8.2.4.1 *and* 9.1.2)

Sl No.	Exposure	Plain Concrete			Reinforced Concrete		
		Minimum Cement Content kg/m³	Maximum Free Water-Cement Ratio	Minimum Grade of Concrete	Minimum Cement Content kg/m³	Maximum Free Water-Cement Ratio	Minimum Grade of Concrete
1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Mild	220	0.60	-	300	0.55	M 20
ii)	Moderate	240	0.60	M 15	300	0.50	M 25
iii)	Severe	250	0.50	M 20	320	0.45	M 30
iv)	Very severe	260	0.45	M 20	340	0.45	M 35
v)	Extreme	280	0.40	M 25	360	0.40	M 40

NOTES

1 Cement content prescribed in this table is irrespective of the grades of cement and it is inclusive of additions mentioned in 5.2. The additions such as fly ash or ground granulated blast furnace slag may be taken into account in the concrete composition with respect to the cement content and water-cement ratio if the suitability is established and as long as the maximum amounts taken into account do not exceed the limit of pozzolona and slag specified in IS 1489 (Part 1) and IS 455 respectively.

2 Minimum grade for plain concrete under mild exposure condition is not specified.

TABLE XV: IS 10262:2009 (Table1)

Table 1 Assumed Standard Deviation

(*Clauses* 3.2.1.2, A-3 *and* B-3)

Sl No. (1)	Grade of Concrete (2)	Assumed Standard Deviation N/mm² (3)
i)	M 10	3.5
ii)	M 15	
iii)	M 20	4.0
iv)	M 25	
v)	M 30	5.0
vi)	M 35	
vii)	M 40	
viii)	M 45	
ix)	M 50	
x)	M 55	

NOTE — The above values correspond to the site control having proper storage of cement; weigh batching of all materials; controlled addition of water; regular checking of all materials, aggregate grading and moisture content; and periodical checking of workability and strength. Where there is deviation from the above, values given in the above table shall be increased by 1 N/mm².

TABLE XVI: IS 10262:2009 (Table2)

Table 2 Maximum Water Content per Cubic Metre of Concrete for Nominal Maximum Size of Aggregate
(*Clauses* 4.2, A-5 *and* B-5)

Sl No.	Nominal Maximum Size of Aggregate	Maximum Water Content [1]
	mm	kg
(1)	(2)	(3)
i)	10	208
ii)	20	186
iii)	40	165

NOTE — These quantities of mixing water are for use in computing cementitious material contents for trial batches.

[1] Water}content corresponding to saturated surface dry aggregate.

TABLE XVII: IS 10262:2009 (Table3)

Table 3 Volume of Coarse Aggregate per Unit Volume of Total Aggregate for Different Zones of Fine Aggregate
(*Clauses* 4.4, A-7 *and* B-7)

Sl No.	Nominal Maximum Size of Aggregate	Volume of Coarse Aggregate[1] per Unit Volume of Total Aggregate for Different Zones of Fine Aggregate			
	mm	Zone IV	Zone III	Zone II	Zone I
(1)	(2)	(3)	(4)	(5)	(6)
i)	10	0.50	0.48	0.46	0.44
ii)	20	0.66	0.64	0.62	0.60
iii)	40	0.75	0.73	0.71	0.69

[1] Volumes are based on aggregates in saturated surface dry condition.

TABLE VIII: IS 383:1970 (Table2)

TABLE 2 COARSE AGGREGATES

(*Clauses* 6.1 *and* 6.2)

IS Sieve Designation	Percentage Passing for Single-Sized Aggregate of Nominal Size						Percentage Passing for Graded Aggregate of Nominal Size			
	63 mm	40 mm	20 mm	16 mm	12·5 mm	10 mm	40 mm	20 mm	16 mm	12·5 mm
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
80 mm	100	—	—	—	—	—	100	—	—	—
63 mm	85 to 100	100	—	—	—	—	—	—	—	—
40 mm	0 to 30	85 to 100	100	—	—	—	95 to 100	100	—	—
20 mm	0 to 5	0 to 20	85 to 100	100	—	—	30 to 70	95 to 100	100	100
16 mm	—	—	—	85 to 100	100	—	—	—	90 to 100	—
12·5 mm	—	—	—	—	85 to 100	100	—	—	—	90 to 100
10 mm	0 to 5	0 to 5	0 to 20	0 to 30	0 to 45	85 to 100	10 to 35	25 to 55	30 to 70	40 to 85
4·75 mm	—	—	0 to 5	0 to 5	0 to 10	0 to 20	0 to 5	0 to 10	0 to 10	0 to 10
2·36 mm	—	—	—	—	—	0 to 5	—	—	—	—

TABLE VIX: IS 383:1970 (Table4)

TABLE 4 FINE AGGREGATES'
(*Clause* 4.3)

IS Sieve Designation	Percentage Passing for			
	Grading Zone I	Grading Zone II	Grading Zone III	Grading Zone IV
10 mm	100	100	100	100
4·75 mm	90-100	90-100	90-100	95-100
2·36 mm	60-95	75-100	85-100	95-100
1·18 mm	30-70	55-90	75-100	90-100
600 micron	15-34	35-59	60-79	80-100
300 micron	5-20	8-30	12-40	15-50
150 micron	0-10	0-10	0-10	0-15

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