

EXPERIMENTAL INVESTIGATION OF THE STRUCTURAL PERFORMANCE OF CONCRETE CONTAINING CELLULOSE FIBRES AND THE ANTI-CORROSIVE EFFECT OF GREEN CORROSION INHIBITORS

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Abstract

This research was aimed to find the structural behaviour of *Calotropis procera* fibres added concrete and the effect of *Azadirachta indica* leaf slurry blended to concrete and exposed to seawater. Conventional and fibre reinforced concrete samples fabricated keeping the curing time as 7, 14 and 28 days were subjected to compression, split tensile, rebound hammer and ultrasonic pulse velocity test. Increase in curing time increased the structural properties and *Calotropis procera* fibres added samples produced respectively 19.5%, 15%, 10.3 and 14.5% improvement in the compression, tensile, rebound hammer and ultrasonic pulse velocity values. These fibres reduced the brittleness of the specimen and avoided unprecedented failures. Accelerated corrosion and gravimetric mass loss test conducted to estimate the anti-corrosive property of *Azadirachta indica* leaf slurry showed decrease in corrosion rate. The corrosion rate of concrete samples without and with inhibitors was 0.0654 ± 0.008 and 0.056 ± 0.011 mm/year respectively evincing the anti-corrosive effects of *Azadirachta indica* leaves. Thus green materials are compatible with concrete and can be used in making sustainable concrete structures.

Keywords: *Calotropis procera* fibres, fibre reinforced concrete, structural properties, accelerated corrosion test, green corrosion inhibitors

1. INTRODUCTION

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Concrete is an important construction material which possesses a very high compressive strength and a very low tensile strength (about one-tenth of compressive strength) and is consumed to a tune of 7.5 billion tons annually [1, 2]. They withstand heavy loads until they are cracked and after which they do not transfer stress leading to collapse. In order to avoid this, and to improve the durability and reduce brittleness concrete is reinforced using fibres and this is called as fibre reinforced concrete (FRC) [3]. Steel fibres as rebars or mesh are usually used for making structural elements. These structures are effective in withstanding load and are better than conventional concrete. FRC is thus a cement based composite material that hosts fibre as reinforcements. The role of fibres is to improve durability. FRC needs to meet certain building codes and guidelines followed in some countries while steps are undertaken to include them in upcoming Model codes also. Many different types of concretes such as self compacting concrete, ultra-high performance concrete and hybrid fibres reinforced concrete are available and are suited for specific applications making it as a material of complexity. The properties that are required for concrete are good compressive strength, flexural strength, and tensile strength, setting time, seamless flow of mixture of dense reinforcement into the complicated shape, longest durability and extended economic life of the structures [4]. FRC should be designed to reduce pores and cracks, impervious to chemicals, resistance to absorb moisture and resist corrosion.

Commonly used fibres in FRC are steel fibres, carbon fibres, basalt fibres, and glass and polypropylene fibres. In addition, nowadays emphasis on sustainability has caused the usage of bio-fibres or cellulose fibres derived from plants as reinforcements in concrete [5, 6]. The type of fibre material, volume or weight fraction of fibres and slenderness ratio of the fibres pose significant influence on the structural properties of concrete. Fibres with different volume fractions and slenderness ratios have produced considerable effects on the strength of the concrete. Optimally designed concrete mix is needed to balance strength and workability. Mere increase in fibre volume does not pronounce positive effect on the concrete since more fibres need more plasticizers to improve workability. Architectural applications mostly use glass or polypropylene fibres since they reduce plastic shrinkage cracks during the early days of concrete and thus increase the post-cracking feature of concrete. American Concrete Institute (ACI) (2009) had listed certain factors in the use of vegetal fibres as reinforcements in concrete. For a constant volume fraction of fibres, it was reported that fibre length influences the compression strength of the concrete. Short fibres impose more compression strength than the long fibres.

Materials derived from plants were used in concrete as a part of sustainability and they are called green materials. Kriker et al. (2005) used date palm fibres of varying lengths and vol% to reinforce concrete. It was found that inclusion of high volume% of fibres into the concrete mix reduced the compression strength to 90%, while 3wt% fibres of 60mm length reduced the compression strength to 55% when compared to the control concrete specimens [7]. The reason for this behaviour was mentioned as the non-uniform nature of the fibres and the increased number of defects found in high volume% fibres. Yazici et al. [8] used different fibre volumes (0.5, 1.0, and 1.5 %) and aspect ratios (45, 65, and 80) in concrete mix. It was found that compressive, tensile and flexural strength values increased 10, 14 and 58% respectively with high aspect ratio fibres. Bagasse fibres with volume percentages of (3%, 8%, and 12%) were used as reinforcement for fly ash based composite material by Tian et al. (2016) [9]. Increased fibre content caused 50% reduction in compressive strength during early stages (7 days). However after 28 days, the difference was not so high and inclusion of fibres was found to positively improve the mechanical strength of the concrete and also it was reported by many researchers that fibre content would improve the compression strength and reduce the brittleness of concrete [10-11]. Excess fibre loading caused deterioration of the properties and is a major design concern [11, 12]. De Klerk (2020) used 3wt% of sisal fibre in concrete and reported increase in tensile strength and flexural strength

by 14% and 11% respectively [13]. Thus it is clear that fibre type, fibre length, fibre morphology and fibre proportion, control the mechanical properties of concrete [14-15]. Self compacting concrete possess high fluidity and does not require any compaction but produces a dense and homogeneous mixture [16]. Mechanical and durability studies of concrete containing varying vol% of discarded rubber tyre particles were investigated by Nadi et al. (2022). It was reported that rubberized concrete mixtures had better workability and lower density than the control mixes along with decreasing compression strength [17]. Cellulose fibres added concrete was demonstrated by Jamshaid et al. (2022) to control the water absorption and mechanical performance of concrete [18]. The structural behaviour of concrete made using two varying wt proportion (0, 10, 20 and 30) of different sands namely granite waste sand and tile waste sand was studied by Kherraf et al. (2023)[19]. It was concluded that the addition of granite sand till 20wt% improved compression strength and anti-corrosion properties, while the split tensile increased for 30wt% sand addition. In a study on adding cellulose fibres to concrete, it was seen that 1-1.5wt% of bamboo fibres added to concrete showed appreciable increase in structural parameters of concrete [20]. A study on eco-friendly concrete reinforced using palm fibre has shown that concrete containing 1wt% of these fibres and cured for 28 days produced maximum compression strength [21]. It was also seen that crack propagation was resisted due to fibre addition. Researchers have reported that fibre type, fibre content, curing time and fibre geometry are the major factors influencing the strength of the concrete.

Corrosion is a common phenomenon that causes a material to deteriorate and a metal when corroded will lose its load standing capability and fails at low loads. The rate of corrosion depends on the area of the corroding rebar, mass flow rate and the environmental conditions. Many researchers had conducted research on corrosion, its effects on the structural properties of concrete and also found some ways to reduce it. Studies conducted to reduce corrosion had shown that usage of galvanized steel bars with high performance concrete delayed depassivation. Gravimetric weight loss method and electrochemical methods are techniques that are used to study corrosion and its effects. Gravimetric weight loss method is a simple technique that does not use current or voltage. Immersion test is also used to study corrosion related problems. Pradhan and Bhattacharjee (2009) found out that Portland Pozzolana Cement is efficient in resisting chloride ingress and rust formation [22]. Corrosion inhibitors are used to protect the structures from getting corroded. These inhibitors improve the strength and life of the buildings against corrosion. Of course, they need capital expenditure and many studies are reported to reduce corrosion and improve the reinforcements made of steel [23]. Synthetic materials are mostly commonly used to avoid corrosion. In spite of the availability of many corrosion inhibitors, natural materials are also used as corrosion inhibitors. Jain et al. (2014) conducted studies on mild steel in acidic environment (HCl and HNO₃) [24] using extracts of *Azadirachta indica*. The study showed that the extracts were found to possess anti-corrosive properties. Similarly Ajanaku et al. (2015) had shown that Al metal in 1.85M HCl was found to be protected against acidic attacks with the extracts of Neem leaves [25]. The effect of corrosion on the concrete beams subjected to sustained loads was studied by Mahmoud Miri et al. (2019) using normal concrete beams and beams treated with nano-wollastonite. It was found that the treated beams outperformed normal concrete in terms of service life, decreased mass loss and crack initiation time [26]. In a study on the accelerated corrosion by Kharma et al (2022), mass loss of 9 and 16.2% were noticed for a period of 48 and 96 days respectively [27]. Recent study by Kumar Harish et al. (2022) showed that *Azadirachta indica* leaves are very good anti-cracking, and anti-pitting agents for mild steel with 89.25% corrosion inhibition efficiency [28]. Considering the serious long term consequences of corrosion, researchers conduct many studies to reduce their effect on structures [29, 30]. Both synthetic and organic corrosion resistors are studied for their effectiveness in reducing corrosion.

With this background, the current study was proposed and conducted. The main aim of this study was to find the effect of adding *Calotropis procera* cellulose fibres as secondary fillers in concrete mix. Previously many studies were conducted on *Calotropis procera* fibres in reinforcing polymer composites. But as far as our knowledge is concerned, studies on *Calotropis procera* fibres added concrete are hardly found. *Calotropis procera* plant belongs to the Asclepidaceae family with flowers resembling a cup. It is a native of Africa and is found in Africa, Asia, Caribbean islands and in China. This plant is rich in fibres with annual yield annual more than 700 kg/ha. *Calotropis procera* fibres were currently used by researchers as reinforcements in polymer composites [31] and were found to influence the mechanical and thermal properties of the composites [32]. Neem tree also called as *Azadirachta indica* is a green material with a wide scope of opportunities for almost all applications containing anti-inflammatory, anti-pyretic, analgesic, immunostimulant, anti-fertility, anti-carcinogenic, anti-malarial, and hepatoprotective properties. It is known as “Vembu” in Tamil, “Arishth” in Sanskrit and is called as “Village Dispensary” in India. Recognizing its prudence, US National Academy of Sciences, declared “Neem – a tree for solving global problems” in an official report during 1992. In this study, conventional concrete samples and *Calotropis procera* fibres reinforced concrete samples were fabricated. Compression test, split tensile test, rebound hammer test and ultrasonic velocity test were conducted both on the conventional concrete samples and *Calotropis procera* fibres added concrete samples with 7, 14 and 28 days of curing. Results obtained were compared and the effect of adding cellulose fibres was studied and reported. In addition, the usage of slurry made from *Azadirachta indica* leaves in resisting corrosion was found out by conducting accelerated corrosion and gravimetric weight loss test on the concrete samples. The effect of natural fibres on the structural properties of concrete is gaining interest among researchers nowadays [33]. The novelty of this research is the addition of two green materials namely *Calotropis procera* and *Azadirachta indica* to concrete. The flowchart of this present research is shown in Figure 1.

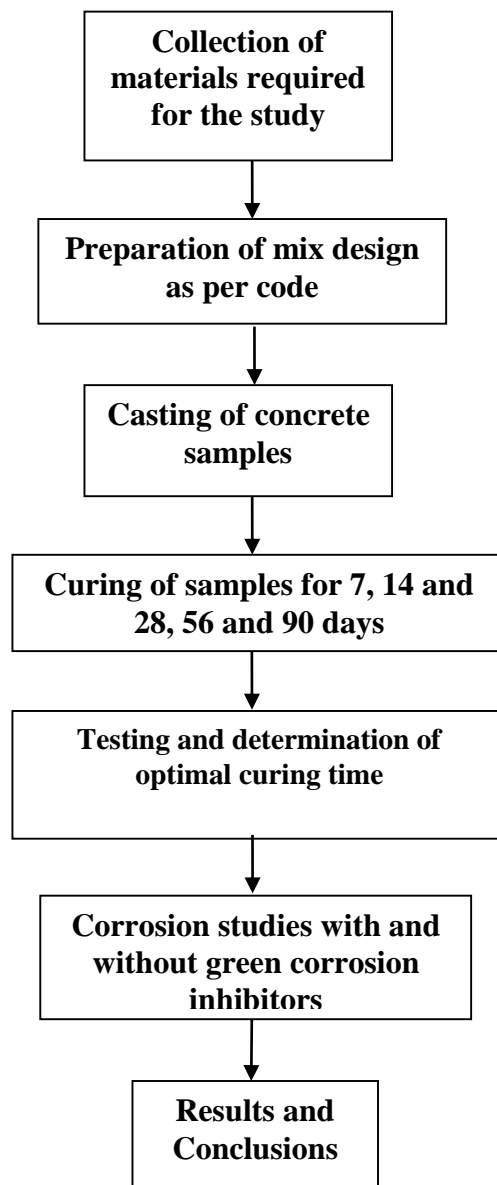


Fig. 1. Flowchart of the research

2. MATERIALS AND METHODS

2.1. Materials

The raw materials used in this present study include cement, aggregates (coarse and fine) and steel bars as reinforcement and seawater. *Calotropis procera* fibres were extracted by water retting technique from the stem of a plant. The extracted fibres were washed thoroughly in normal water and are dried under

sunlight to remove moisture content. Then they were cut into 15 - 20mm lengths and are used in the concrete mix for the present study. Figure 2 shows the *Calotropis procera* plant. The chemical composition of the *Calotropis procera* fibres is shown in Table 1.



Fig. 2. *Calotropis procera* plant

Table 1. Chemical composition of *Calotropis procera* fibres

Ingredients	Composition [wt %]
Cellulose	54.5
Hemicellulose	12.3
Lignin	14.8
Ash	7.6
Moisture content	9.3
Wax	1.5

2.1.1 Cement

Cement is the most important binder that integrates discrete ingredients made from naturally found raw materials and is mainly preferred for construction purposes. In this present work 43 grade Portland Pozzolana Cement (IS: 1498-1976) was used. Table 2 shows the important properties of the cement.

Table 2. Properties of Cement

Ingredients	Desired Range of Percentage [wt %]
(CaO)	62 to 67
Silica (SiO ₂)	17 to 25
Alumina (Al ₂ O ₃)	3 to 8
Calcium Sulphate (CaSO ₄)	3 to 4
Iron Oxide(Fe ₂ O ₃)	3 to 4
Magnesia (MgO)	0.1 to 3
Sulphur (S)	1 to 3
Alkali	0.2 to 1

2.1.2 Fine aggregates

The fine aggregate refers to the materials that are less than 4.75mm in size and were obtained from pits, river, lake or seashores. In this study, river sand was obtained from a nearby quarry. The specific gravity and water absorption of the fine aggregates were found to be 2.66 and 2.5% respectively. The sieve analysis data of sand confirmed IS: 383-1970 standards [34].

2.1.3 Coarse aggregates

Coarse aggregate refers to the material that remains retained on a 4.75mm sieve and they were between 6 and 10.5mm in size in this study. They were obtained after crushing granites, limestone, schist, and good quality sand stones. The coarse aggregate had a specific gravity of 2.74 and the water absorption was found as 1.0%. The coarse aggregates conformed to IS: 383-1970 specifications [34]. Figure 3 shows the grading curve of fine and coarse aggregates.

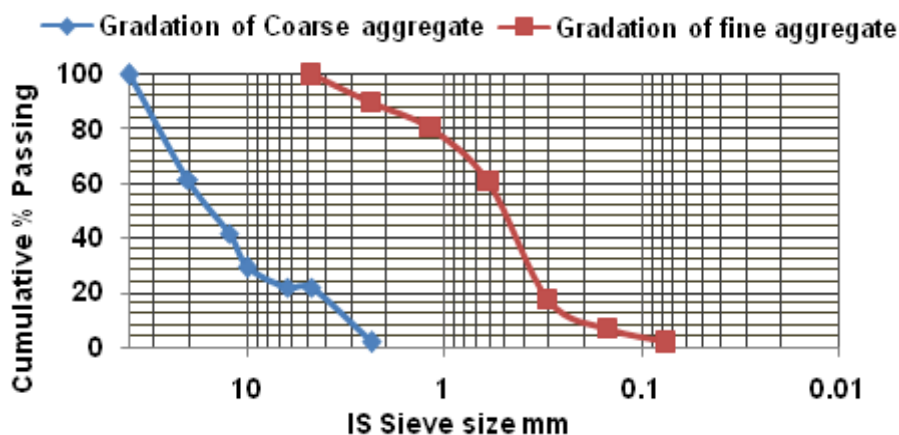


Fig. 3. Grading curve of fine and coarse aggregates

2.1.4 Water

Water is used as a lubricant for the fine and coarse aggregate and forms binding paste for the aggregate. In this study sea water was used.

2.1.5 *Calotropis procera* fibres

In this study fibres were extracted from the bast of the *Calotropis procera* shrub by water retting process. The extracted fibres were washed thoroughly to remove impurities present in them. They were cut into uniform lengths and used for the study.

2.1.6 *Azadirachta indica* leaves

In this study *Azadirachta indica* (Indian Neem) leaves were used as corrosion inhibitor. It is a natural material which is non-toxic, bio-degradable and possesses superior anti-bacterial and anti-corrosive property as compared with other fibres. The leaves were initially plucked and washed with water several times and then they were made as slurry using normal water. 2wt% of *Azadirachta indica* slurry was blended to the concrete mix to find the influence of corrosion inhibitors on the corrosion rate of the steel bars.

2.2. Methods

2.2.1 Mix design

The selection of appropriate mixture of cement, fine aggregate, coarse aggregate and the relation between the water/cement ratio and target strength are the bases for the mix design. In this present study, the maximum water/cement ratio was maintained as 0.5 and the mix ratio was taken as per IS 10262 standards [35]. Portland cement and sand was measured and blended to obtain a homogeneous mixture. Water was constantly added to the mixture until they were equally distributed. The concrete mix was then placed in an appropriate mould and made suitable for conducting various tests. The samples were covered with a damp hessian sack for 24 hrs and then remoulded and put into temperature-controlled water tanks at 23°C for 28 days. The mix ratio considered in this present study is shown in Table 3.

Table 3. Mix proportion of 1m³ of concrete

Cement [kg]	412
Water [kg]	186
Fine aggregate [kg]	721
Coarse aggregate [kg]	1161.6

2.2.2 Specimen preparation

The concrete cube specimens were made in the shape of a cube of 15cm side. The test specimens are made after mixing to produce full compaction of the concrete without segregation and excessive laitance. All these specimens were cast both in conventional and fibre reinforced types. *Calotropis procera* fibres of 1wt% were added as secondary fillers for preparing fibres reinforced composites. The age of the concrete specimens were selected as 7, 14 and 28 days for testing. These time periods were selected as the samples remain unaffected during the tests [36].

2.3. Experiments

2.3.1 Preliminary tests

The specific gravity of the cement was found using pycnometer and kerosene as liquid of immersion. Similarly the specific gravity of coarse and fine aggregates was found employing pycnometer using standard techniques. The workability of fresh concrete was determined in line with IS 1199-1959 standards using slump cone, tamping rod and consistometer [37]. Figure 4 shows the slump test conducted on the concrete mix. The initial setting time and the consistency tests were performed using a Vicat's apparatus. For this, a cement paste was prepared using cement with 0.85 times the water to get a paste of standard consistency. All the tests were repeated thrice and the average value was reported.



Fig. 4. Slump test

The concrete samples prepared for conducting the tests are shown in Figure 5.



Fig. 5. Concrete samples

2.3.2 Compressive strength test

A compressive test on the prepared concrete samples was carried out on steel cube moulds of dimensions 150 mm× 150 mm×150 mm in accordance with IS 516: 1959 standards using a universal testing machine [38]. The specimen was aligned centrally on the base plate of the machine. The sample faces were kept intact with the plane surfaces of the mould and compression load was applied till the specimen failed. Ultimate load at fracture was recorded and the compressive strength was calculated. The test was conducted three times and the average value was specified. Figure 6(a-d) shows the experimental setup of the tests conducted on the concrete samples.

2.3.3 Split tensile strength test

The split tensile test on the prepared concrete samples was carried out on Universal Testing Machine in accordance with IS 5816: 1999 standards [39]. A plywood strip was kept above and below the sample and the load was continuously applied without shock at a constant rate until the specimen fails. This

breaking force was recorded and the split tensile strength was calculated. The test was conducted three times and the average value was specified.

2.3.4 Rebound hammer test

A Rebound hammer test was carried out on the concrete specimens cured for 28 days in accordance with IS 13311(Part 2):1992 standards using a Schmidt rebound hammer [40]. The surface of the sample was made flat by grinding and the test was conducted by placing the plunger perpendicular to the concrete surface. Three readings were taken from each test area on two opposite faces in the non-casting direction and the average value was calculated. The test was conducted three times and the average value was specified.

2.3.5 Ultrasonic Pulse Velocity (UPV) Test

The ultrasonic pulse velocity test was tested on the concrete members cured for 28 days as described in IS 13311:1992 codes [40]. The pulse velocity was measured using the path length and time of travel of an ultrasonic pulse passing through the concrete. This test is used to detect discontinuities cracks and internal deterioration in the structure of concrete. The test was conducted three times and the average value was specified.

2.3.6 Accelerated corrosion test

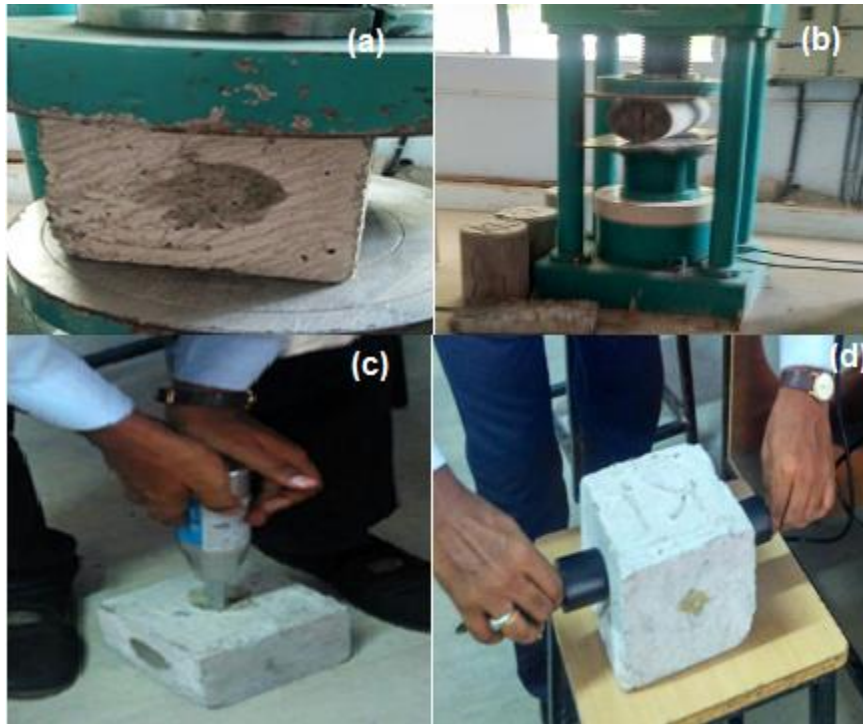
The initiation time of corrosion was found out using accelerated corrosion test. Concrete cylinders of 300mm length and 150mm diameter were cast using 16mm high yield strength deformed steel rebars at the centre. These rods were cleaned and degreased using pickling acid and a PVC sleeve was used to cover the protruding part of the rod. The specimens were cured for 28 days, dried for 24hours and then they were immersed in 3.5% Sodium chloride solution. A constant voltage of 12V was supplied to develop potential difference between the rebar connected to the positive terminal (anode) and the stainless steel plate connected to the negative terminal (cathode) of the power pack and the response of the current was noted [41]. A total of six samples were tested, out of which three samples contain *Azadirachta indica* slurry as corrosion inhibitor in the concrete mix while other three samples are control samples without corrosion inhibitor. In real time environment, significant corrosion damage is felt after few years and is practically difficult to assess the time and rate of corrosion. Hence an ideal environment is simulated in lab to find the rate and time of corrosion.

2.3.7 Gravimetric studies

Steel embedded concrete specimens of 150mm diameter and 300mm length and were cast with and without inhibitors. Steel rebars of 16mm diameter and 400mm length were placed centrally in the concrete cylinders. The initial mass of these rebars was noted and the concrete samples were cured under marine water for 28 days. Then they were immersed in 3% NaCl solution under alternate wetting and drying conditions for 24 hours. At the end of 90th day, the samples were broken and the steel rebars were weighed. Test was conducted three times and the average value was reported. The difference in mass was used to find the corrosion rate in mm per year using the formula in Equation 1 as per ASTM G1-03 standards [42].

$$\text{Corrosion rate (mm/year)} = (87.6 \times W) / D \times A \times T \quad (1)$$

Where W is the weight loss in milligrams, D is the density of the metal in gm/cm³, A is the surface area of the cylindrical specimen and T is the immersion period in hours.



Figures6(a-d) Experimental Setup of compression, split tensile, rebound hammer and ultrasonic pulse velocity tests

3. RESULTS AND DISCUSSION

3.1 Preliminary test results

The setting time and the specific gravity tests conducted on the cement showed that the values are 33 minutes and 3.5 respectively. The specific gravity of the coarse aggregate and fine aggregate were found as 2.74 and 2.66 respectively. Slump test helps to identify whether the freshly made concrete is consistent and can be placed, mixed and compacted. When the slump cone is filled with freshly prepared concrete mix, unsupported fresh concrete flows through the sides and settles down vertically. This sinking height is called as slump. The slump test conducted on the fresh concrete shows the consistency or wetness of the fresh concrete (See Figure 4). It helps to find the ease of flow of concrete and also an improperly mixed concrete. As per IS 456-2000 standards, reinforced cement concrete for beams and slabs should have a slump value in the range 50 - 100mm. Too low and too high values need to be corrected before use. Figure 4 shows the slump test conducted on the concrete mix. In this present study, the slump value was 65mm and the workability of the concrete was found as 10 seconds. The concrete is devoid of segregation and bleeding ensuring that it is workable. According to the classification of slump values, the concrete is said to have medium workability. This is similar to the results obtained for sisal fibres reinforced concrete [43, 44].

3.2 Compressive strength

The compression test conducted on the 7 days cured conventional concrete showed that the average compressive strength was 12.78 ± 0.98 MPa. The concrete cubes were able to withstand load in the range 306 -365 kN. The compressive strength of the concrete samples cured for 14 days was 17.39 ± 0.89 MPa (Mean \pm Standard Deviation) and they were able to withstand load of 370 - 410 kN. This showed that the compressive strength increases with increase in curing time. For a curing time of 28 days, the average compressive strength of the concrete sample exhibited a value of 21.07 ± 1.97 MPa. It is clear that the strength increases with increasing curing time. This is similar to the results of other researchers [45, 46]. This evinces that the cellulose content in fibres are able to provide mechanical strength to the concrete mix [47, 48]. The increase in the compressive strength was 36% and 64.87% respectively for 14 and 28 days when compared to the compression strength of the sample with 7 days of curing. The compression strength values are shown in Figure 7.

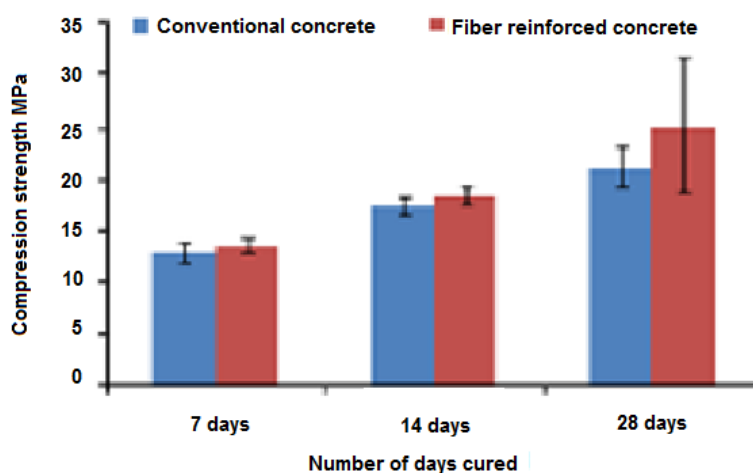


Fig. 7. Compression strength of the specimens

In case of fibre reinforced values the same trend was observed however the values are higher than conventional concrete specimens. The compressive strength of the fibre reinforced concrete specimen were 13.52 ± 0.98 MPa, 18.4 ± 0.88 MPa and 25.18 ± 6.48 MPa respectively for a curing time of 7, 14 and 28 days respectively. This is due to the reinforcing capability of the *Calotropis procera* fibres that help in load bearing phenomenon. It was reported that the addition of jute fibres have caused an increase in the compression strength [12]. The inclusion of fibres improved the compression strength to 36.1% and 86% respectively for curing time of 14 and 28 days. Adding fibres to a minimal level increased the compression strength and this is because of the opposing effect of the fibres to make the concrete samples expand. On external compression loading, these fibres withstand the stress and prevented the expansion of samples along the lateral side, thereby resulting in increased compression strength. But the addition of fibres should be limited to 2% since more fibre volume causes compaction problems and the samples fail [43, 44]. The % increase expected with addition of plant fibres is about 19.5% and the present study exceeds this value. As already quoted, this may be due to the load withstanding capability of the fibres [49]. The conventional concrete specimen broke abruptly and collapsed during the compression test as against the fibre reinforced concrete.

3.3 Split tensile strength

The tensile strength of concrete specimens was found out using this indirect tension test method. The split tensile strength of the concrete specimen cured for 7 days showed a tensile strength of 1.63 ± 0.78 MPa. When the curing was extended to 14 days, the split tensile strength of the concrete samples increased to 2.49 ± 0.22 MPa, a 52.7% increase. This shows that curing time is directly related to the tensile strength of the specimens. On increasing the curing time to 28 days, the split tensile strength of the specimens exhibited a tensile strength of 2.99 ± 0.32 MPa. This is 83.4% increase as compared to the tensile strength of the samples at 7 days curing. This shows that increase in curing time improves the tensile strength of the concrete similar to previous reports [48, 49, 50]. The aggregates and cement present in the concrete undergoes a chemical reaction involving evaporation which makes the linkages stable and hard. Concrete gains strength during the first two weeks and the process is rapid. During this period, concrete gains maximum strength and the curing process continues to gain full strength (about 98%) in about 28 days. Figure 8 depicts the split tensile strength of the concrete specimens.

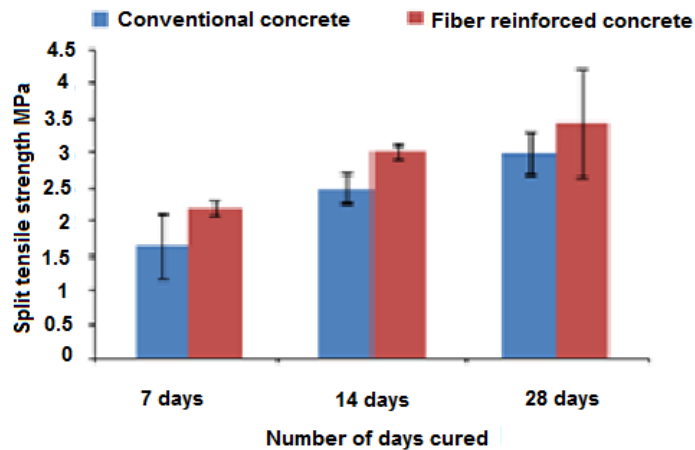


Fig. 8. Split tensile strength of the specimens

In case of fibre reinforced concrete specimens the split tensile strength was observed as 2.2 ± 0.1 MPa which is 35% more than the conventional concrete. The improvement in split tensile strength of the fibre reinforced specimens is due to the high tensile modulus of the fibre used as compared to that of concrete [12,49]. In case of fibre reinforced concrete specimen with 14 days curing the value observed was 3.03 ± 0.11 MPa. This is 22% more than the tensile strength of the conventional concrete. Fibres present in the concrete increase its elasticity and delay the occurrence of the cracks [52]. The fibre reinforced specimen after 28 days curing showed a split tensile strength of 3.44 ± 0.79 MPa, a value 15% more than the conventional concrete proving that the inclusion of fibre has resulted in improved tensile strength. Micro-fractures that may be present in the concrete tend to develop further causing failure. But the fibres added to the concrete controls the fractures during curing and handling. It was reported that the tensile strength of the alfa fibres (*Stippatenacissima*) reinforced concrete increased up to 54.41 % compared to the ordinary concrete in line with the current study [53]. It was reported that 1.2vol % is the optimum fibre content to produce high tensile strength in concrete [54].

The conventional and fibre reinforced concrete specimens cured for 56 and 90 days does not produce significant improvement in the structural properties. Hence in this study 28 days was taken as the curing time and reported.

3.4 Failure mode of tested samples

The failure mode of the concrete sample may be broadly discussed as failure by constituents of the concrete mix and the type of failure in general. The general failure modes of the concrete samples may be any one of the following modes namely conical, shear, split, conical and shear, and conical and split. The failure may occur slowly by the initiation of crack, development of crack followed by propagation of cracks. The type of failure depends on the concrete mix, ingredients and loading, Figure 9 shows the failure mode of the conventional concrete samples and fibre reinforced concrete samples observed in this present study. The concrete cylinders that were subjected to axial compression developed minor cracks at the vicinity of the jaws and then they gradually developed towards the centre of the cylinder along its length. At a certain point, the developed cracks caused splitting and failure. In case of fibre reinforced concrete samples, tiny cracks nucleated in places where there are no fibres. These cracks propagated progressively into the interior of the concrete samples. When the cracks propagate, the cellulose fibres present inside the samples took up the stresses caused by the external loading and hindered crack propagation resulting in high compressive strength. The reason for the higher compressive strength of the fibre reinforced concrete was ascribed to the fibre addition which restricted crack growth and changed the crack path allowing the sample to withstand high loads. The cellulose fibres in the concrete mix support hydration pore refinement and strengthen the bonding making them stronger due to their hydrophilic nature. Also, it was reported that the lignocellulose fibres are capable of determining the failure mode of the composites through their fibre architecture [55]. Fibre inclusion decreases cracking and forms a closed packing structure.

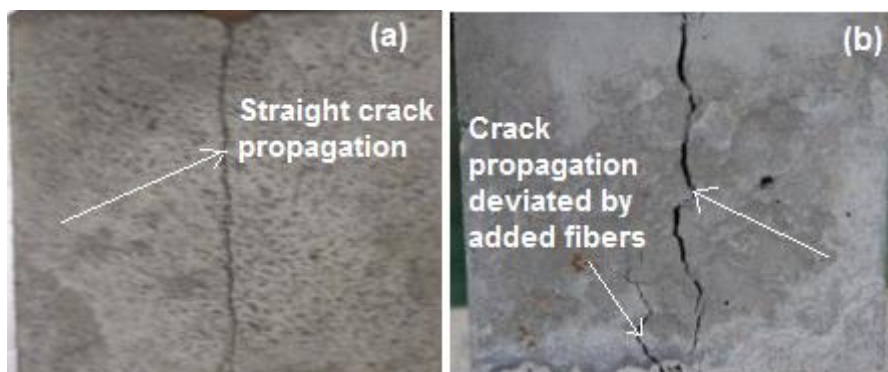


Fig. 9. Crack propagation in conventional and fibre reinforced concrete samples

The constituents of the concrete mix exhibit different failure modes such as interface failure, mortar failure, and aggregate failure; with the first two failures related to low loading rates while the third one relates to high failure rate[53]. Other failure modes are exterior failure, interior failure and complete failure. In this study, the presence of *Calotropis procera* fibres within the concrete protected it by delaying the exterior failure from being propagated towards inside causing interior and complete failure during external loading. It was reported in literature that tensile deformation is vital in determining the

formation of cracks, crack width, number of cracks, etc and are used to assess the corrosion level [54, 56].

3.5 Rebound hammer test

The rebound hammer test conducted on the ordinary concrete samples is shown in the Figure 10. The conventional concrete specimen with 28 days curing showed a rebound compression test value of 19.33 ± 2.3 . As per ASTM C566-19 standards, Rebound hammer number lying between 20 and 30 is considered fair and acceptable [57]. This value depends upon the surface of the material tested. High rebound values may be obtained, if the surface in contact with the rebound hammer is hard and smooth [58]. In case of fibres reinforced concrete sample, the values are 21.33 ± 1.15 . This value is 10.3% higher than the normal concrete specimen evincing that fibre inclusion produced improved strength and can be a constituent in concrete mix. Rough surface and surface with irregularities produce low rebound hammer values.

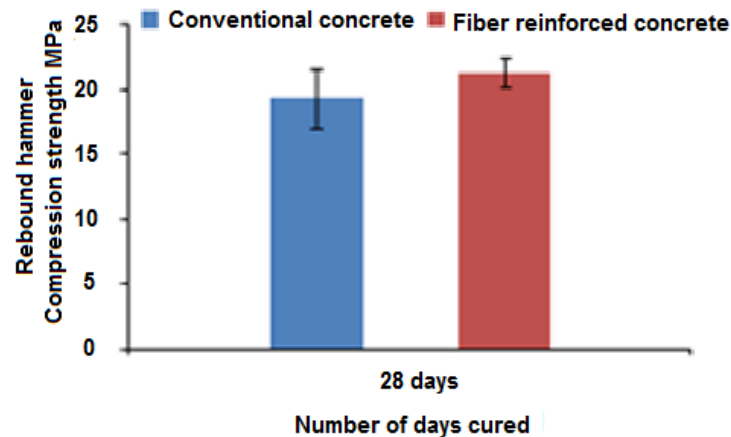


Fig. 10. Rebound hammer test of the specimens

3.6 Ultrasonic Pulse Velocity (UPV) Test

Detection of cracks and internal deterioration in the structure of concrete were found by conducting ultrasonic pulse velocity test. This non-destructive study helps in finding the concrete's strength. In the present study, the Ultrasonic Pulse Velocity of the conventional concrete samples cured for 28 days was found as 4010 ± 876 m/s. This value is comparable to the values reported in the literature [53]. In case of fibre reinforced concrete specimen the mean ultrasonic pulse velocity for 28 days curing was 4606 ± 100 m/s. This is 14.9% more than that of the conventional concrete implying short time range and high wave speeds. Pulse velocity values in the range 3500 – 4500 m/s are considered as a good quality concrete [57] and the present values fall in this range. The adhesion between steel bars and matrix becomes more compact on fibre addition. The Ultrasonic pulse velocity test conducted on the samples is shown in Figure 11.

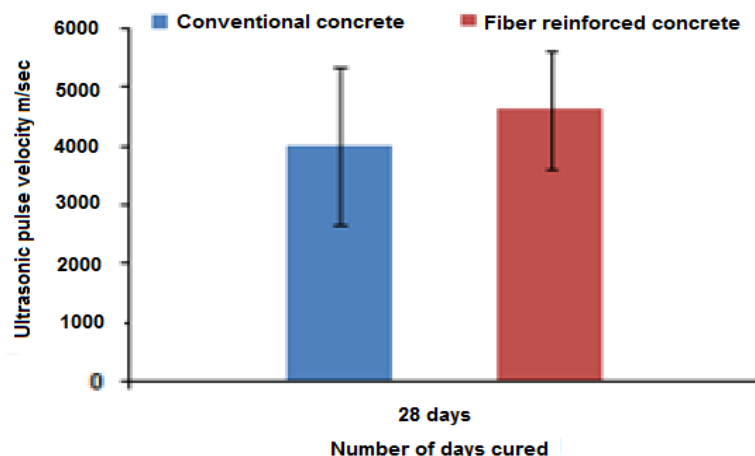


Fig. 11. Ultrasonic Pulse Velocity test of the specimens

The compression strength of conventional concrete sample with 56 days and 90 days curing was in the range 21 – 21.32 MPa. Similarly, for the fiber reinforced concrete the corresponding values were between 25 - 26 MPa. On comparing these values with the values obtained for conventional (21.07MPa) and fiber reinforced concrete (25.18MPa), no significant improvement in values was seen. Hence they are ignored and 28 days was maintained as optimum curing time and reported.

3.7 Validation of results

The results obtained in this study are validated from the results available in literature. It was reported that the compression strength of the concrete samples increased on adding jute fibres with values ranging between 17MPa and 25MPa for 0.1 to 0.5wt% fibres. Optimum strength was obtained with 0.1wt% fibre loading [58]. The compression strength of concrete reinforced with jute, sisal, sugarcane bagasse and coir fibres were reported as 6.75, 5.52, 4.05, 4.34 MPa respectively [59]. The compression test values obtained in this study thus agrees well the values available in literature. Similarly, split tensile strength of the jute fibres concrete samples varied from 1.7MPa to 3MPa for different fibre loadings (0.1, 0.25, 0.5wt%). 0.25wt% jute fibres added concrete exhibited maximum split tensile strength [58]. Most of the studies consider the curing time as 28 days. Concrete samples reinforced using spikelet and stalk fibres of oil palm empty fruit bunch fibre of (1-3wt% and 10-30mm length) were reported to have produced a positive effect on the split tensile strength. Optimal split tensile strength of 4.5MPa was obtained with 2wt% and 15mm length of spikelet fibres. On the other hand, stalk fibres have exhibited a maximum split tensile strength of 3.2MPa with 3wt% fibres of 20mm length [60]. These values are also fairly comparable with the results of this present study. The ultrasound speed test conducted on the tiling sand and granite sand added concrete exhibited ultrasonic pulse velocity values in the range 4320 – 4750m/sec and 4520 – 4790 m/sec respectively [19]. These values too are concomitant with the values obtained in this research. Thus the results were validated with those available in literature.

In addition, a statistical analysis showing the empirical relationship between the structural properties of the concrete with and without fibres was done using trend line equations. Compression strength and split tensile strength was related and it can be observed that there exists a linear relationship between them. It is obvious from the Figure 12 that the correlation coefficient varies

between 0.993 – 0.997, both values nearing unity. These high values of correlation coefficient confirm the strong relationship between the structural properties of both conventional concrete and fibre reinforced concrete. Similarly, the rebound hammer test values of control concrete samples obtained in this study (19.33 MPa) fairly agree with the compression strength of the conventional concrete samples (21.07 MPa). Likewise, the compression strength value of fibre reinforced concrete sample (25.18 MPa) too agrees with the rebound hammer test value (21.33MPa) of fibre reinforced concrete sample. The slight deviation in value is attributed to the experimental conditions and the causal differences existing between the samples tested.

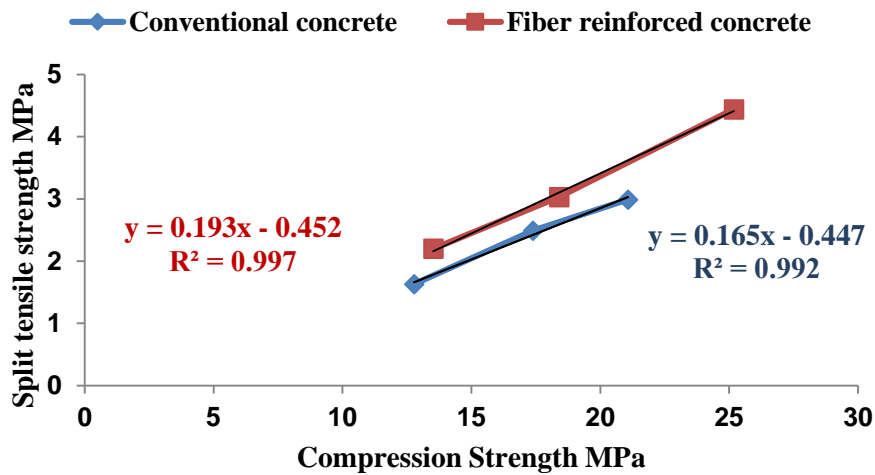


Fig. 12. Statistical analysis of Compression strength Vs Split tensile strength

3.8 Accelerated corrosion test

The study conducted on the concrete samples without corrosion inhibitors showed a more or less constant corrosion current with slight fluctuations during the start of the test. The corrosion current was seen to vary from $60\mu\text{A}$ and extended slowly to reach $264\mu\text{A}$ and $384\mu\text{A}$ respectively after 120 and 144 hours. Then, in between a period of 156 and 180 hours (6.5 – 7.5 days), the corrosion current increased suddenly from $582\mu\text{A}$ and the value reached $734\mu\text{A}$. This sudden increase may be attributed to the time of failure of the sample due to the corrosive action. During this period, the chloride ions reached the surface of the reinforcement through the concrete layer. Initially, a passive layer was formed by the concrete layer which protected the surface of the steel bars from getting corroded. When more chloride ions came into contact, they exceeded the threshold limit causing depassivation [59, 62]. Due to this ingress of chloride ions there was a rapid increase in current leading to the failure of the sample after 6.5 days agreeing with reports in literature [41]. Figure 13 shows the corrosion current Vs time for the concrete samples immersed in 3.5M NaCl solution cast with and without corrosion inhibitors. Also, during the initial stage of the accelerated corrosion, no cracks could be seen, but after 140 hours, thin hairline cracks could be slightly seen on the lateral surface of the concrete specimen. These hairline cracks would be the means of accessibility means through which the chloride ions would have reached the steel bars and started corrosion [63]. The increase in the amount of chloride ions is the reason for the sudden increase in corrosion current. Chloride ions, water and oxygen are responsible for the corrosion initiation. Reinforced concrete corrodes due to carbonation and chloride attack under humid

conditions. Generally, C4AF chemically known as Tetracalcium alumina ferrite present in cement provides satisfactory resistance against corrosion. It is present in high ferrite Portland cement than ordinary cement and Portland cement [64].

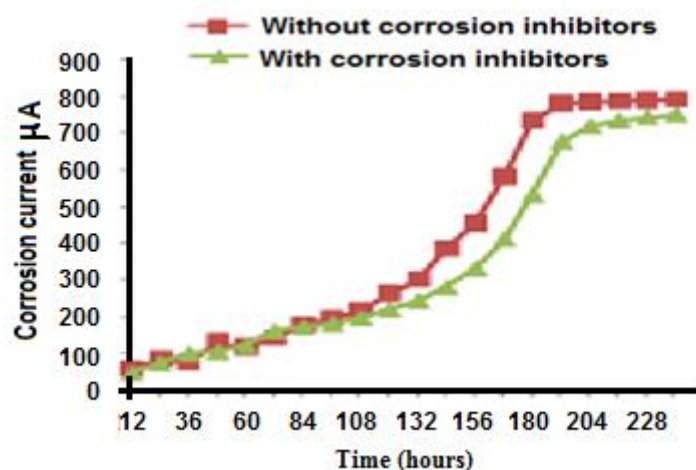


Fig. 13. Corrosion current Vs time for the concrete samples immersed in 3.5M NaCl solution

In case of the samples cast with corrosion inhibitors the same trend was observed. However, the corrosion current was quite higher as compared to the control samples without corrosion inhibitors. Corrosion current increased initially followed by a decrease in value after 48 hours. Then the value again rose to 134µA after 60 hours. Thereafter only insignificant changes were noticed. Then, an abrupt increase in the sample was found after 7 days and the corrosion current was found as 720µA. This is due to the increase in the penetrated chloride ions that caused corrosion. Similar to the samples without *Azadirachta indica* slurry, here too no signs of cracks were found during the initial stage (130 hours). After 150hours, relatively thin and small hairline cracks were seen feebly. These hairline cracks might have allowed chloride ions to pass through them reaching the steel bars eventually resulting in increase in corrosion current [63]. But the corrosion current of the inhibitor added specimens is lesser than the control specimens thus showing that there is an anticorrosive effect due to the inhibitor. It is clear that the increase in current is related to the reduction in time required to induce corrosion. The study showed that the inclusion of *Azadirachta indica* slurry had delayed the rate of corrosion process. This reveals that *Azadirachta indica* has the potential to reduce corrosive effects confirming the studies available in literature [24, 25].

3.9 Gravimetric weight loss test

The gravimetric weight loss test to determine the effect of adding corrosion inhibitor conducted on the fibre reinforced concrete samples cured for 28 days showed that the *Azadirachta indica* slurry can impede the corrosion activity. The rate of corrosion on the fibre reinforced concrete samples without corrosion inhibitors was 0.0654 ± 0.008 mm/year while the corrosion rate of the samples with corrosion inhibitors was 0.056 ± 0.011 mm/year exemplifying 14% decrease in corrosion rate. This is in agreement with the anti-corrosive activity reported in literature [41, 58]. Based on the results of the gravimetric test, the weight loss percent of concrete samples without corrosion inhibitors was found as $1.11 \pm 0.001\%$ as against the weight loss of samples with corrosion indicators found as $0.948 \pm 0.001\%$. The

weight loss is due to the chloride ion penetration into the steel bar. This mass loss percentage is normal and is comparable with the mass loss% of steel bars reinforced in ordinary Portland cement based concrete mix [65]. Corrosion rate is classified as mild corrosion (if corrosion rate < 0.025 mm/year), moderate corrosion ($0.025 - 0.13$ mm/year), high corrosion ($0.13 - 0.25$ mm/year) and severe corrosion (corrosion rate > 0.25 mm/year). In this study, the corrosion rate exceeded 0.025 mm/year but less than 0.13 mm/year and is classified as moderate corrosion[66]. Addition of *Azadirachta indica* slurry is proved to be an effective corrosion inhibitor.

The study thus shows that *Calotropis procera* cellulose fibres can be used with concrete mix. Addition of these fibres produced improvement in the structural strength of the concrete. In addition, it deferred crack formation and avoided the sudden failure of the specimen. *Azadirachta indica* leaf slurry can also be effectively used as corrosion inhibitor to safeguard the steel bars present as reinforcements in concrete. Thus it is recommended that these two green materials are compatible with concrete and can be used in making concrete samples as a sustainable measure. This study does not consider the influence of different fibre lengths on the structural properties of the concrete samples. Also, the effect of varying fibre content and corrosive inhibitor slurry on the structural properties of the concrete mix was not considered in this study. Chemical treatments on the cellulose fibres were proved to improve the structural integrity of the fibres [67]. Hence, this study can be explored further by considering these factors.

4. CONCLUSIONS

The study considered addition of *Calotropis procera* fibres and curing time as the important parameters for the concrete samples preparation. The following conclusions were derived from the study.

1. The inclusion of *Calotropis procera* fibres enhanced the compression, split tensile properties of the concrete samples. Cellulose content is responsible for the reinforcing efficacy of the fibres added concrete samples and these fibres can be used in concrete mix with sea water. The fibres resisted, deferred crack development and changed the direction of crack propagation during external loading.
2. The curing time was found to be directly related to the structural properties of the concrete samples. Optimum structural properties were obtained for both the conventional and fibre reinforced concrete samples when the curing time was 28 days. Longer the curing time, more is the durability of the concrete.
3. A considerable improvement of 19.5%, 15%, 10.3% and 14.5% in the compression, tensile, rebound hammer and ultrasonic pulse velocity values respectively was achieved for the fibre reinforced concrete samples compared to conventional concrete samples.
4. Accelerated corrosion test showed sudden increase in corrosion current after 140 hours due to the formation of tiny hairline cracks that caused penetration of chloride ions into the steel rebars. Corrosion current was found to be lesser in case of corrosion inhibitor added concrete mix than the sample without corrosion inhibitor.
5. Gravimetric weight loss test showed that *Azadirachta indica* leaf slurry is effective in reducing the corrosion rate about 14% due to its anti-corrosive property. The corrosion rate experienced in this present study was 0.025 mm / year exemplifying moderate corrosion. Weight loss of 1.11% and 0.948 % was found in concrete samples without and with corrosion inhibitors respectively.

Thus the study conducted has proved that the *Calotropis procera* fibres can be used as fillers in the fabrication of concrete samples and *Azadirachta indica* leaves can be used as corrosion inhibitors.

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