STUDIES ON THE EFFECT OF A TREE POD BASED NATURAL PLASTICIZER ON DURABILITY BEHAVIOR OF CONCRETE

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Abstract

This paper describes the results of a comparative study between concrete mixes containing natural plasticizer (NP) and chemical plasticizer (CP) as well as the control mix concerning durability performance. NP used in this investigation was prepared from the extract of rain tree pod and used in concrete. Tests on resistance to acid attack, water absorption, chloride ion penetration, water permeability, and sorptivity were carried out as per codal provisions. The chloride permeability of concrete was moderate with the addition of the developed natural plasticizer. The strength decrease of cubes subjected to acid attack with the addition of NP was lesser in comparison to the control mix. The results obtained show that the concrete mix with NP exhibited better performance than the control mix and worked equally efficiently as the mix with CP with respect to durability. Therefore, the NP prepared from the rain tree pod extract has the potential to be used as an alternative to CP for enhancing durability of concrete.

Keywords: Compressive Strength; Durability; Natural plasticizer; Rapid chloride permeability test (RCPT); Sorptivity; Water absorption; Workability.

1. INTRODUCTION

Sustainability in construction is of utmost importance to ensure reduction of carbon footprint in the environment. Concrete is the most extensively used material for construction purposes and a major contributing factor for CO_2 emissions. The increase in the worldwide production of cement from, 1.39 billion tons in 1995 to 4.4 billion tons in 2021 indicates the extent of growth in construction industry [1]. Globally, the emissions accounted to 36.1 Gt in 2019. Cement production alone contributed to more than 7 % CO_2 emissions in 2019 [2]. The CO_2 emissions can be indirectly reduced by reducing the cement consumption in concrete by improving concrete's mechanical properties as well as durability [3]. It has therefore become essential to develop innovative materials in construction, which would help in reducing the carbon footprint and contribute to sustainability. Majority of the chemical admixtures being used are known to have harmful effects on the environment apart from being costly [4]. Hence, novel materials are being developed to ensure production of sustainable concrete. Bio-polymeric admixtures are being researched upon as alternatives to chemical admixtures for improving the mechanical as well as durability properties of concrete [5-11]. In this investigation, extract from a locally available tree pod was considered as a low-cost and environmentally friendly alternative to chemical plasticizer for production of sustainable concrete. The results obtained on durability performances of cement concrete in the presence of a pod extract were reported.

2. MATERIALS

Ordinary Portland cement (OPC) of grade 53 conforming to IS: 12269 (2013) [12] was used for the current study. Table 1 shows the constituents of cement and Table 2 shows the physical properties of cement. Crushed aggregate (manufactured sand or m-sand) was utilized as fine aggregate and 20 mm down size aggregates were used as coarse aggregates. The properties

Table 1: Constituents of cement

CONSTITUENTS	QUANTITY (% BY MASS)
Calcium oxide (CaO)	61.69
Silica (SiO_2)	20.80
Alumina (Al_2O_3)	5.10
Iron oxide (Fe_2O_3)	4.23
Magnesium oxide (MgO)	1.61
Sulphur anhydrite (SO_3^{2-})	2.17
Insoluble residue	2.05
Loss on ignition	3.50
Chloride content (Cl^-)	0.02
Alkali content: Sodium oxide	0.34
Potassium oxide	0.38

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Table 2: Physical properties of cement

PROPERTY	RESULT
Normal consistency (%)	30.0
Specific gravity	3.0
Initial setting time (min)	270
Setting time, final (min)	415
Weight of residue left on IS 90-micron sieve (%)	3

Table 3: Properties of fine aggregates

PROPERTY	RESULT
Specific gravity	2.57
Water absorption (%)	2.00
Fineness modulus	3.0
Loose bulk density (kg/m^3)	1649

of fine and coarse aggregates are shown in Table 3 and Table 4, and were determined as per IS: 2386 (2002) (Part III) [13]. The grading of fine aggregates was determined as per IS: 2386 (Part 1) [14] and was found to be within the limits of IS: 383 (2016) (Table 9) [15] conforming to grading zone II. Potable water was utilized for mixing of concrete and all other experiments. The chemical plasticizer used was sulphonated naphthalene formaldehyde based plasticizer conforming to IS: 9103 (2018) [16]. The properties of chemical plasticizer (CP) are as shown in Table 5.

2.1 Preparation of natural plasticizer (NP)

The natural plasticizer used in this study was prepared by using the pods of the rain tree (Samanea saman) shown in Figure 1(a), which is usually grown as shade trees along roadsides. The pods of the tree are as shown in Figure 1(b). The pods were collected and deseeded; the hard fibers on its sides were removed and then crushed to form a uniform paste. The crushed paste was dissolved in water in the ratio 1:2 (paste to water ratio) and left soaked for 3 days. The solution was then filtered to remove the suspended solids. The filtered solution was fermented

Table 4: Properties of coarse aggregates

PROPERTY	RESULT
Specific gravity	2.73
Water absorption (%)	0.63
Fineness modulus	8.0
Loose bulk density (kg/m^3)	1513
Crushing value	24.2
Impact value (%)	24.5
Abrasion value (%)	28.0
Flakiness index (%)	27.2
Elongation index (%)	4.50

Table 5: Properties of SNF based chemical plasticizer

SNF BASED CP	RESULT
Appearance	Dark brown
Density kg/L	1.2
pH	6.7
Solid Content	42-44 %

using baker's yeast for a period of 12 days. The solution was then boiled to arrest any further fermentation. As the obtained solution was found to be acidic in nature, having a pH of less than four, it was brought to an alkaline range by adding lime to get the NP. The NP is a liquid and dark-brown in its appearance as shown in Figure 1(c). The test results of uniformity tests as per IS: 9103 (1999) [16] are shown in Table 6.

2.2 Characterization of natural plasticizer

Gas chromatography-mass spectrometry (GC-MS) is an instrumental technique used to separate chemical compounds (GC component) in a mixture and identify the compounds (MS component) based on their molecular weights. A GC-MS Shimadzu QP2020 system was used for the analysis of the

(a) (b) (c)

Figure 1: (a) Rain tree; (b) Rain tree pods; (c) Natural plasticizer

Table 6: Uniformity test results on natural plasticizer as per IS: 9103 (1999) [16]

NATURAL PLASTICIZER	RESULT
Dry material content (DMC), % by mass	8-10 %
Ash content (AC), % by mass	1.50
Chloride content , % by mass	0.062
pH	7.5-8.50
Relative density	1.06

Table 7: Compounds present in NP (GC-MS analysis)

RETENTION TIME	AREA %	HEIGHT %	COMPOUNDS
9.941	78.86	31.37	Glycerin
10.155	5.29	12.63	1,2,3-Propanetriol, 1-acetate
11.181	3.17	13.38	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-
11.931	0.32	1.61	1-Deoxy-2,4-0,O-methylene-d-xylitol
12.922	2.42	3.91	1-Deoxy-d-arabitol
22.242	7.63	27.46	5,10-Diethoxy-2,3,7, 8-tetrahydro-1H,6H-dipyrrolo 1,2-a:1', 2'-d]pyrazine
23.615	0.35	2.02	9,12-Octadecadienoic acid (Z,Z) methyl ester
23.675	0.71	3.96	11-Octadecanoic acid, methyl ester
24.070	1.24	3.67	Oleic acid
	100.00	100.00	

natural plasticizer. The GC column was a Rtx-5MS fused silica capillary with an internal diameter of 0.25 mm and a crossbond diphenyl dimethyl polysiloxane stationary phase with a film thickness of 0.25 m. Helium was used as the carrier gas, and its flow rate was 1 mL/min. Both the ion source and the input temperature were kept at 220°C. The GC oven temperature program was used as follows: 65°C initial temperature, hold for 5 minutes, increased at 10°C upto 300°C and hold for 6 minutes. The sample was dissolved in methanol and injected using a split injection method. The identification was done in scan mode. Chemical components were identified using scan mode by comparing their mass spectrum fragmentation patterns. Figure 2 shows the chromatogram of NP obtained from the GC-MS analysis and Table 7 shows the different compounds present in the NP.

3. METHODS

M25 design mix concrete with the proportions of concrete mix as follows: Cement = 375 kg/m^3; Fine aggregate = 659 kg/m^3; Coarse aggregate = 1190 kg/m^3 (1:1.17:3.17), and water-cement ratio = 0.45 was used for all experiments. The ideal dosages of CP and NP (percentage by weight of cement) for a design slump of 90 mm with the above mix proportions of concrete determined by trial and error were found to be 0.5 % (CP) and 1.25 % (NP) respectively. These dosages were used to cast all the specimens for strength and durability experiments. 28 days water cured concrete cube specimens of 150 × 150 × 150 mm size were cast for experiments on compressive strength, acid

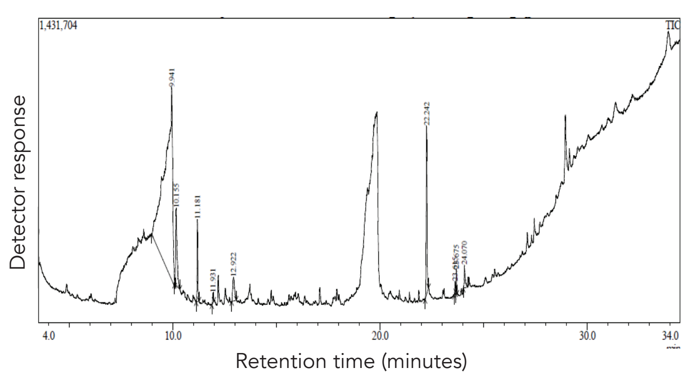


Figure 2: GC-MS chromatogram of NP

attack, water absorption and water permeability tests as per relevant codes. For experiments on rapid chloride permeability test (RCPT) and sorptivity tests, cylindrical specimens were cast with 150 mm diameter and 300 mm length, which were then cut into slices of 50 mm thickness as per the codal requirements. For all tests on strength and durability, three specimens were tested for each mix and the average values reported in the study.

3.1 Workability

Slump test was carried out on fresh concrete using the standard slump cone according to IS: 1199 (1959) [17] on control mix and mixes with CP and NP. The dosages required for achieving a slump of 90 mm were determined from the slump test.

3.2 Compressive strength

Compressive strength test was carried out according to using compressive testing machine of 3000 kN capacity on 150 × 150 × 150 mm cube specimens, as per IS: 516 (1959) [18] under loading rate of 140 kg/cm^2/min.

3.3 Durability

Durability performance was evaluated by conducting tests on resistance to acid attack, water absorption test, water permeability test, sorptivity test, rapid chloride permeability test on control mix and mixes with CP and NP.

3.3.1 *Resistance against acid attack*

In the acid resistance test, the strengths of concrete cubes under compression were compared with the cubes exposed to acid resistance test for all the three types of mixes viz., control mix, mix with chemical plasticizer and mix with natural plasticizer. 150 × 150 × 150 mm concrete cubes were tested according on ASTM C 267-01 [19]. The specimens were cured in water for a period of 28 days and allowed to dry for a day. Weights of the specimens were recorded after which the specimens were exposed to 5 % sulphuric acid solution with a pH of 1.5 for 28, 56 and 90 days. The pH was checked at regular intervals and care was taken to maintain the pH of acid solution at 1.5. After the period of immersion in acid solution, the cube specimens were taken out and washed in running water. The specimens were kept in atmosphere for 2 days to achieve constant weight. They were then weighed and tested for compressive strength. The acid resistance of concrete for all three types of mixes was found by the percentages of weight loss and loss in compressive strength upon immersion in acid solution.

3.3.2 *Water absorption test*

The test on water absorption was carried out as per ASTM C 642-2013 [20] to determine the water tightness of concrete which is an important parameter considering durability of any structure. The concrete cubes were dried in the oven at 100°C for at least 24 hours until the difference between two successive values was less than 0.5 % of the lowest value. The weights of dry specimens were recorded. The specimens were then immersed in water at around 23±2°C for not less than 48 hours an until the two successive values of mass of surface-dried sample at intervals of 24 hours showed a mass increment of less than 0.5 % of the larger value. The specimens were immersed in water such that a level of 50 mm water was maintained above the top surface of specimen. The weights of wet specimens after surface drying were recorded. The difference in weights of dry and wet specimens gave the extent of absorption.

3.3.3 *Water permeability test*

The water permeability of concrete specimens was measured by carrying out permeability test according to German standard DIN: 1048 Part V-1991 [21]. Concrete cube specimens of size 150 × 150 × 150 mm at an age of 28 days were subjected to a water pressure of 5 kg/cm^2 (5 bar) on one surface of the specimen for a duration of 72 hours. The specimens were then taken out and split into two using a compression-testing machine. The maximum depths of water penetration for all the specimens were measured in mm.

3.3.4 *Sorptivity test*

Sorptivity test measures the rate of absorption of water in the concrete specimens. This test was carried out according to ASTM C 1585-2013 [22]. The test specimens were dried in an oven at 100°C for a duration of 72 hours after which they were stored at room temperature for a period of 24 hours. The side surfaces of the specimens were coated with an epoxy coating. The weights were noted and the specimens were kept in a tray with 5 mm water filled in it such that only the bottom surfaces of the specimens had an access to water. The specimen weights were also noted at regular intervals after the excess water was removed from the wet surface. The readings were taken for a period of 30 minutes at every 2-minute intervals. The absorption (I) was calculated as the difference in weight divided by the product of specimen's cross-sectional area and water density. The initial rate of water absorption (mm/sec$^{0.5}$) was calculated as the slope of line, which best fits the graph plotted of absorption (I) versus square root of time ($\sqrt{t}$) using data points from one minute to six hours. The secondary rate of water absorption was determined as the slope of line which best fits the graph of absorption (I) versus square root of time ($\sqrt{t}$) using data points between one day and seven days. The slopes were determined using least-squares linear regression.

3.3.5 *Rapid chloride permeability test*

The rapid chloride permeability test (RCPT) is an important test and the most commonly adopted test for measuring concrete's durability. It measures the level of chloride ion penetrability of concrete. RCPT was conducted as per ASTM C1202-1997 [23] for all concrete mixes after the specimens were cured for 28 days. The RCPT apparatus consists of two cells at either ends of the specimen as shown in Figure 3. One cell was filled with 3.0 % NaCl and the other with 0.3 N NaOH solutions. The NaCl electrode was connected to negative terminal of the power supply and NaOH was connected to the positive terminal of the power supply. The cells were connected to a power source and potential difference of 60 volts is maintained between them. The chlorides from the negative cathodic end moves towards the positive anodic end through concrete.

The sides of the specimens were coated with epoxy and cured until they were no longer sticky to touch. The specimens were then subjected to conditioning using vacuum saturation method. The specimens were placed between the negative

Figure 3: RCPT apparatus

and positive cells and a direct current was applied to maintain a potential difference of 60 V. The amount of current passing through is recorded every 30 minutes for a period of 6 hours and converted to a charge passed in terms of Coulombs by integrating the area underneath the graph of current versus v/s time. The electrical conductance of concrete was measured by the total charge passed during the testing period. The total current passed is given by, $Q = 900 \times [I_0 + 2I_{30} + 2I_{60} + 2I_{90} + 2I_{120} + 2I_{150} + 2I_{180} + 2I_{210} + 2I_{240} + 2I_{270} + 2I_{300} + 2I_{330} + I_{360}]$, where Q = charge passed, Coulombs; I_0 = current measured at an instant after application of voltage, amperes; I_t = current measured at t minutes after application of voltage, amperes. As per the classification of ASTM C-1202-97 [23], the chloride-ion permeability based on charge passed ranges from negligible to high. The range for moderate chloride permeability is 2000-4000 Coulombs.

4. RESULTS AND DISCUSSION

4.1 Workability

Table 8 shows the slump values for a water cement ratio of 0.45. The dosage of plasticizer by trial and error for a design slump of 90 mm was found to be 0.5 % for CP and 1.25 % for NP respectively. The control mix (without any plasticizer) exhibited a slump of only 15 mm. However, with further trials it was determined that a water cement ratio of 0.49 was required for the control mix to achieve a slump of 90 mm. The CP showed the required slump at a comparatively lower dosage than NP displaying good cement particle dispersion, thereby releasing the entrapped water for fluidifying the mix. The mix with NP produced the same slump; however, at a higher dosage. To achieve a slump of 90 mm, a *w/c* ratio of 0.49 was required for control mix without any plasticizer. The same slump of 90 mm was obtained in the mix with NP (1.25 % by weight of cement) at a *w/c* ratio of 0.45 keeping the cement content constant. Therefore, by using NP, a reduction of 8 % in the water content was achieved. In general, there is an improvement in the workability indicating the usefulness of pod based plasticizer to enhance the flowability of concrete. Improvement of workability may be related to the glycerin content present in the NP (refer Table 7).

Table 8: Slump test results (*w/c* = 0.45)

SAMPLE TYPE	DOSAGE OF PLASTICIZER, % BY WEIGHT OF CEMENT	SLUMP, mm
Control mix	--	15
Mix with CP	0.50	90
Mix with NP	1.25	90

Table 9: Comparison of compressive strength results with control as reference

SAMPLE TYPE	PERCENTAGE INCREASE IN COMPRESSIVE STRENGTH
Mix with CP	1.9 %
Mix with NP	2.5 %

4.2 Compressive strength

Table 9 shows the percentage increase in compressive strengths of mixes with CP and NP, when compared to the control mix without any plasticizer. It can be seen that there is an increase in the strength of concrete on addition of natural plasticizer. The 28-days compressive strength of the mix with NP was also slightly higher than the mix with CP. The obtained strengths can be attributed to the dense formation of CSH gel and CH as seen in the SEM images of control mix and mix with NP as shown in Figures 7a and 8a.

4.3 Durability

Durability performance was evaluated by conducting tests on resistance to acid attack, water absorption test, water permeability test, sorptivity test, rapid chloride permeability test on control mix and mixes with CP and NP.

4.3.1 *Resistance against acid attack*

By visual observation, it was seen that there was surface deterioration of the cube specimens with the aggregates being exposed. Dissolution of the cement paste occurs in concrete due to the acid attack leaving behind a weak concrete mass. Hydrogen ions diffuse into the CSH gel after dissolution of $CaOH_2$ and get leached out. The CSH gel disintegrates at a low pH leading to the loss of cementitious content as observed from the SEM images in Figures 7b and 8b for control mix and mix with NP. Figures 4 and 5 show the percentages of loss in

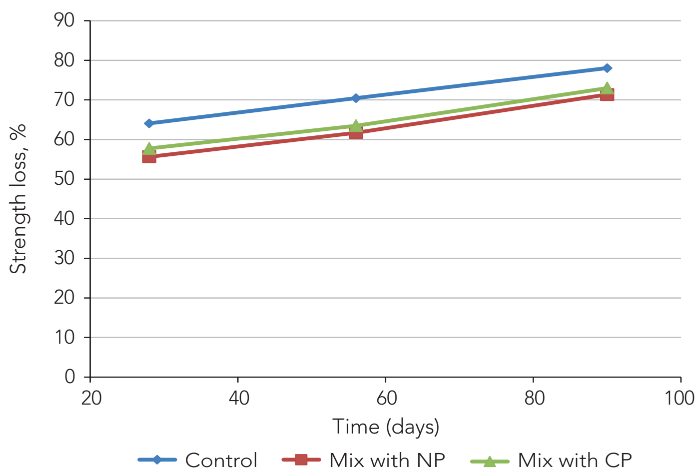


Figure 4: % loss in compressive strength of concrete cube samples after immersion in acid solution

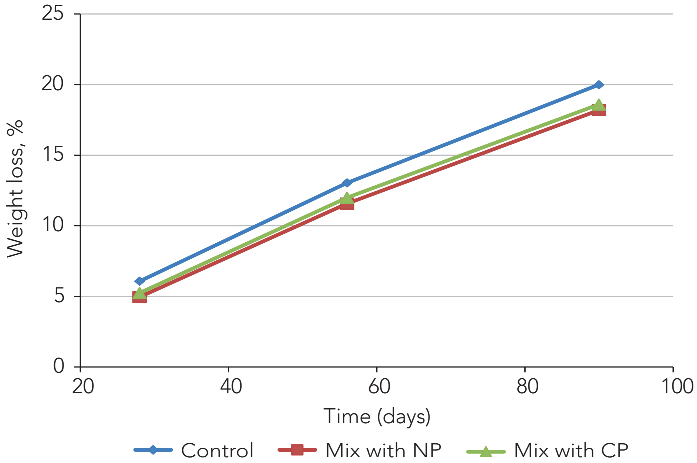


Figure 5: % loss in weight of concrete cube samples after immersion in acid solution

compressive strengths and weights of different mixes after immersion in acid for duration of 28, 56, and 90 days. Figure 6 shows the concrete specimens with aggregates exposed after 28 days of immersion in acids solution.

Figure 7 and Figure 8 show the scanning electron microscope (SEM) images of control mix and mix with NP before and after acid attack. It was observed that CSH gel and CH are seen scattered in the acid attacked sample whereas in the samples without immersion in acid, dense formation of CSH gel, ettringite and CH was observed.

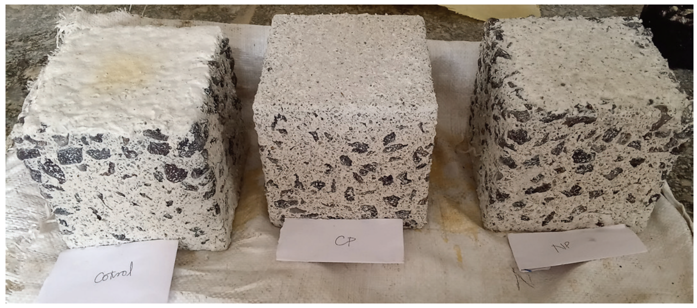
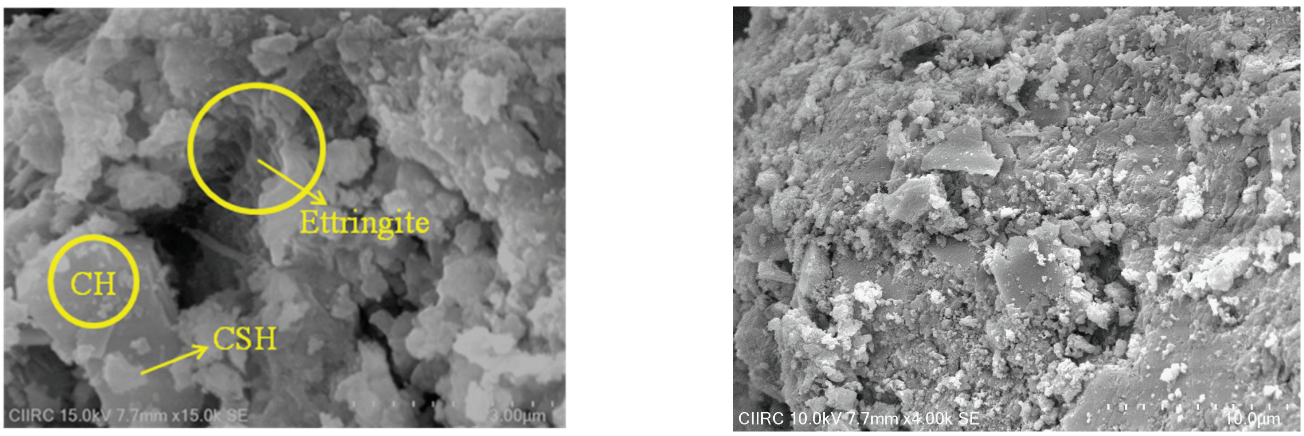


Figure 6: Concrete cube specimens after 28 days immersion in acid solution

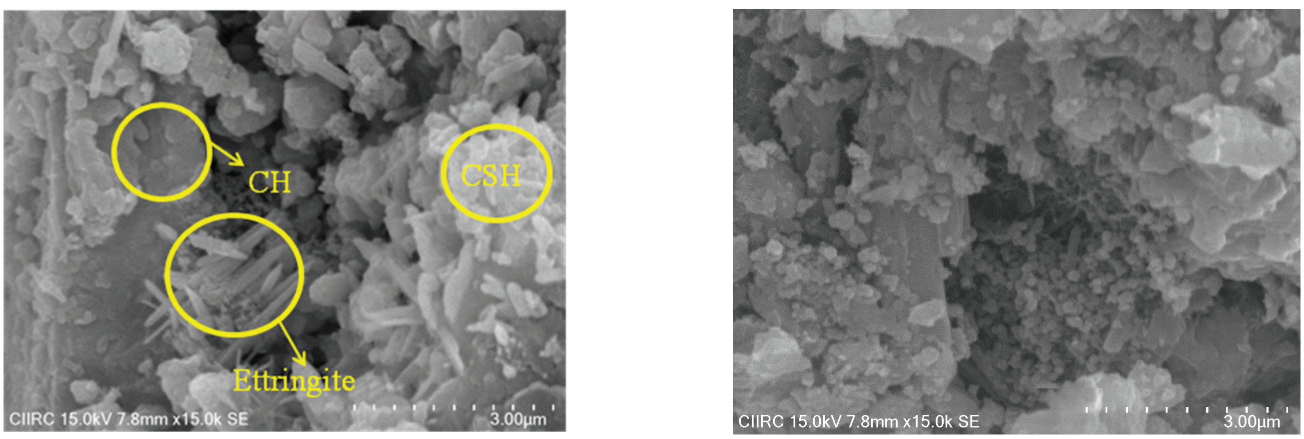
4.3.2 *Water absorption test*

The results obtained from water absorption test are as shown in Table 10. The absorption of water was the highest for control mix followed by mix with CP and NP. It was seen that the absorption of water by mixes with CP and NP were 3.5 % and 8 % lesser than the control mix. The reduced water absorption in mix with NP may be due to the reduced porosity.



(a) (b)

Figure 7: SEM images of control mix before and after acid attack



(a) (b)

Figure 8: SEM images of mix with NP before and after acid attack

Table 10: Water absorption capacities of concrete specimens

SAMPLE TYPE	WATER ABSORPTION, %
Control mix	3.90
Mix with CP	3.77
Mix with NP	3.59

4.3.3 *Water permeability test*

It can be clearly observed from the results shown in Table 11 that the maximum water penetration depth for concrete mixed with natural plasticizer was minimum when compared to control mix and mix with chemical plasticizer. This may be due to the reduced porosity in the mix with NP. Figure 9 shows the concrete cube specimens after splitting under the application of compressive load. Therefore, addition of natural plasticizer offers more resistance to water penetration and can be used in places, which are continuously exposed to water.

4.3.4 *Sorptivity test*

Figure 10 shows the variation of absorption with square root of time of concrete specimens. The results of initial rates of absorption of all the three specimens as shown in Table 12 show that the control mix has the highest value while there is not much difference between the values of mixes with CP and NP, although the value of mix with NP was lesser than that of the mix with CP. The secondary rate of water absorption however could not be determined, as the correlation coefficient for the data points was less than 0.98 and showed a non-linear between 1 day and 7 days.

Table 13 shows the results of the charge passed for different concrete specimens. Control mix showed the highest chloride penetration value when compared to the mixes with CP and NP. All the three mixes fall in the moderately permeable category of 2000-4000 Coulombs. However, it was observed that the mixes with CP and NP offered lesser permeability to chloride-ions than that of control concrete by 12 % and 15 % respectively. The reduction in chloride ion permeability may possibly be due to the decreased porosity in the mixes with CP and NP.

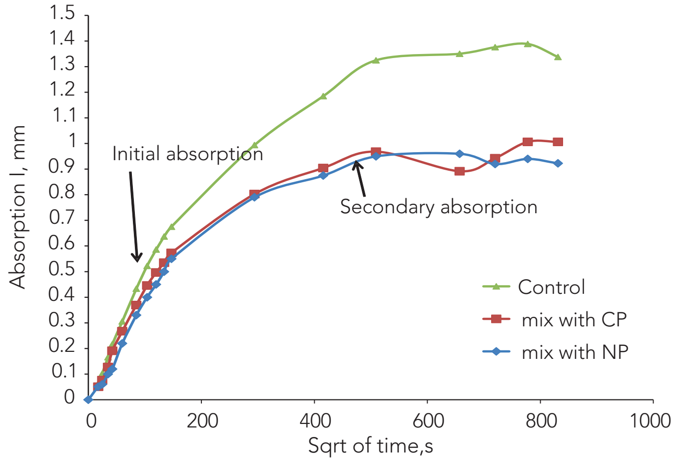


Figure 10: Absorption versus square root of time graph

Table 11: Water permeability depths of concrete specimens

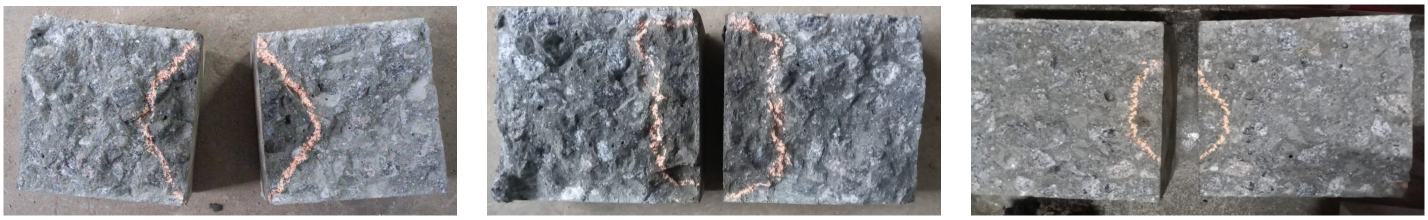
SAMPLE TYPE	WATER PENETRATION DEPTH, mm
Control mix	4.5
Mix with CP	4.3
Mix with NP	2.3

Table 12: Initial rate of absorption values for concrete specimens

SAMPLE TYPE	INITIAL RATE OF WATER ABSORPTION
Control mix	4.8×10^{-3} mm/√s
Mix with CP	4.1×10^{-3} mm/√s
Mix with NP	4.0×10^{-3} mm/√s

Table 13: Charge passed for concrete specimens

SAMPLE TYPE	CHARGE PASSED (COULOMBS)
Control mix	3378
Mix with CP	2976
Mix with NP	2874



(a) (b) (c)

Figure 9: Water permeability depths of test specimens after splitting for control mix, mix with CP and mix with NP

4.4 GC-MS analysis of NP

The chromatogram of the fermented NP sample obtained by GC-MS analysis is displayed in Figure 2 and the compounds present in the NP are shown in Table 7. According to the GC-MS results, glycerin (glycerol) is the major component present, along with esters of long-chain fatty acids like oleic acid, which help to increase the fluidity of concrete. By lowering the intermolecular friction between the particles and assisting in lubrication, the workability of concrete is improved. Glycerin, also referred to as glycerol is a polyol, an alcohol containing three hydroxyl groups that are responsible for the water-soluble nature and hygroscopic nature of glycerin. Glycerin is used as a humectant, plasticizer, emollient and solvent for flavours and food colours. It is also used in the manufacture of lubricants, cosmetics, pharmaceuticals and personal care products [24, 25]. Glycerine has been shown to improve the compressive strength of mortar and concrete [26]. Additionally, glycerol is a useful cement additive, which acts as a grinding aid in cement by decreasing the attractive forces between cement particles that cause agglomeration [27]. Therefore, the good workability and compressive strength attained with the addition of NP may be attributed to the presence of glycerin in the developed NP.

5. CONCLUSIONS

A study was undertaken to evaluate a natural plasticizer prepared from a tree pod extract as a replacement to chemical plasticizer in cement concrete. In this study, the workability, compressive strength and durability performance of concrete were evaluated by carrying out experiments of slump cone test, compressive strength test and tests on resistance to acid attack, water absorption, RCPT, water permeability and sorptivity. The experiments were carried out on concrete mixes with the addition of natural plasticizer as well as chemical plasticizer and concrete mix without the addition of any plasticizer (control mix) for comparison. Following conclusions are drawn from this study.

1. There is an increase in slump value of fresh concrete by 500 % due to the addition of CP (0.5 %) and NP (1.25 %) respectively for a water-cement ratio of 0.45.
2. There was 8 % reduction in the water content due to the addition of NP when compared to the control mix, to achieve a slump of 90 mm.
3. The compressive strength did not get affected by the use of NP. The average 28 day compressive strength in the mix with NP was 2.5 % higher than the control mix and 0.6 % higher than the mix with CP.
4. Glycerin was found to be the major component in the prepared NP, which could be responsible for the enhanced workability and compressive strength of the mix with the NP.
5. The residual compressive strength of concrete specimens (after 28 days exposure to acid solution) with natural plasticizer was 21 % more than the control mix and 2.4 % more compared to the mix with CP, indicating better strength retention of the mix with NP in aggressive conditions of acid attack.
6. The water absorption of mix with NP was found to be lesser than the control mix by 7.9 % and lesser than the mix with CP by 4.8 %. The NP exhibits decreased water absorption indicating lower level of porosity in the concrete cubes. In addition, the water penetration depth (maximum) was found to be the least for mix with NP, and therefore can be beneficially used in severe environment conditions.
7. The chloride ion penetration of the concrete specimens due to the addition of natural plasticizer was found to be lesser than that of the control mix by 14.9 % and lesser than the chloride ion penetration of the mix with CP by 3.4 %.
8. The durability performance of concrete mix containing the natural plasticizer was found to be better than the control mix and mix containing chemical plasticizer in the study carried out. The results of this study are encouraging and prove beneficial for use of the natural plasticizer prepared from the extract of rain tree pods as an efficient, low-cost and environmentally friendly alternative to chemical plasticizers.

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