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Enhancement of crack healing efficiency and performance of SAP in biocrete

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ABSTRACT. Concrete usage in Construction becomes more common in this speedy world. Despite its benefits, concrete often exhibits crack which appear due to stresses. Larger cracks cause Structural integrity problems and smaller cracks may result in durability issues. A novel environmental friendly strategy to restore or remediate cracks formed in the structures is bio-mineralization of calcium carbonate using microbes such as Bacillus Subtilis (used in this study), as manual repair and maintenance is costly. In this Paper, an idea of using Super Absorbent Polymer in Bacterial Concrete was analysed which increases the strength and durability properties of concrete and also which acts as a protection to bacteria, where Self-Healing nature is viewed. In the span of 90 days, the results of Bacterial concrete cured under normal water providing nutrients inside with SAP shows healing up to 74 % and without SAP displays 49 % and when it is cured under nutrient medium, Bacterial Concrete having SAP displays healing up to 66 %, whereas without SAP it displays 57.4% of healing. During the observation it is discernible that the crack width ranging from 0.10 mm near 0.45 mm show better self-healing capacity. XRD analysis displays the presence of Calcium carbonate precipitation in cracks.

1. Introduction and Literature Study

Formation of Cracks is a habitual phenomenon that can be observed in Concrete Structures. These Cracks get developed due to various reasons such as shrinkage, loading stress, creep or may be due to thermal expansion. Few cracks which can allow the ingress of oxygen, chlorides, water and other aggressive substances present in air which may cause premature corrosion of steel reinforcement and decreases the structures durability. Concrete cracks can be there at areas which are difficult to access and since manual repairs are exorbitant, precipitation of $CaCO_3$ induced by bacteria, was executed at Delft University of Technology for the development of self-healing concrete for the new structures as well as for mortar and liquid-based systems for repair of prevailing concrete structures. Due to the metabolism reaction, urease is produced by bacteria, whereas urease again catalyses urea and produces CO_2 and NH_3, resulting in ions of Calcium and Carbonate that precipitate as $CaCO_3$ which seals the cracks [1]. Sporosarcina pasteurii was the bacteria incorporated to study on the strength parameters at different cell concentrations with the mixing water which showed that a 33% increase in 28 days' compressive strength of cement mortar was achieved with the addition of about one optical density (1 OD) of bacterial cells with mixing water [2]. In the strength enhancement study of soil bacteria, when 28 days' compressive strength is examined in bacterial cement mortar it is observed that strength increased up to 18 % when the mortar is cured with water carrying nutrients and only 12% increase is seen in specimens cured in normal water [3]. In this study, SAP is added to check the efficiency of healing in cracks, few studies discussed on the strength aspects of SAP dosage from 0.1% to 0.7% at an interval of 0.2%. It is observed that 0.1% gives higher strength of concrete [5]. It is necessary to study the Durability of concrete, so conventional and bacterial specimens are immersed in 5% H_2SO_4 to gain knowledge on the strength and weight loss of both. Bacterial specimens were found to have good resistance to adverse environment when compared to normal specimens [8]. The water absorption values of specimens mixed with bacterial cells at all OD concentrations (0.5, 1, and 1.5) are lower than those of control specimens proving that the bacterial embedded specimens have less water absorbency than conventional specimens [2]. Self-healing of cracks by bacteria which is considered to be an environmental friendly technique is experimented to observe the efficiency of its healing and the width of crack that could be healed in certain days. Healing ratio in the specimens with bio microcapsules was higher (48%–80%) than in those without bacteria (18%–50%). The maximum crack width healed in the specimens of the bacteria seems to be 4 times higher than the non-bacteria specimens [6]. When cement-based materials specimens were pre-cracked at the age of 7, 14, 28 and 60 days to study the repair capacity influenced by cracking time, the width of cracks for the study were between 0.1 and 1.0 mm. Bacteria incorporated specimens exhibited excellent repairing capacity for cracks 0.4 mm and 0.48 mm respectively [7,8]. Various Bacteria were examined to identify the bacteria genus which have maximum self- healing ability. Three bacterial strains Bacillus pasteurii, Bacillus sphaericus and Bacillus flexus were isolated and compared for its efficiency of healing. MICP was quantified by X-Ray Diffraction (XRD) analysis and visualized by Scanning Electron Microscopy (SEM). This investigation demonstrates that Bacillus flexus have better potential of calcite production than the other two bacteria [4]. XRD images were studied to obtain knowledge of Calcite precipitated peak. The initial peaks for calcium carbonate is obtained at Bragg's angle (2θ) in the range between 26° and 30°. Higher the peak suggests the higher amount of precipitation of calcite. From the results of XRD, it could be confirmed that there is presence of $CaCO_3$ in the cracks produced Microbially [9-11]. In this present study, Initially the water absorbency of SAP is found out. The serial diluted Bacillus subtilis bacterial solution is utilized along with Super-Absorbent Polymer to examine the efficiency of crack healing ratio. Here, the healing is studied for a period of 90 days. Along with the efficiency of healing, strength and durability studies are also undergone for the utilization of concrete in mere future.

2. Materials utilized

2.1 Bacterial Strain

Bacterial strains of Bacillus genus bacteria were found to flourish in an environment which is highly-alkaline. Bacillus Subtilis (MTCC NO:121) is a gram positive bacteria, which has thick cell wall which helps them to survive and grow in adverse environmental conditions. The incubation period is 48 hours and the sub-culturing period is 30 days. The bacterial strains were acquired from MTCC (Microbial Type culture collection and Gene Bank) in Chandigarh. Initially, the bacteria were received in ampoules and it was in a freeze- dried condition. Bacteria is first cultured in solid media (agar) are then transferred to nutrient broth (liquid media) which is sterile and kept in shaking incubator (to ensure uniform growth) for about 48 hrs. From this we would get the Mother culture of the bacteria (as in figure 1). Later, it is diluted by Serial Dilution Technique, so that it can be utilized during the concrete mix having a cell concentration of 10^5 cells/ ml. Concentration of cells is measured by Haemocytometer and optical density is found by spectrophotometer analysis before adding bacteria to cement composites.

Figure 1 Mother culture of Bacillus Bacteria

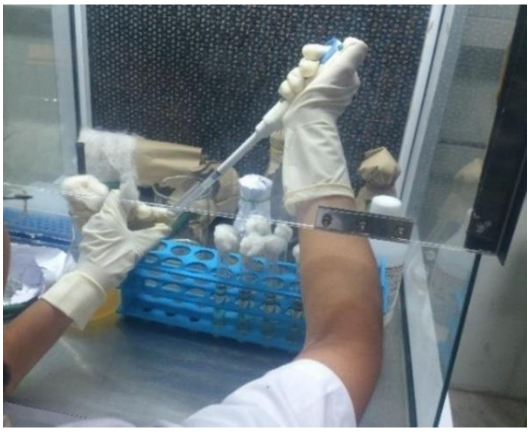

Figure 2 Serial Dilution Technique done to obtain a diluted form to be used in concrete

2.2 Cement Sand and Aggregates

Table 1 The necessary materials required for the study

Material	Grade/Zone/Size	Reference code
Cement	OPC 53	IS: 12269-1987
Fine Aggregate	Zone II	IS: 383- 1970
Coarse Aggregate	20 mm	IS: 383- 1970

Concrete mix proportion designed as per IS 10262-2009 for the grade of M25.

2.3 Super Absorbent Polymer

Super Absorbent Polymer($(C_3H_3NaO_2)_n$ handled in this project is Sodium Polyacrylate white in colour whose particle size is 85-50 mesh, which is also known as Super-Slurper, acrylic sodium salt polymer and simply SAP, is a super-absorbent chemical polymer (i.e. it can absorb water many times its own weight). SAP is added at 0 % 0.1 % 0.3 % & 0.5 % of weight of cement in this study.

Figure 3 SAP after the addition of Water

2.3.1 Water absorbency of SAP

In a 500 ml cylindrical measuring jar, 1 gram of SAP is taken initially and water is added in ml. Since the SAP can absorb water, it swells. When the SAP swells its height in the jar is measured. Since the

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diameter of the measuring jar is known, its volume is calculated accordingly. At certain point (Here above 86 ml of water) it is observed that SAP can't absorb further water, which represents its optimum absorbency. The maximum water absorbency of SAP has been found as 86 g of water/gram of SAP. The percentage increase in volume from initial amount of water added to the final amount of water is represented in the graph below.

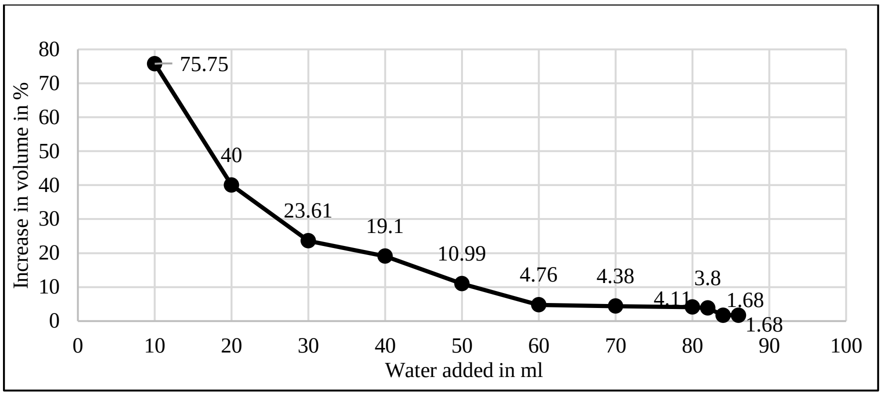

Figure 4 Indicating the decrease in water absorption of SAP after optimum level

2.3.2 Discussion on Water Absorbency of SAP

Per gram of SAP cannot absorb further water after 86 g and there is no swell observed. Figure 4 shows the gradual increase in volume when water is added to the SAP. The maximum % increase in volume of SAP when water is added about 75.75 % at the initial addition and it decreases up to 1.68 % at the optimum point. At this particular level, the water gets retained on the top which indicates the optimum absorbency of this SAP. Hence, During the time of casting the water absorbency of SAP is also considered for getting a proper mix of concrete.

2.4 Addition of Nutrients

Nutrients which would be required for effective bio-precipitation, including deposition medium (calcium chloride and urea) and food for bacteria (yeast extract, YE of 0.35%) were also incorporated. The dosage of calcium chloride and urea are 50 g/l and 20 g/l of water respectively [1,6]. Addition of nutrients may increase the strength of concrete and decrease the water absorption, which is an added advantage for the SAP embedded Bacterial concrete.

3. Experiments carried for study

Experiments are conducted with the support of Bacterial strain, SAP and other materials mentioned above for studying the strength, durability and self-healing properties of Biocrete.

3.1 Testing of Bacterial and Normal Specimens

M_{25} grade of Concrete Cubes and Cylinders are cast to undergo study on Compressive strength, water absorption, acid factor and self-healing efficiency on Bacterial Specimens containing SAP and without SAP as well as normal specimens. The obtained results are compared with the normal specimens. Prisms of Bacterial Specimens with SAP and without SAP are cast for studying the self- healing effectiveness.

3.1.1 Compressive Strength and Water Absorption Test

For a M25 grade of Concrete mix, cube mould of size 10 cm x 10 cm x 10 cm for Bacterial Specimens with SAP and without SAP as well as normal specimens are cast and tested for 7, 14 and 28 days as per IS 516 -(1959). The Water absorption test is conducted for Bacterial Specimens with SAP and normal specimens as per ASTM C 1585-13.

3.1.2 Acid Immersion Test

A Small Experimental study is done by immersing the Bacterial Specimens Containing SAP and normal specimens in 5 % solutions of Sulphuric Acid as a part of a Durability Tests as per ASTM C 666 - 1997. Percentage weight loss and strength loss is calculated as follows; $W = (W_i – W_r) / W_i * 100$ %, where W_i is the initial weight and W_r is the final weight at refined age. $S = (S_i – S_r) / S_i * 100$ %, where S_i is the reference compressive strength and S_r is the final compressive strength at refined age.

In the current study, the "Acid Factors" are calculated. The relative strengths of the values are taken always with respect to the 28 days' value. The formulae used to calculate the Acid Factors are given below. Acid Durability Factor (ADF) of a specimen = $S_r * N / M$; where, S_r = relative strength of concrete at N days (%), N = no. of days of immersion in acid at which the durability study factor is required, M = total no. of days of immersion when it is to be terminated. The "Acid Attack Factor" (AAF) is measured as follows; AAF = (Loss in all eight corners of each of 2 cubes in mm) / 4. The weight loss and strength loss percentage are studied at 15, 30, 45 and 60 days of immersion in H_2SO_4. The Acid Factors ADF and AAF denotes the capability of bacterial and normal specimens in acid [8].

3.2 Self-Healing Efficiency

The filling efficiency of the cracks in prisms can be evaluated by the ratio of healing (r), which is the ratio between healed crack area and initial crack area.

3.2.1. Creation of cracks and incubation conditions of Specimens

Bacterial Prism specimens with and without SAP are subjected to one-point loading, 28 days after casting, in Universal Testing Machine. The un-reinforced prisms take load between 4 and 10 KN. The loading is stopped at a point when a crack width of 0.1 to 1 mm is observed. The crack width is measured using a microscope. The prism specimens which are cracked were subjected to two conditions of incubation: 1) Firstly, immersing in normal water providing nutrients inside the prism during the time of casting; 2) Immersion in the deposition medium containing calcium chloride and urea.

3.2.2 Evaluation of the self-healing ratio of specimens

Self-healing by the assistance of microorganisms is predominantly due to the precipitation of $CaCO_3$ (calcium carbonate) by the microbes. Measurement of healed area in the crack indicates the healing efficiency. The crack filling efficiency can be assessed by the ratio of healing (r). The initial images of the cracks (0 days) in the specimens were taken immediately after the loading. During the incubation period, the specimens were observed every 15 days to check the amount of healing in the cracked portion. From XRD Analysis, the precipitation of calcium carbonate can be understood.

4. Results and Discussions

4.1 Cubes tested for Compressive Strength

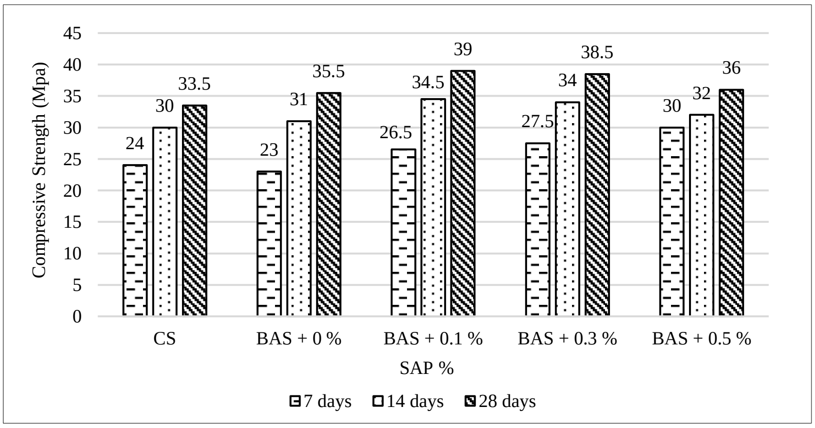

Figure 5 Compressive Strength of M_{25} grade concrete in Mpa

From the Figure 5, it is clearly evident that Bacterial Specimens Containing 0.1% SAP gives Maximum Strength of 39 N/mm^2 whereas the Normal Specimen gives 33.5 N/mm^2 at the end of 28 Days. Therefore, Bacterial specimens containing SAP shows an increase of 14.1 % strength compared to normal specimens. (CS- Control specimen, BAS- Bacterial specimen, 0 to 0.5% - SAP %)

4.2 Water Absorption Test - Control Specimen and Bacterial Specimen with SAP
The Water Absorption of Control Specimen is observed as 4.82 %. The Water Absorption of Bacterial Specimens containing various percentage of SAP is represented in the Figure 6 underneath.

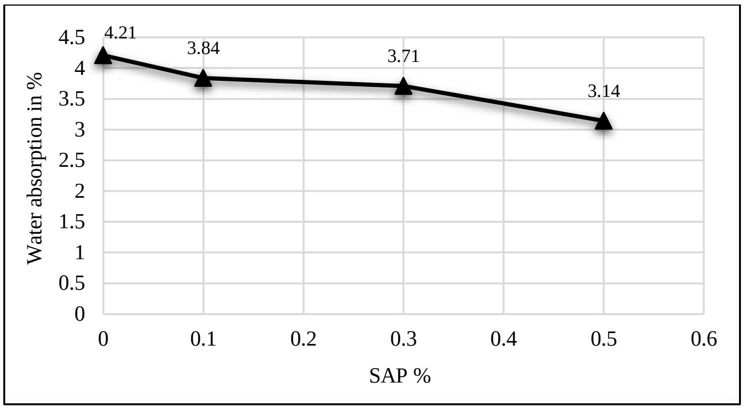

Figure 6 Variation in Water Absorption for various percentage of SAP added

The water absorption of Bacterial Subtilis Specimens comprising various percentage of SAP gets decreased from 4.21 % to 3.14 %. So, addition of 0.5% SAP reduces water absorption percentage nearly 34.8 % when compared to the conventional specimens. This implies SAP added bacterial specimens shows a healthier property when compared to normal specimens. So, addition of SAP along with bacterial specimens decreases the water absorbing property of concrete which will be a beneficial application in the required areas of construction. But, sometimes higher usage of SAP might reduce the strength of concrete [5].

4.3 Acid Immersion Test

4.3.1 Percentage Weight loss and Strength loss

Table 2 Representation of % Weight loss and % Strength loss of Normal and Bacterial Specimen

Days of Immersion in Acid	Normal Specimen	Bacterial Specimen	Normal Specimen	Bacterial Specimen
	% Weight loss		% Strength loss	
15 Days	2.06	1.97	11.94	10.26
30 Days	2.36	2.15	19.40	17.94
45 Days	3.19	2.29	25.37	23.07
60 Days	3.56	3.50	31.34	28.21

From Table 2 it is visible that Bacterial Specimens with SAP has an ability to withstand the weight loss and strength loss at Ages of Immersion in Sulphuric Acid when compared to the Normal Specimen.

4.3.2 Acid Durability Factor and Acid Attack Factor

Table 3 Representation of ADF and AAF of Normal and Bacterial Specimen

Days of Immersion in Acid	Normal Specimen			Bacterial Specimen		
	ADF = S_r* N/M	loss in 8 corners (mm)	AAF	ADF = S_r* N/M	loss in 8 corners (mm)	AAF
15 Days	22.02	16	0.50	22.44	15	0.47
30 Days	40.30	20	0.63	41.03	18	0.56
45 Days	55.97	28	0.88	57.70	24	0.75
60 Days	68.66	32	1.00	71.79	30	0.94

Table 3 gives a clear view of the Acid Attack Factor of Bacterial Specimens with SAP at Ages of Immersion in Sulphuric Acid and the Normal Specimen. From this we can infer that the Bacterial Specimens with SAP show slightly a better result to that of Normal Specimen.

4.4 Self –healing efficiency in Prisms

The evolution of healing of crack can be spotted from the pictures of cracks taken at regular time intervals such as 15, 30, 45, 60 up to 90 days. From the pictures it was clearly noticed that the crack area gets decreasing gradually as the time passes.

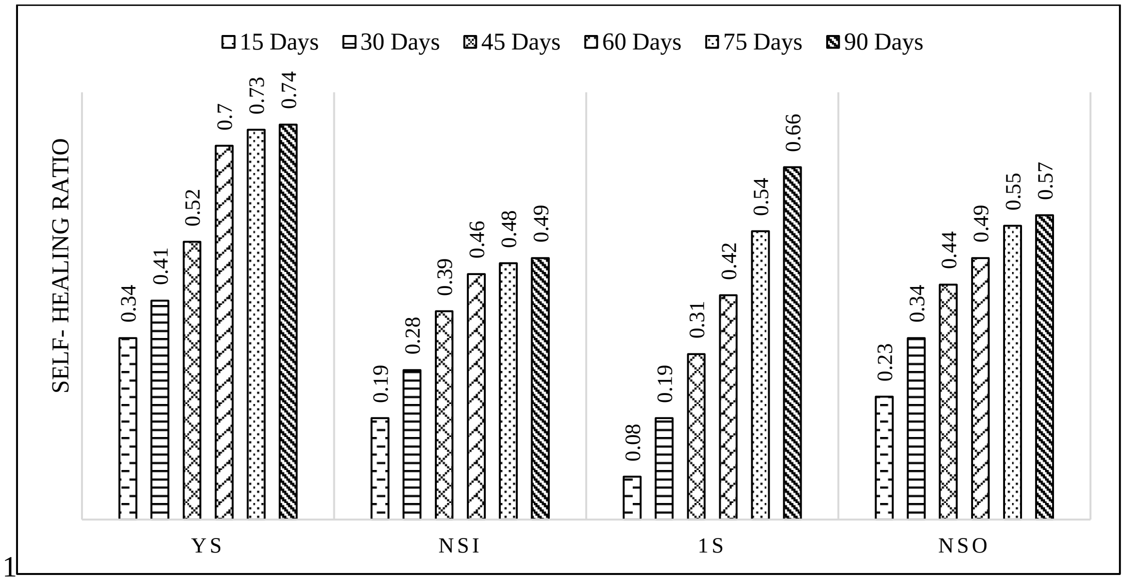

Figure 7 represents the self-healing ratio observed for Type I and Type II curing

YS – BAS with SAP (Type I curing)
NSI – BAS without SAP (Type I curing)
1S – BAS with SAP (Type II curing)
NSO – BAS without SAP (Type II curing)

Observation from figure 7 clearly says that, by the end of 90 Days, the Bacterial concrete with SAP cured under normal water providing Nutrients inside displayed 74.3 % of healing and which are cured under Nutrient medium showed healing up to 66 %. And also, the Bacterial specimen cast without SAP which were cured under normal water providing Nutrients inside shows a healing of 49.7 % and which are cured under nutrients shows healing up to 57.4 %. The width of the crack in various specimens are differed from 0.1 mm to 0.5 mm. High healing ratios were observed for the images A and B shown below. Though the cracks were not completely healed at the span of 90 days, the minimum healing ratio of crack is noticed in image D.

Figure 8 A) represents SAP added Bacterial Specimen with nutrients inside cured in normal water. **B)** represents Bacterial Specimen without SAP having nutrients inside cured in normal water.

Figure 8 C) represents SAP added Bacterial Specimen cured in nutrients (calcium chloride and urea). **D)** represents Bacterial Specimen without SAP cured in nutrients (calcium chloride and urea).

4.4.1 Discussion on the Self –healing ratio in Prisms

Figure 8 A) has a healed area of 19.46 mm^2 of 26.17 mm^2 showing a healing ratio of 0.7439. Figure 8 B) representing a healed area of 20.48 mm^2 of 41.19 mm^2 showing a healing ratio of 0.4974. Figure 8 C) comprises a healed area of 8.46 mm^2 of 12.82 mm^2 showing a healing ratio of 0.6606. Figure 8 D) owns a healed area of 14.874 mm^2 of 25.893 mm^2 showing a healing ratio of 0.5744.

4.5 XRD Analysis

The Bacterial specimen with SAP which showed better self-healing is cut at the portion where the crack is healed and it is powdered for the study of X-Ray Diffraction pattern. XRD Analysis are done to obtain the structural information of the required sample, such as powdered sample (In this study). Here, X-Rays are produced by bombarding a Cu metal target with a beam of electrons of 1.54 x 10^{-10} Angstrom. When they hit the sample this diffraction pattern is observed. The intensity of X-Ray is 40 Kv/40 mA. The scan range of the instrument is between 0°and 110° (2θ degree) and its speed is about 0.5°/min. From the results of XRD pattern obtained below shown in figure 9, it is clear and evident that there is deposition of calcium carbonate in the cracks of the specimen. The initial peak observed in the current study at the angle 2θ (Bragg's angle) = 29°, has confirmed the existence of calcium carbonate with reference to the peak obtained from the literature [11,12].

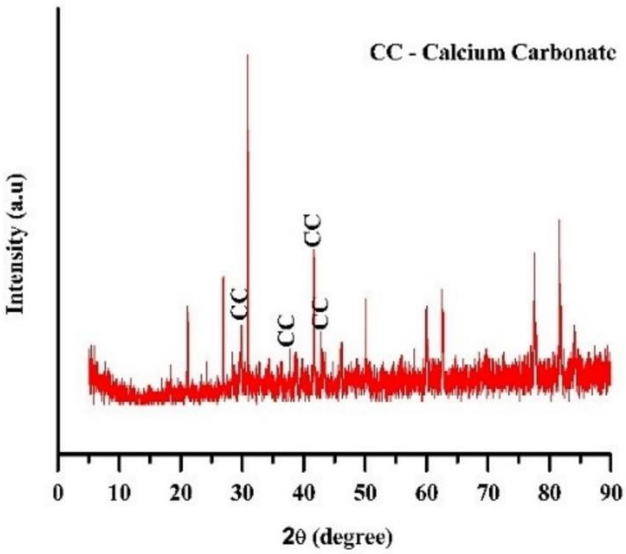

Figure 9 X-Ray Diffraction pattern

5. Conclusion

In this paper, results of investigation on the microbial precipitation of calcium carbonate by Bacillus Subtilis are presented. Two conditions of incubation were tried to attain more information on the higher efficiency of crack healing. In addition to the above study, Compressive Strength, water absorption, acid factors were carried out to appraise the Strength and Durability parameters of conventional and bacterial specimens. The following conclusions have been drawn.

i) Bacterial specimens with SAP is said to improve the strength parameters of the concrete and ensures reduced water absorbency.
ii) Bacterial specimens were found to show excellent resistance to the aggressive acidic environments when compared to the conventional specimens in usage.
iii) While considering the self-healing study, the cracked Bacterial specimens with SAP displays better healing when compared to the specimens without SAP. Maximum width of crack observed in this study that can be healed in bacterial specimens lie in the range between 0.10 mm and 0.45 mm.
iv) Addition of Nutrients found to be very important as it plays a vital role in healing process and improvement of strength.
v) The present idea of SAP embedded subtilis Biocrete seems to be an eco-friendly repair approach which would be useful in mere future if it comes into existence.

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