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RESEARCH ARTICLE

RESISTANCE OF BIO-BASED CEMENTITIOUS MATERIAL TO ACID ATTACK

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ABSTRACT

Concrete although is a mechanically strong construction material, but suffer from several inherent drawbacks, such as low tensile strength, permeability to liquid and consequent corrosion of reinforcement, susceptibility to chemical attack and low durability, ultimately reduced service life. Therefore, properties such as strength, permeability, crack formation and corrosion properties defines the overall quality of concrete. To improve the overall performance of concrete from these deficiencies, microbial mineral precipitation resulting from metabolic activities of some specific microorganisms in concrete has been attempted. Concrete incorporated bacteria can produce copious amounts of minerals which can potentially seal freshly formed cracks. However, its performance in presence of acid need to be understood. This paper reports the effects of microbial calcite precipitation on parameters affecting the transport processes and durability of mortar. To study the effect of durability, mortar cubes with and without Bacillus cohnii were cast and subjected to acid attack. Treatments were evaluated by visualizing and calculating the extent of acid attack in terms of % strength loss and % weight loss on 0.5%HCl and H_2SO_4 exposed mortar specimens. The durability of the bacteria treated specimens have been studied by measuring the resistance against acid attack in terms of acid attack factor and increased durability in terms of acid durability factor. Microbial calcite precipitation was quantified by X-ray diffraction analysis and visualized by SEM. Bacterial deposition of a layer of calcite on the surface of the specimens resulted in reduction in pore percentage and therefore, a decrease of acid ingress inside the mortar specimen was detected. From all the aforesaid studies it was revealed that bio-concrete is more durable.

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INTRODUCTION

Although concrete is the widely used construction material, despite its versatility in construction, it suffers from cracks due to various reasons in its service life. The mechanisms such as drying shrinkage, freeze-thaw reactions and mechanical compressive forces give rise to cracks in concrete (Jonkers, 2007). Although micro cracks (width smaller than 0.2mm) can self-heal, do not affect strength properties of concrete structures directly but they contribute to material porosity and permeability which leads to ingression of aggressive chemicals such as chlorides, sulfates and acids that results in concrete matrix degradation and premature corrosion of the embedded reinforcement hampering the structural performance in the long term (Jonkers and Schlangen, 2008). Crack prevention is often employed by steel reinforcement which is highly expensive and requires large amount of steel which will never be acceptable. From the perspective of durability, rapid sealing of freshly formed surface cracks is important as this prevents the ingress of moisture and other aggressive chemicals into the concrete matrix (Jonkers, 2009). Many traditional methods are in use for crack repair like impregnation of cracks with epoxy based fillers, latex binding agents such as acrylic, polyvinyl acetate, butadiene styrene, etc. However, there are several disadvantages associated with these in vogue repair systems, namely i)different thermal expansion coefficient/s compared to concrete, ii) weak bonding, iii) environmental and health hazards along with iv) high cost of the polymers/chemicals (De Belie and De Muynck 2009). Although cement has autonomous capacity to self-heal cracks smaller than 0.2mm width but in such cases, the alternative and robust way to overcome these disadvantages is by use of bioconcrete (bacteria incorporated in concrete). Currently, urease enzyme activity in most of the microorganism's metabolic processes have been used as a tool to induce the precipitation of calcite.

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Copious production of endospore forming bacteria-mediated bio-minerals subsequently results in clogging and crack closure, reducing material permeability and leakage. Biologically induced self-healing is a promising technology to increase the strength, life and durability of concrete structures. Biomineralization is one of the best eco-friendly techniques to tackle the problem of cracks in concrete structures. The consumption of oxygen during the metabolic biochemical reactions to form $CaCO_3$ helps in arresting corrosion of steel because the oxygen is responsible to initiate the process of corrosion. This method was already showing positive results in the field of prevention of acid mine drainage, prevention of leaching in channels and other such areas (Ghosh and Mandal, 2006). The evidence of microorganism involvement in calcite precipitation has led the development of bioprocess technology in the field of construction materials. Such bio based concrete is able to autonomously reduce matrix permeability substantially will be less susceptible to leakage and consequent reinforcement corrosion. It will not only save maintenance and repair cost considerably but also increases the service life of a construction. This phenomenon will reduce the need of raw materials, energy consumption and emission of waste products, particularly increased service life and reduced consumption of synthetic repair materials which are traditionally used for crack repair. Self-healing bioconcrete thus represents a more durable and sustainable alternative of classical concrete.

Experimental investigation

MATERIALS

The following are the material composition for development of bio-concrete mix.

Bacterial sources

The cultures of *Bacillus cohnii* (MTCC 10221) and *Bacillus megaterium* (MTCC 10086) were obtained from Microbial Type Culture Collection and Gene Bank, CSIR- Institute of Microbial Technology, Chandigarh. Both the strains of bacteria which were used for investigation were found gram positive and endospore forming. The urease positive test was carried out using phenol red indicator, which change the color of urea based broth from yellow to pink.

Maintenance of stock cultures

The bacterial strains were maintained constantly on nutrient agar slants and nutrient agar plates at pH 8.5 and were preserved in deep freezer (-80ºC) until further use. All the inoculated plates were incubated at 30ºC in Innova 44 shaker incubator. Colonies were observed after 24 and 48 hrs for *B.cohnii* and *B.megaterium*, respectively. Sub culturing of culture medium was carried out in every 15 days and contamination was checked periodically by streaking on nutrient agar plates.

Media composition for growth of bacteria

The bacteria were cultured in Himedia urea based broth. Calcium chloride hydrate was used as a source of calcium for precipitation of calcium carbonate. Aseptically 20g/liter of 40% urea and calcium source (calcium chloride) was also added after sterilization.

Cement

Ordinary Portland cement of 43 grade was used in the mix. The cement has been tested for various properties as per IS: 4031-1988 and found to be confirming to various specifications of IS: 12269-1987.

Sand

Natural river sand well graded passing through 60 µm sieve was used. Specific gravity was found to be 2.63. Fineness modulus was found to be 2.1, water absorption 0.26% and confirming to zone III.

Water

All mortar mixing was done using tap water.

Specimen preparation

Preparation of bio-admixture incorporated mortar cubes

Mortar cubes were prepared in 50.8 mm^3 cube mould, composed of one part of cement, four parts of standard sand by mass and (P/4 + 3) per cent (of combined weight of cement and sand), water (where P is the percentage of water required to produce a paste of standard consistency) and prepared, stored and tested in the manner described in IS 4031: Part 6 (1988). Control mortar specimens were casted without the addition of bio-admixture i.e biomass + chemical feed (calcium lactate). Optimised dose of bio-admixture was incorporated into mortar specimens in non-control/bacterial mortar specimens. Casting was undertaken in two sets for two different acids namely HCl and H_2SO_4, each set comprise of control specimens, biomass and calcium lactate incorporated specimen. In each set, total 36 cubes having 18 specimens each of control and bio-admixture were casted. Out of 18 specimens of control 9 were kept in water, another 9 in 0.5% HCL and/or 0.5% H_2SO_4. Same trend is followed in bacteria, calcium lactate incorporated specimens such that effect of acid can be observed distinctly. Each 9 specimens of water and acid exposed bio-admixture treated and non-treated specimens were categorized in sets of three specimens for durability studies after 30,60 and 90 days duration.

Standardized method for crack generation

Cracks of 0.3 mm width and 10-15 mm depth were generated on mortar cubes of dimension 50.8 mm^3 through insertion of aluminum plate (0.3 mm) at the time of casting .The aluminium plate was inserted into the wet mortar mixture to a depth of 25 mm and removed. Demolding of moulds was done after 24hrs. After demolding all specimens were cured in water at room temperature (27± 2°C) for 28 days.

Tests procedures

After casting of 50mm^3 mortar specimens and initial curing for 28 days, one set of specimens (with and without bio-

admixture), were immersed in 0.5% HCl and another set was immersed in 0.5% H_2SO_4 to study the effect of exposure to acidic environment. The arrangement of the specimens in the curing container was done in manner such that the clearance around and above the specimen should not be less than 30mm. The solution was refreshed in every 15 days. The effect of acid on the specimen were constantly monitored through visual observation, residual alkalinity, weight loss, strength loss, Acid Durability Factor and Acid Attack Factor measurement during the experimentation.

Study of bacterial growth curve

Fresh medium was inoculated with *B. cohnii* and the population growth was monitored over a period of time. Graph of optical density (O.D) verses time was then plotted by taking O.D at 600 nm after every 5 hours till 30hrs of incubation.

Visual observation

On exposure of 0.5% HCl and H_2SO_4, the mortar specimens (control and bacterial) were observed after regular interval of time for color change at surface, corners and edges. The extent of deterioration was observed with time and difference in destruction level in specimens was noticed and compared.

Residual alkalinity

Residual alkalinity was determined after cutting the specimens into halves us in low speed saw and spraying 1% phenolphthalein solution on the freshly cut surface.

Weight loss, Strength loss

Universal testing machine was employed to determine the compressive strength of the specimen at regular intervals. Specimens for weight change measurements were initially primed in water for 3 days and its weight in saturated surface dry condition was taken as initial weight.

Acid Durability Factor and Acid Attack Factor on control and bacterial mortar

To determine the concrete resistance to aggressive environment such as acid attack, the durability factors are proposed by the author as described in ASTM C666-1997, as the basis. In the present investigation, the "Acid Durability Factor" is derived in terms of relative strength of the specimens which is taken with respect to 28 days value.

Acid Durability Factor (ADF) = Sr N/M

Where,

Sr = relative strength at N days, (%)

xposure period.

N = number of days at which the durability factor is needed.
M = number of days at which the exposure is to be terminated.

Acid Attack test was terminated at 90 days, So, M is 90 days in this case. The extent of deterioration at each corner of the struck face and the opposite face is measured in terms of the solid diagonals (in mm) for each of two cubes and the Acid Attack Factor (AAF) per face is calculated as follows:

Acid Attack Factor (AAF) = (Loss in mm on eight corners of each of 2 cubes)/The percentage weight loss and strength loss at 30, 60 and 90 days of acid exposure has been presented in Table-1 and Fig.5. Acid Durability Factor (ADF)and Acid Attack Factor(AAF) are presented in Table-2,3 and Figures 5,6.

SEM - EDAX Analysis

SEM-EDAX analysis was carried out on the tested samples of the mortar cubes after 28 days of curing. Samples were gold coated with a JFC-1200 fine coater prior to examination. SEM micrographs were obtained using a ZEISS Ultra- Plus FEG-SEM. The morphology of bacteria and remediated crack was analysed with SEM at various magnifications [1x, 2x, 5x, 10x]. Calcite layer formed by the bacterial isolates were completely dried at room temperature and then examined by SEM.

RESULTS AND DISCUSSION

Study of bacterial growth curve

Maximum OD was seen between 6 to 10 hours and referred as log phase (Fig. 1). Spectrophotometer reading showed that the cultures reached the stationary phase after 10 hours. However, after 24 hour bacterial growth was inhibited due to the depletion of media components and release of secondary metabolites.

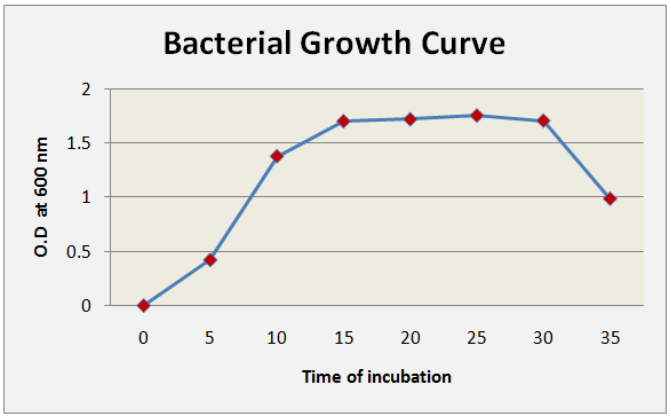

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Fig.1. Growth phases of Bacillus Cohnii

Visual Inspection

Specimens showed noticeable change in color, turned rested yellow at surface, corners and edges. Color change was found

to be more prominent in control specimens than in bacterial specimens. Similarly, the extent of deterioration was alleviated at surface, corners and edges of control specimens in contrast to bacterial specimens when exposed to 0.5% H_2SO_4 and 0.5% HCl solution.

Fig.2. HCl exposed mortar specimens (with and without Bio-admixture) after 30,60,90 days

Fig.3. H2SO4 exposed mortar specimens (with and without Bio-admixture) after 30,60,90 days

The surface seen more destructed from outer surface though inside remain structurally intact. On exposure to 0.5% sulfuric acid and hydrochloric acid, the deterioration of the surface was seen to increase with time and the extent of deterioration was easily differentiated in 30, 60 and 90 days. The deteriorated corroded surface of specimens observed at regular intervals and photographs at various stages of exposure are presented in Fig. 2, 3 for specimens in hydrochloric acid and sulfuric acid.

Residual Alkalinity

The residual alkalinity of the specimens were examined roughly by spraying a 1% phenolphthalein solution on the freshly cut surface. On spraying, dealkalized (acidic) part of specimen showed colourless while remaining part exhibited a magenta colour indicating its residual alkalinity. Fig.4 shows the residual alkalinity of specimen in acid solutions. It was noticed that the process of dealkalization progressed inwards with time. Alkalinity were seen to have almost lost in 12 weeks for bacterial specimen while control specimen were completely dealkalized earlier. For the same exposure duration, specimens in sulfuric acid solution showed faster dealkalization than its counterparts in hydrochloric acid.

Fig.4. Control and Bio-admixture incorporated specimens showing residual alkalinity after 30, 60, 90 days in HCl and H_2SO_4

Weight Reduction and Strength Loss

The results of studies on percentage of weight reduction and percentage of strength loss in conventional/control mortar and optimized dose incorporated bacterial mortar specimens at

different ages (30,60 and 90 days) are given in Table 1, Fig. 5,6. In this case study, it can be observed that pores are partially clogged by material growth in bio-admixture incorporated mortar.

Reduction in pore due to such material growth will obviously increase the density of the material and strength. It is observed that there is less percentage of weight reduction and compressive strength of bacterial mortar when compared with conventional mortar specimens. In addition to this, it is seen that the specimens immersed in water showed no weight loss and strength loss in comparision to acid immersed specimens.

ADF and AAF

On immersion in HCl and H_2SO_4, the ADF, AAF of conventional/control and bio-admixture incorporated mortar specimens are given in Table 2 and 3 and Figures 7,8 respectively for 30,60,90 days.

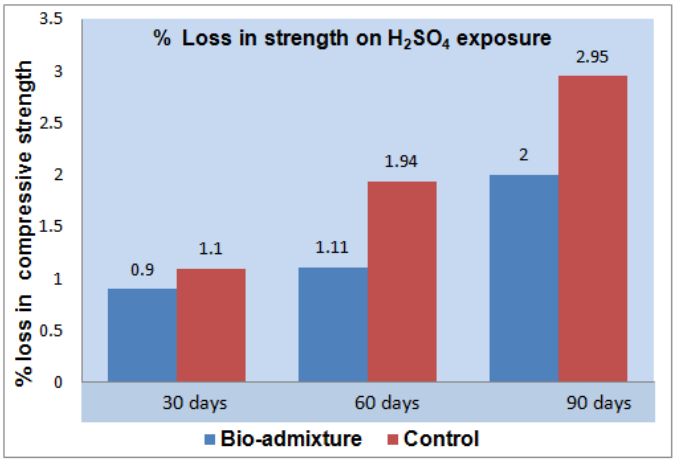

Fig.6. % Loss in compressive strength on H_2SO_4 exposure

Table I. % Loss in weight on acid exposure

Acids	HCl						H_2SO_4					
	Control			Bio-admixture			Control			Bio-admixture		
No. of days	30	60	90	30	60	90	30	60	90	30	60	90
Initial weight	2.560	2.560	2.560	2.560	2.560	2.560	2.560	2.560	2.560	2.560	2.560	2.560
Weight at refined age	2.540	2.485	2.438	2.550	2.507	2.468	2.533	2.477	2.385	2.545	2.529	2.440
%Weight reduction	0.78	2.92	4.76	0.39	2.07	3.59	1.05	3.24	6.83	0.58	1.21	4.68

Table II. Acid durability factor of control and bacterial mortar specimens

Period of immersion in HCl	Relative strength		N	M	Acid DurabilityFactor	
	Control	Bio-admixture			Control	Bio-admixture
30 days	92.69	96.72	30	90	32.23	32.24
60 days	85.51	90.98	60	90	57.00	60.04
90 days	77.14	89.67	90	90	77.14	89.67

N = number of days at which the durability factor is needed.
M = number of days at which the exposure is to be terminated

Table III. Acid durability factor of control and bacterial mortar specimens

Period of immersion in H_2SO_4	Relative strength		N	M	Acid DurabilityFactor	
	Control	Bio-admixture			Control	Bio-admixture
30 days	86.21	92.60	30	90	28.73	30.86
60 days	77.10	90.90	60	90	50.88	59.99
90 days	65.20	83.60	90	90	65.20	83.60

N = number of days at which the durability factor is needed.
M = number of days at which the exposure is to be terminated

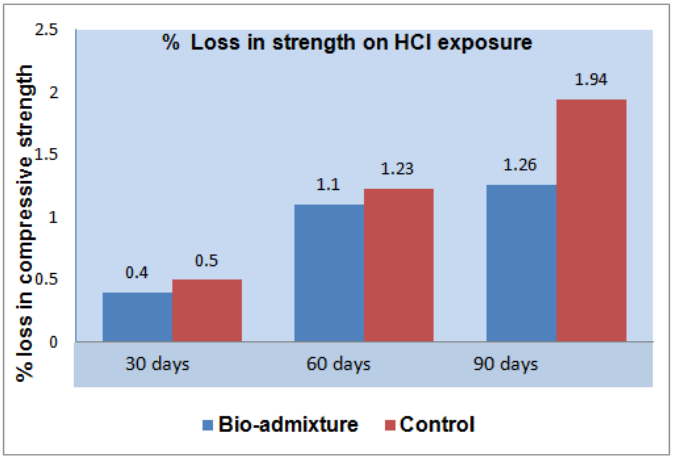

Fig.5. % Loss in compressive strength on HCl exposure

SEM - EDAX Analysis

Fig. 9 A is scanning electron micrograph of control mortar matrix indicating atomic % of calcium 4.48.

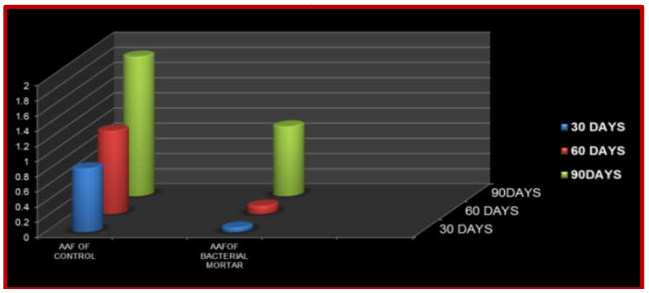

Fig.7. Acid Attack (HCl) on Control & Bacterial Mortar specimens

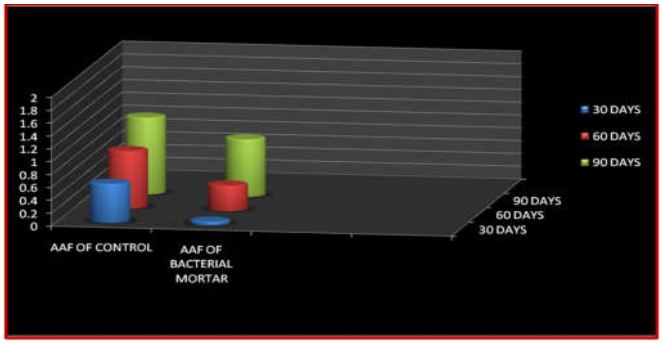

Fig.8. Acid Attack (H_2SO_4) on Control & Bacterial Mortar specimens

Microbial calcite precipitation in the biomass incorporated mortar specimen is evident through SEM. Fig.9 B,C shows micrographs of the specimen prepared with *B.cohnii* (biomass) and calcium lactate. The sample showed calcite crystals grown on the cell wall of rod shaped bacteria. Calcium % in this treated specimen was 24.75 and 25.87 respectively. The abundant presence of Ca was evident and the precipitation was inferred to as calcite (CaCO3) crystals.

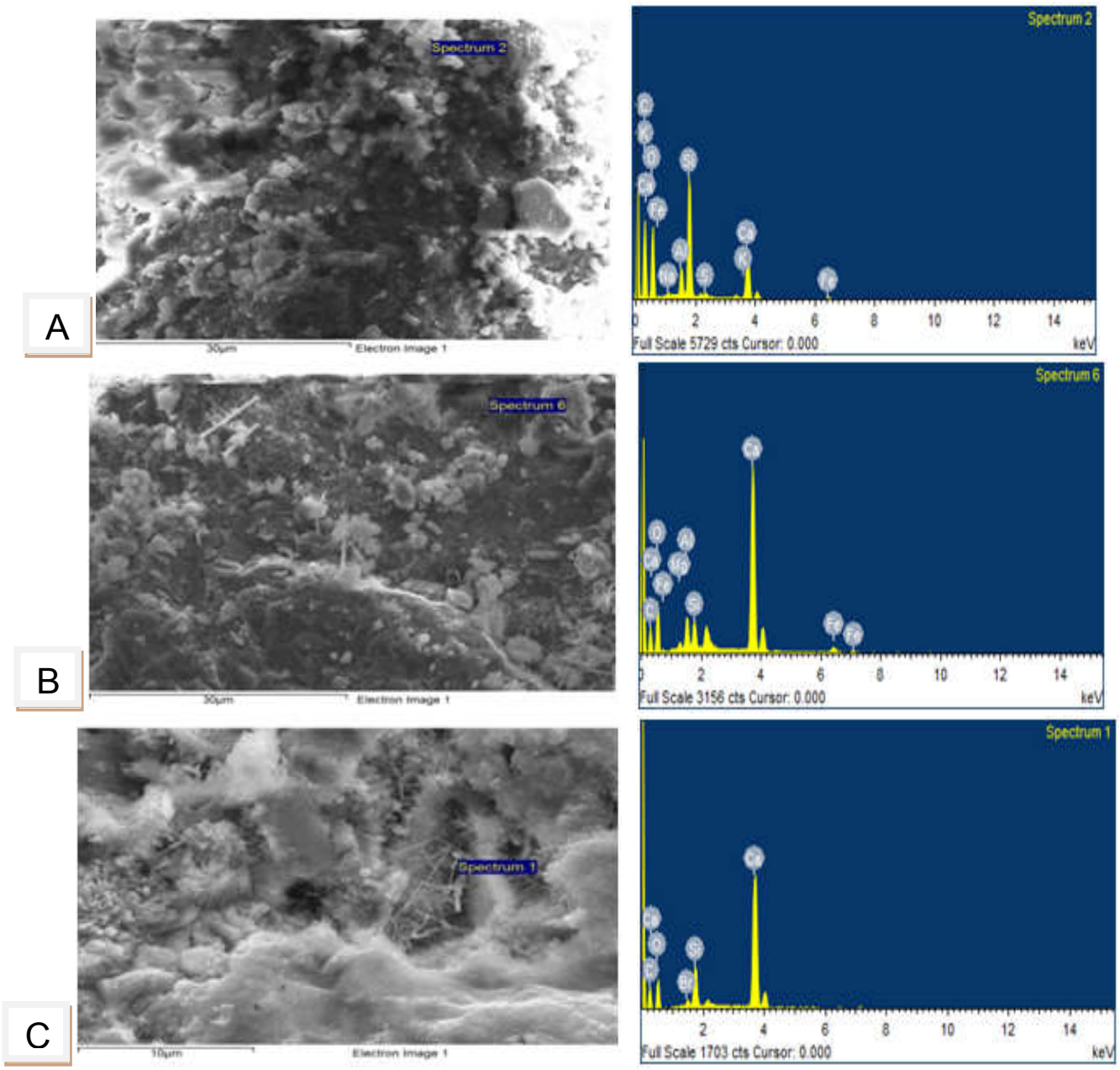

Fig.9. Scanning electron micrographs and EDEX of A) Control and B,C) Bio- admixture incorporated mortar specimens shows copious amount of calcite on the surface of embedded rod shaped bacteria inside cement sand matrix

Conclusion

The current study demonstrated the link between permeability and degradation in cementitious materials. Result confirms that, incorporation of ureolytic bacteria in mortar increases the resistance of mortar specimens towards degradation processes. The deposition of calcite and microbial biomass resulted in a decrease of the permeation properties of cementitious materials by clogging the pores in the cement sand matrix. As a result, the ingress of aggressive chemicals was limited. Moreover, the use of calcium lactate as a chemical feed show that a similar protective performance was obtained as in case of biomass alone.

Calcium carbonate is solubilised in acidic environments, therefore the prime concern of future research is to focus on the effect of acidic rain on the durability of the biodeposition treatment and use of different chemical feed.

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