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Studies on Pore Structure Characterization of Bacterial Concrete

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***Abstract* - The aim of the present investigation is to understand the pore structure of bacteria incorporated concrete using Brenauer-Emmett-Teller's (BET) Nitrogen (N2) nitrogen adsorption method and other porosity tests. These tests confirm the modification in pore size distribution in bacterial concrete is due to the addition of microorganisms. *Bacillus subtilis* JC3 of 10^5 bacterial cells / ml, when added to the concrete during mixing stage improves the pore structure due to metabolic deposition of $CaCO_3$ in the voids /or pores within cement–sand matrix modifying the pore structure of bacteria induced cement mortar specimens generating the greatest reduction in porosity. Reduction in pores due to such material precipitation (calcium carbonate) will eventually increase the concrete strength. In the present study, porosity of bacterial concrete in terms of specific surface area, pore size distribution, and pore volume, was examined using the Brenauer-Emmett-Teller BET nitrogen adsorption method based on DIN 66131.**

Keywords - Bacterial Concrete, Porosity, BET, DIN 66131, permeation properties

I. INTRODUCTION

The porosity of a material can be expressed in various forms. The total porosity can be expressed either as a ratio of the void volume to the bulk volume of the material, or as a ratio of the void volume to the solid volume. The first ratio is generally referred to as porosity; the latter ratio is referred to as voids ratio. Measurement of the true total porosity is difficult. Most testing procedures which claim to evaluate total porosity, in fact only measure the effective porosity for various conditions. The pore system in cement-based materials is classified as in increasing order of size: (a) gel pores (b) capillary pores (c) macropores - due to deliberately entrained air and inadequate compaction. In concrete, in addition to the above pores, there can be cracks at aggregate-mortar interface due to shrinkage. The gel pores, which are mostly of 1.5-2.0 nm size, do not influence the strength of concrete adversely through its porosity, although these pores are directly related to creep and shrinkage. Capillary pores and other larger pores, on the other hand, are responsible for reduction in strength and elasticity, etc. Thus, while dealing with an empirical strength-porosity relationship of concrete, contribution of the gel pores in the overall porosity and pore size distribution of concrete can be neglected, without introducing any significant error. It is widely recognized that the porosity of a material exerts an enormous influence on its physical properties. The residue of water filled space in fresh concrete become voids in hardened concrete. As hydration progresses, the amount and distribution of porosity between capillary and gel pores will change. Initially all the pores are capillary pores. As hydration precedes the capillary pore volume is reduced because the capillary space becomes filled with hydration products, and the gel porosity increases. The durability of concrete structures is often related to the capacity of concrete to prevent the penetration of aggressive agents (sulphates, chlorides, CO2…) in its porous structure. The aptitude of concrete cover to resist to these external chemical attacks is conditioned by its properties of transfer (porosity, permeability and diffusivity). These physical properties are related to the characteristics of the porous environment (the shape of the pores, distribution of the sizes of the pores and tortuosity). Two independent methods, proposed for pore structure analysis, for estimating pore volume and pore diameter, are the gas adsorption method (also known as the Brenauer- Emmett-Teller method) (BET) [1] [2] and the mercury porosimeter method (MIP). These two methods, with various modifications, are used by industry and in research. Results of the nitrogen adsorption technique cover both gel and capillary pores of the concrete mixtures, while entrapped air and capillary pores of diameter larger than 300nm are not accounted. In the present study gas adsorption method (also known as the Brenauer- Emmett-Teller method) (BET) [3] is used to understand the pores structure of bacteria embedded concrete.

II. WORKING PRINCIPLE OF BACTERIAL CONCRETE

B. Subtilis JC3 member of the genus Bacillus is Gram-positive, rod-shaped, endospore forming bacteria commonly found in soil; precipitate calcium carbonate ($CaCO_3$) in its micro-environment by the ammonification of amino acids into ammonium (NH_4^+) and carbonate (CO_3^{2-}) ions. Microbiologically induced (also called ‘‘bacteriogenic’’) calcite carbonate precipitation by *Ammonification* (Ammo acid degradation) comprises of series of complex biochemical reactions. Amino acids released during proteolysis (the process of enzymatic breakdown of proteins by the microorganisms with the help of proteolysis enzymes) undergo deamination in which nitrogen containing amino (-NH_2) group is removed. Thus, process of deamination which leads to the production of ammonia is termed as "ammonification". The process of ammonification is mediated by *Bacillus subtilis* JC3.

Ammonification usually occurs under aerobic conditions (known as oxidative deamination) with the liberation of ammonia (NH_3) or ammonium ions (NH_4) when dissolved in water .The biochemical reactions of ammonification in peptone based medium is represented as follows

$$Ca^{2+} + \mathit{B.subtilis}\ \text{Cell} \rightarrow \mathit{B.subtilis}\ \text{Cell-}\ Ca^{2+} \quad (1)$$

$$CH_3CH(NH_2)COOH\ (\text{Peptone}) + ½O_2 \text{ --------->} C_2H_2 + H_2CO_3 + NH_3 \quad (2)$$

$$H_2CO_3 \text{ ---------->} H^+ + HCO_3^- \quad (3)$$

$$NH_3 + H_2O \text{ -------->} NH_4^+ + OH^-\ (\text{pH increase}) \quad (4)$$

$$HCO_3^- + H^+ + OH^- \text{ ---------->} CO_3^{2-} + H_2O \quad (5)$$

$$\mathit{B.subtilis}\ \text{Cell-}\ Ca^{2+} + CO_3^{2-} \rightarrow \mathit{B.subtilis}\ \text{Cell-}CaCO_3 \quad (6)$$

III. EXPERIMENTAL INVESTIGATIONS

To quantify and understand the pore structure of bacteria induced cocnrete the following tests were conducted:

1. Pore structure analysis using BET Nitrogen (N_2) Adsorption Method as per DIN 66131
2. Porosity by the gravity method
3. Density, Water Absorption Capacity and Volume of permeable voids as per ASTM C642

A. *Brenauer-Emmett-Teller (BET) Nitrogen (N_2) Adsorption Method*

Porosity characteristics of bacterial concrete including total volume of pores, pore size distribution and specific surface area were determined using the BET nitrogen adsorption method. The surface area (s) to pore volume (v) ratio of a porous medium plays an important role in determining permeability and affects absorption in diffusion-based processes. Porosity tests were carried out using the nitrogen adsorption method (per DIN 66131) [4] [5]. The experimental procedure of using Brenauer-Emmett-Teller's (BET) nitrogen adsorption method [6] to determine the volume, size and surface area of pores in concrete is as follows: The ground samples were passed through a sieve to obtain sample sizes ranging from 12 to 10 mm. The sieved samples were put into test tubes and were degassed. This process involved temperature desiccation and gas elimination using a vacuum. After the samples were degassed, they were saturated (at atmospheric pressure) using an inert gas (nitrogen). Thereafter, they were sealed using a plug. The sealed tube was opened for the subsequent analysis process. This consists of extracting the inert gas and starting the test. Finally, during the testing process the sample was covered with a cell that guaranteed constant thermal conditions throughout the test. Small samples (2 g) were taken from concrete specimens that were moist cured for 28 days. The nitrogen adsorption technique used in this study has the ability to measure pores with a diameter between 0.3 and 300 nm. All various tests were carried out at a room temperature. Results of the nitrogen adsorption technique cover both gel and capillary pores of the concrete mixtures, while entrapped air and capillary pores of diameter larger than 300 nm are not accounted. Nitrogen sorption isotherms were determined after degassing for two hours using Quantachrome NOVA1000. Specific surface area was calculated using BET (Brunauer- Emmett-Teller) method (Brunauer *et al.*, 1938) and pore size distribution was obtained according to DH (Dollimore-Heal) method (Dollimore and Heal, 1964). The gas adsorption method is based on the phenomenon of gas condensation in narrow pores at pressures lower than saturated vapour pressure of the examined material. Classically, volumes of gas progressively adsorpted by the material, and those of gas progressively desorpted, are represented by the isotherms .The gas adsorption technique is performed by the addition of a known volume of gas (adsorbate), typically nitrogen, to a solid material in a sample vessel at cryogenic temperatures. At cryogenic temperatures, weak molecular attractive forces will cause the gas molecules to adsorb onto (attach to the surface of) a solid material. An adsorbate (gas) is added to the sample in a series of controlled doses, the pressure in the sample vessel is measured after each dosing. There is a direct relationship between the pressure and the volume of gas in the sample vessel. By measuring the reduced pressure due to adsorption, the ideal gas law can then be used to determine the volume of gas adsorbed by the sample. The resulting relationship of volume of gas adsorbed vs. relative pressure at constant temperature is known as an *adsorption Isotherm*. From the analysis, and the cross-sectional area of the adsorbate gas molecule, the surface area and pore size distribution of the sample can be derived. The gas adsorption can be used to determine pore size distributions from sorption isotherms. Experimental adsorption isotherms are the most common way of describing adsorption phenomena. Methods for obtaining the adsorption data for the adsorption isotherms are based on measuring the amount of gas (liquid) removed from the gas (liquid) phase during adsorption, and on various ways of determining the amount of the adsorbed substance (adsorbate) on the surface of the adsorbing substance (adsorbent); for example, volumetric method, gravimetric method, etc. Adsorption isotherms are used to calculate the specific surface area of materials, mean pore size or mean size of deposited particles, as well as pore size or particle size distribution. The Barrett–Joyner–Halenda (BJH) interpretation uses pore condensation principle to evaluate the pore size distribution from the adsorption isotherm [7]. Also from BJH interpretation of adsorption isotherm the pore specific area is evaluated through pore condensation volume and the average pore size with cylindrical pore geometry assumption [8] [9].

B. *Porosity by the gravity method*

This method consists of saturating the concrete sample of size 100 x100 mm cube. Once it is fully saturated, it is weighted with centigram precision and its volume V is determined by weighing. Then, the sample is submitted to moderate oven drying at a temperature of 60 ±2 °C. The drying is stopped when the weight of the sample remains constant. The weight of the dried sample is obtained after 21 days of drying.

Porosity, p is then determined using the following formula:

$$P = \frac{M_{sat} - M_{dry}}{\rho_w V}$$

Where ρ_w the unit mass of water (1 g/cc), V is the volume of sample (100 x 100 x100 mm^3) , M_{dry} and M_{sat} denote the weight of the dried and fully saturated samples, respectively. The porosity can be expressed either as a fraction or as a percentage.

C. *Density, Absorption and Voids in Hardened Concrete (ASTM C642)*

For measuring the voids and absorption of the hardened concrete samples, the ASTM C642 (2006) standard test method was used. A balance, water bath, and container suitable for immersing the specimen are needed for performing the test. After the 100x 100 mm cube samples were cured in the curing room for the required time, three samples were removed from the curing room and put into an oven at 100° C for 24 hours. The dried samples were taken from the oven and put on the cabinet to cool for about 30 minutes. The samples were then weighed (M_a) using a balance with an accuracy within 0.01 grams. The samples were submerged in the water tank for 24 hours. It should be noted that if warm samples were put in the tank, they might crack, so they were allowed to cool first for 4 hours. After 24 hours, the samples were removed from the water tank and their surface was dried with a paper towel to obtain a saturated surface dry (SSD) condition. The weight (M_b) of the SSD samples was measured. In the next step, the samples were put into a water bath with boiling water for 5 hours. The total time that the samples were in the water bath was about six and half hours, including 90 minutes for heating up. The samples were removed from the boiling water and left in the laboratory environment (on the cabinet) for 12 hours. The weight of the samples was measured (M_c). On the same day, the apparent weight of each sample (M_d) was measured by immersing the samples in the water using a hanging balance. Using the measured weights (M_a to M_d) the absorption after immersion, the bulk density, the apparent density, and the volume of permeable voids can be calculated.

$$\text{Water Absorption Capacity (WAC)} = \frac{M_b - M_a}{M_a} \times 100$$

$$\text{Bulk density} = g_1 = \frac{M_a}{M_c - M_d} \times \rho$$

$$\text{Apparent density} = g_2 = \frac{M_a}{M_a - M_d} \times \rho$$

$$\text{Volume of permeable voids (VPV)} = \frac{g_2 - g_1}{g_2} \times 100$$

Where:

M_a = mass of oven-dried sample in air, kg

M_b = mass of surface-dry sample in air after immersion, kg

M_c = mass of surface-dry sample in air after immersion and boiling, kg

M_d = apparent mass of sample suspended in water after immersion and boiling, kg

g_1 = bulk density, dry (kg/m^3) and g_2 = apparent density (kg/m^3)

ρ= density of water (1000 kg/m^3)

The rate of water absorbed into concrete through the pores gives important information about the microstructure and permeability characteristics of concrete. Experimental results show that the depth of water absorbed into concrete increases linearly with respect to the square root of wetting time (Parrott 1992). In terminology, the sorptivity is the change in volume of water absorbed per unit area against the square root of time (Claisse et al. 1997). Water absorption and sorptivity can suggest useful data regarding the pore structure of the concrete.

The water absorption was determined on 100mm cubes as per ASTM C-642 by drying the specimens in an oven at a temperature of 105^0 C to constant mass and then immersing in water after cooling to room temperature. The specimens were taken out of water at regular intervals of time and weighed. The process was continued till the weights became constant (fully saturated). The difference between the water saturated mass and oven dry mass expressed as a percentage of oven dry mass gives the Water Absorption Capacity (WAC). The Water Absorption Capacity (WAC) of concrete is a measure of the pore volume or porosity in hardened concrete, which is occupied by water in saturated condition. The porosity obtained from absorption tests is designated as effective porosity. It is determined by using the following formula.

Effective Porosity = (Volume of voids / Bulk volume of specimen) x 100

The volume of voids is obtained from the volume of the water absorbed by an oven dry specimen or the volume of water lost on oven drying a water saturated specimen at 105^0 C to constant mass. The bulk volume of the specimen is given by the difference in mass of the specimen in air and it's mass under submerged condition in water.

Table 1: Durability Classification as per ASTM C 642

Classification	Volume of Permeable Voids (VPV) (% by volume)	Water Absorption Capacity (% by weight)
Excellent	<14	<5
Good	14-16	5-6
Normal	16-17	6-7
Marginal	17-19	7-8
Bad	>19	>8

IV. TEST RESULTS

Pore diameter, Total pore surface area, Total pore volume and porosity of controlled and bacteria incorporated concretes of M20, M40, M60 and M80 grades are obtained and tabulated in Table 2 from the adsorption isotherms and BJH isotherms and corresponding graphs are plotted as shown in Figure 1 and 2.

Table 2: Pore diameter, Total pore surface area, Total pore volume and porosity of various grades of controlled and bacteria incorporated concretes

	M20	M20B	M40	M40B	M60	M60B	M80	M80B
Average Pore Diameter (nm)	19.121	3.386	5.084	3.097	3.189	3.095	3.096	3.083
Total Pore Surface Area (m²/g)	1.747	5.047	3.346	5.088	5.152	6.795	5.333	6.812
Total pore volume (cc/g)	0.0161	0.0071	0.0137	0.0057	0.0052	0.0042	0.0047	0.0038
Porosity(%)	1.6	0.7	1.37	0.57	0.52	0.42	0.47	0.38

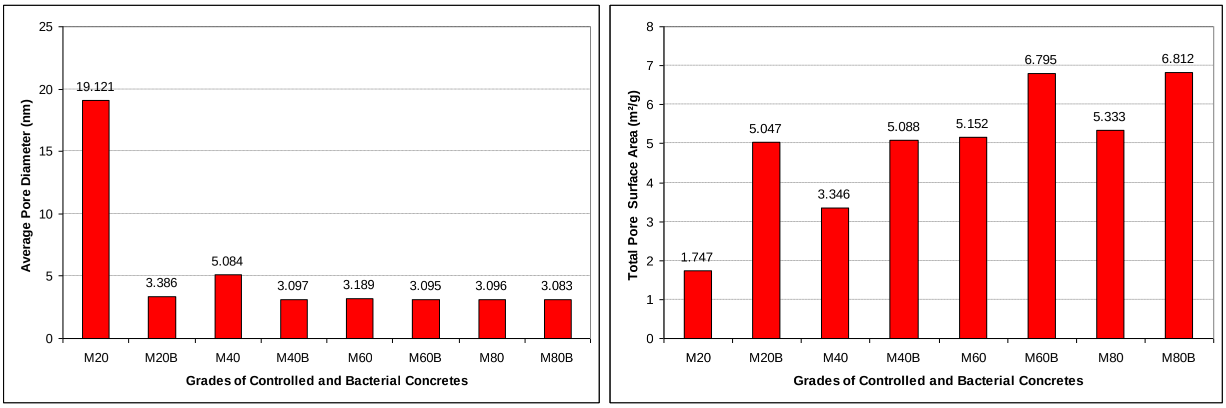

Figure 1: Pore diameters and Total pore surface area of various grades of controlled and bacteria incorporated concretes

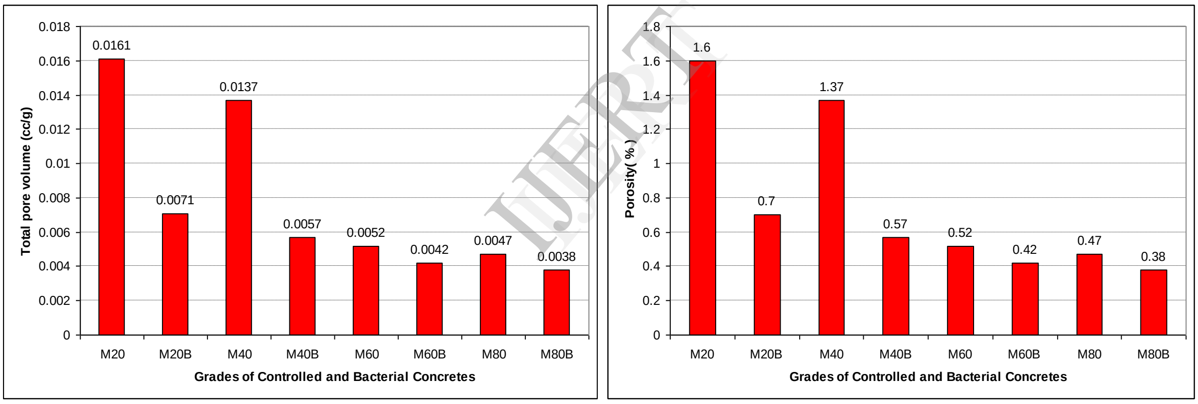

Figure 2: Total pore volume and Porosity of different grades of controlled and bacteria incorporated concretes

Table 3 and Figure 3 presents the porosity of various grades of controlled and bacteria incorporated concrete at different ages found using gravity method. Table 4 and Figure 4 give the water absorption capacity (WAC), volume of permeable voids and apparent porosity of all grades of controlled and bacteria incorporated concrete specimens.

Designations used:
M20 – M20 Grade Controlled Concrete
M20B – M20 Grade Bacterial Concrete
M40 – M40 Grade Controlled Concrete
M40B – M40 Grade Bacterial Concrete
M60 – M60 Grade Controlled Concrete
M60B – M60 Grade Bacterial Concrete
M80 – M80 Grade Controlled Concrete
M80B – M80 Grade Bacterial Concrete

Table 3: Porosity of various grades of controlled and bacteria incorporated concrete at different ages

	Controlled Concrete				Bacterial Concrete			
	M20	M40	M60	M80	M20	M40	M60	M80
M_{sat} (kg)	2.63	2.58	2.63	2.66	2.55	2.56	2.62	2.66
M_{dry} (kg)	2.49	2.51	2.59	2.63	2.51	2.53	2.60	2.64
Porosity, P at 28 days	0.14	0.07	0.04	0.03	0.04	0.03	0.02	0.02
Decrease in Porosity	-	-	-	-	72%	57%	50%	34%

	Controlled Concrete				Bacterial Concrete			
	M20	M40	M60	M80	M20	M40	M60	M80
M_{sat} (kg)	2.63	2.58	2.63	2.66	2.55	2.56	2.62	2.65
M_{dry} (kg)	2.52	2.53	2.60	2.64	2.52	2.54	2.60	2.64
Porosity, P at 60 days	0.11	0.05	0.03	0.03	0.03	0.02	0.02	0.02
Decrease in Porosity	-	-	-	-	73%	60%	34%	34%

	Controlled Concrete				Bacterial Concrete			
	M20	M40	M60	M80	M20	M40	M60	M80
M_{sat} (kg)	2.63	2.58	2.63	2.66	2.55	2.56	2.62	2.66
M_{dry} (kg)	2.49	2.51	2.59	2.63	2.51	2.53	2.60	2.64
Porosity, P at 90 days	0.10	0.04	0.03	0.03	0.03	0.02	0.02	0.02
Decrease in Porosity	-	-	-	-	70%	50%	34%	34%

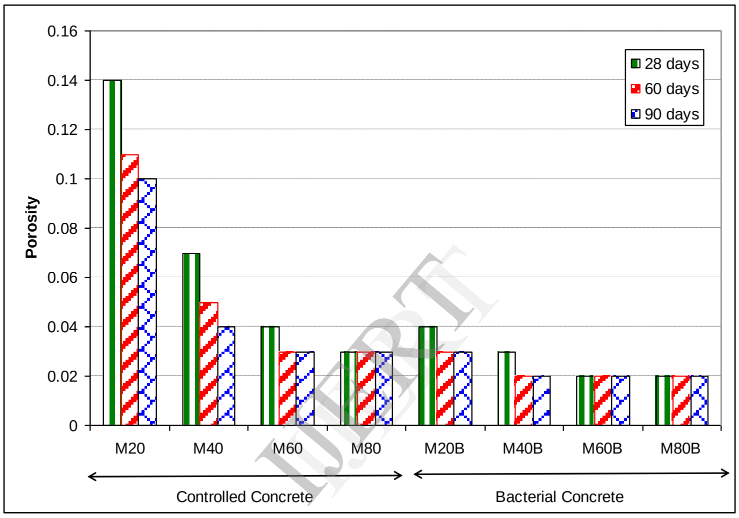

Figure 3: Porosity of controlled and bacteria incorporated concrete specimens of various grades at different ages

Table 4: Water Absorption Capacity (WAC), Volume of Permeable Voids and Apparent porosity of controlled and bacteria incorporated concrete specimens for different grades

	Controlled Concrete				Bacterial Concrete			
	M20	M40	M60	M80	M20	M40	M60	M80
M_a	2.49	2.51	2.59	2.63	2.51	2.53	2.60	2.64
M_b	2.63	2.58	2.64	2.67	2.58	2.56	2.62	2.65
M_c	2.64	2.59	2.65	2.69	2.59	2.57	2.64	2.67
M_d	1.49	1.45	1.5	1.51	1.46	1.46	1.5	1.51
bulk density (g_1) (kg/m^3)	2184.21	2221.24	2271.93	2267.24	2241.07	2300.00	2321.43	2315.79
apparent density (g_2) (kg/m^3)	2490.00	2367.92	2376.15	2348.21	2390.48	2364.49	2363.64	2336.28
Water Absorption Capacity (WAC) (%)	5.62	2.79	1.93	1.52	2.79	1.19	0.77	0.38
Volume of permeable voids (VPV) (%)	12	6	4	3	6	3	2	1

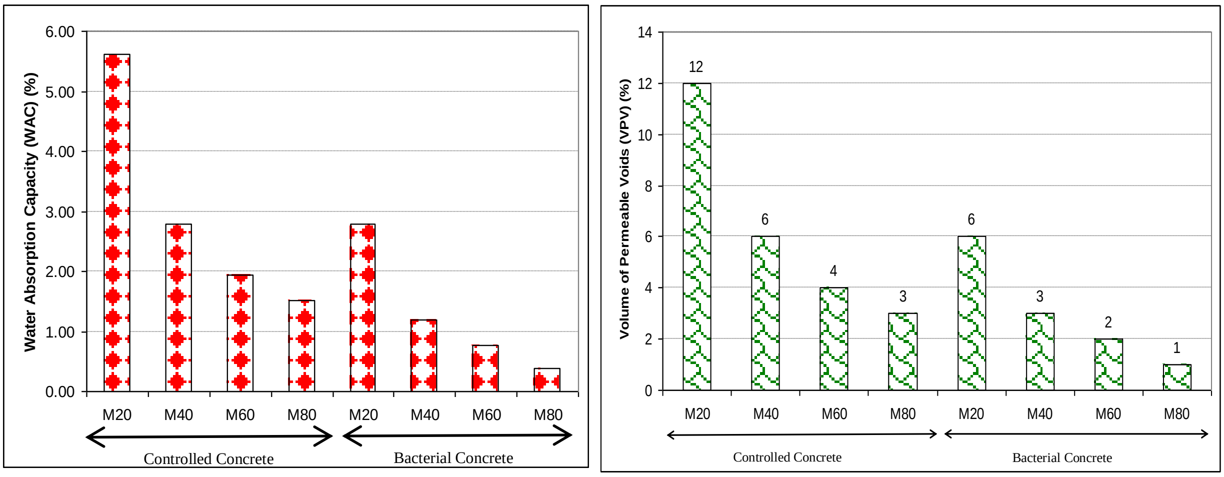

Figure 4: Water absorption Capacity and volume of permeable pore space of controlled and bacteria incorporated concretes

V. DISCUSSIONS

The analysis of the BET isotherms and hysteresis produced by the adsorption and desorption branches prompted the following major conclusions.

The shape of the isotherm in M20 , M40, M60 and M80 controlled concrete specimens is Type-1 isotherm which is characteristic of micro porous solids whose microstructure is continuously graded pores where as shape of the isotherms in M20 , M40, M60 and M80 bacterial concrete specimens is characteristic of non-porous solids of Type-2 and Type-3 isotherms. Average pore diameter, observed using BJH method, decreases from lower to higher grades of concrete and this observation is significant in bacteria incorporated concrete in which average pore diameter decreases drastically due to mineral precipitation in the pores because of microbial metabolic activity. Total Pore surface area (from Multi-point BET data) increases with the grade of concrete. Surface area of bacterial concrete specimens of different grades is found to be more than surface area of controlled concrete specimens. As average pore diameter decreases from lower to higher grades of concrete, total pore volume also decreased as grade increases. The decrease is more in case of bacteria incorporated concrete samples. Total porosity decreases significantly as grade of bacteria induced specimens' increases. As the pore diameter increases, the pore volume also increases leading to high porosity, possibility of water absorption and a decrease in density. Porosity in bacteria incorporated concretes is reduced by 30 to 75% for high to low grades. Total pore volume in bacteria incorporated concretes is reduced by 20 to 60% for high to low grades. A significant decrease in porosity and average pore diameter was observed in bacteria incorporated concrete by the addition of bacteria as compared to the control concrete. Concrete specimens incorporated with bacteria showed significantly less water absorption capacity compared to controlled specimens. This decrease in water absorption capacity of all grades of bacteria incorporated concretes is attributed to the reduction of pores in the concrete. Water Absorption Capacity (WAC) of bacteria incorporated concrete specimens is reduced by nearly 50 to 75% for low to high grade concretes as compared with WAC of controlled concrete specimens due to pore plugging with bacteria produced calcite minerals thereby modifying the pore structure of the cement –sand matrix. The absorption characteristics indirectly represent the volume of pores and their connectivity. Porosity of concrete specimens measured using gravity method shows that with induction of bacteria into concrete are reduced by nearly 34 - 73% for high to low grades. The possible reason for this is calcite mineral precipitation in the pores reduced the average pore radius of concrete. This means that the time taken for the water to rise by capillary action in bacteria incorporated concrete is longer and thus proved that these bacteria induced concretes are less porous compared to the control concrete. Volume of permeable voids present in bacteria incorporated concrete is less by 50-65 % than in controlled specimens. The rate of water absorbed into concrete through the pores gives important information about the microstructure and permeability characteristics of concrete.

VI. CONCLUSIONS

Based on the research and results presented in this paper, the following conclusions are reached:

The porosity decreases significantly in all the grades of bacteria incorporated concrete in comparison with the porosities shown by the corresponding grades of controlled Concrete because pore diameter and total pore volume are very low in all grades of bacteria induced concrete due to calcite mineral precipitation in the pores by *Bacillus subtilis* JC3 metabolic activity. The water absorption values for bacteria induced concrete are lower than the controlled concrete. These results emphasize the beneficial effect of incorporating microorganisms to increase the durability of concrete. The effect is more pronounced in high strength grade concrete. Total pore surface area increases in bacteria incorporated concrete specimens of different grades due to formation of dense microstructure. It can be concluded that all grades of bacteria incorporated concretes have less water absorption capacity compared to corresponding grades of controlled concrete specimens due to pore plugging with bacteria produced calcite minerals. This reduction of porosity in bacteria incorporated concretes indicates the presence of less volume of permeable voids. Volume of permeable pores (VPV) of bacteria incorporated concrete specimens are reduced since calcite mineral precipitation in the pores reduced the average pore radius of concrete by inducing pore discontinuity in the hydrated cement paste. This means that the time taken for the water to rise by capillary action in bacteria incorporated concrete are longer and thus proved that these concrete are less porous compared to the conventional concrete.

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