obtain uniformly cemented specimen. The results shown in Fig. 2 indicate that an increase in concentration of cementation media is limited in its effectiveness to enhance MICP. SEM images in Fig. 3 show that the high  $CaCl_2$  concentration led to more precipitation on the specimen surface than the low  $CaCl_2$  concentration.

Specimen s14 was treated with multiple bacteria injection with less retention time and shows the most uniformly cemented specimen ash shown in Fig. 4 (a). A single bacterial solution injection was not enough to get a uniform and well-cemented specimen. Bacteria will be partially lost during nutriment treatment cycles. Multiple injections of bacterial solution, in the middle of nutrient cycles, provided more bacteria into the soil and resulted in more calcite precipitation. Specimen s15 and s16 as shown in Fig. 4 (c) shows the best cemented specimens among all 16 tests. Fig. 5 shows the bacteria traces and precipitated calcite on treated sand grain surface.

# CONCLUSIONS

MICP tests of sands were performed on 16 specimens by varying different injection method, procedure, and drainage condition. Visual observation and SEM images of the treated specimens show that an increase of bacteria and chemical concentrations increased the urease activity and precipitation rate. However, there remains the problem of getting uniform precipitation due to the accumulation of bacterial cells and clogging of chemicals near injections. The effect of particle size on cementation also gave an important conclusion that the particles should be neither too fine nor too coarse for a good cementation. Based on the results of all the above described tests, the test soil should be fine to medium coarse and the concentrations of treatment solutions should be low, with a larger number of bacteria and nutrient treatment cycles, to achieve a more uniform MICP precipitated specimen.



FIG. 2. Photo of MICP specimens treated with different CaCl<sub>2</sub> concentrations.

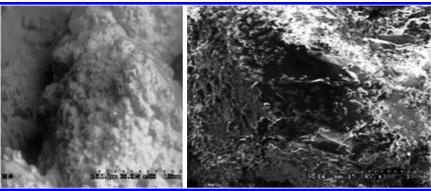


FIG. 3. SEM Images of precipitated calcites at 100 m scale: (left) 0.25 M/L CaCl<sub>2</sub> solution, (right) 0.025 M/L CaCl<sub>2</sub> solution.

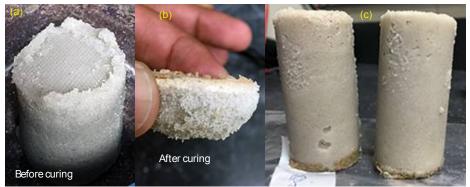


FIG. 4. Photo of treated specimens, (a) and (b) specimen s14 treated with multiple bacteria injections, (c) specimen s15 and s16.

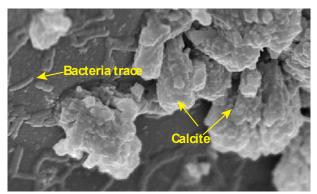


FIG. 5. SEM Images of precipitated calcites at 100 m scale

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# Influence of Pore Size Distribution on the Properties of a Stabilized Soil Cement System

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**Abstract**: Results of an investigation on soil cement system are presented in this paper. Materials used, preparation of sample is presented followed by results on porosity and bulk density. The pore size distribution (PSD) results obtained through Mercury Intrusion Porosimetry (MIP) are presented next, followed by results on thermal conductivity measurements. A general equation for PSD of porous construction materials is proposed earlier by the author and his team at IIT Delhi. The estimated parameters of above PSD equation are presented. Influence of some PSD parameters on dry and moist thermal conductivity is discussed next. The strength results are presented and role of porosity and PSD on strength is presented at the end to illustrate their influence on properties of soil cement blocks.

## **INTRODUCTION**

Stabilization of soil by cement is a well-known practice for long, in the road construction (Sherwood, 1993). Use of soil cement block as masonry unit has been also reported in the past. The embodied energy of fired clay bricks is high besides the carbon emission from clay burning process is also considerable. Thus alternative masonry units such as normal strength concrete blocks, aerated concrete blocks, lime fly-ash blocks are used in building construction. Ordinary Portland cement is the second largest contributor of carbon to the atmosphere and hence clinker factor reduction is a major concern in cement industry. Soil cement blocks may prove to be more sustainable masonry unit in building wall construction than others. Work reported in literature in past on cement stabilized soil are reviewed next.

Horpibulsuk (2012) reported studies on strength and, microstructure of cement stabilized clay system using Scanning Electron Microscopy (SEM), Mercury Intrusion Porosimetry (MIP) and thermo-gravimetric analysis (TGA). Soil containing about 53% clay, 45% silt and 2% sand exhibited strength enhancement up to a cement content of 10% consequent to refinement in pore sizes and lowering of porosity. For higher cement content improvement was not observed and, beyond a point deterioration has been reported. Montgomery et al. (2001) presented the basic requirements of soil composition, mechanisms and curing process etc., for cement stabilized soil blocks. MIP studies on stabilized clay were reported by Kawamura and Diamond (1975), Reddy and Gupta (2005) studied the characteristics of sandy soil cement blocks and reported the surface pore size distribution as observed through SEM. Balaji et al. (2015) suggested empirical relationship between density and thermal conductivity of soil cement blocks.

Porosity, pore sizes, and their interconnectivity govern performance of porous materials. The strength and thermal properties are governed by porosity, PSD and type of pores. (Kumar et al., 2003, Bhattacharjee et.al., 2004, Kondraivendhan et al., 2014) Pore size distribution of most of the porous materials can be expressed through the equation (1) proposed by Patil and Bhattacharjee (2008) as given below.

$$V = \frac{Pr_{0.5}^{\ d}}{r^{d} + r_{0.5}^{\ d}}$$
(1)

In equation (1), V is the cumulative pore volume fraction associated with radius r starting from the minimum radius. The PSD is characterized by three parameters: porosity P, median pore radius  $r_{0.5}$  and a dispersion coefficient d. P,  $r_{0.5}$  and d can be related to cement content, water to cement ratio etc., for cement based materials (Kondraivendhan and Bhattacharjee, 2010, 2013). The strength and permeability are also related to these parameters. (Kumar et al., 2003, Kondraivendhan et al., 2014)

In this paper, experimentally obtained strength, PSD parameters obtained through MIP and thermal conductivity results are presented and influence of PSD on performance related parameters are discussed.

#### MATERIALS AND EXPERIMENTAL INVESTIGATIONS

The tests were carried out at IIT Delhi with locally available soil; silt passing through 2.36 mm was used in this investigation. The soil contained 2.5 % clay, 55% silt and 42.5% sand as par grain size distribution. This soil exhibited 27% and 19.5%, liquid and plastic limits respectively, the plasticity index and shrinkage limits measured are 8.5% and 18.5% respectively for the soil. The optimum moisture content determined through Proctor's compaction test was 10.5% and the corresponding optimum dry density was 1850 kg/m<sup>3</sup>. Ordinary Portland Cement (OPC), 33 grade, as par Indian Standard (IS269:1989) was used as the stabilizer. The cement contents varied from 4-10% by mass of soil in steps of 2% to have 4 levels. Laboratory water was used for mixing. Water content was varied from 8.5% to near shrinkage limit i.e., 18.5%, at 4 levels: 8.5%, 10.5%, 14.5%, and 18.5%. Thus, a wide variation in porosity of the specimens was expected. Each test was performed with 3 replicates. The soil and requisite amount of cement was mixed dry followed by uniform mixing with measured quantity of required water. The samples for individual tests were then compacted to maximum possible compaction. All samples were cured at a temperature of 60°C, for 24 hours in a saturated humid condition for accelerated curing. The samples were then cooled and cured by covering with wet jute bags continuously for 6 days at room temperature ranging from 25-30°C. The samples were then allowed to dry in air in the same condition for next 15 days followed by tests.

The bulk density of the samples was determined by 48 hours saturation in clear colourless kerosene of specific gravity of 0.65. The saturated specimen ensured that no

further kerosene would be absorbed while measuring suspended immersed weight in liquid which was then determined. The difference between oven dried weight and suspended immersed weight in kerosene provided the mass of kerosene displaced by the specimen when immersed. Thus this difference in weight divided by specific gravity of kerosene corresponds to the volume of specimen. The bulk density of the specimens was thus calculated from these weight measurements. The obtain the porosity of the specimen the solid material of the blocks were made in to powder passing through 150 micron sieve and specific gravity was measured using Le Chatelier's flask with kerosene as the liquid (Kumar, 1987).

MIP test was performed on Quantachrome Autoscan-33 mercury porosimeter having a pressure range from sub-ambient to 227 MPa (33,000 psi). The contact angle and the surface tension of mercury were assumed to be 117° and 0.484 N/m respectively for the oven-dried samples. The largest radius (pore size) that can be accounted in the pore size distribution is 0.2 mm with sub-ambient pressure filling apparatus. The smallest size of the pore radius that could be measured is 2 Nano-meters. The sample cell fitted with the base cell of capacity 17.7 cc. was used throughout the experiment. All tests were performed at a constant moderate scanning rate indicated by point 5 of the machine knob on it 0-10-scale. (Kumar and Bhattacharjee, 2003) The data generated from pressure versus cumulative intrusion volume curve obtained from the measurement was converted in to cumulative volume versus pore entry radius data using Washburn equation. In equation (1), at V/P=0.5, r=r\_{0.5}, hence from a plot of V versus r, with known value of P obtained from MIP,  $r_{0.5}$  can be estimated. This P obtained from MIP is usually lower than that obtained from liquid displacement method mentioned earlier. The equation (1) can be rewritten as:

$$\left(\frac{P}{V}-1\right) = \left(\frac{r}{r_{0.5}}\right)^d \tag{2}$$

Thus the curve of  $\ln(P/V-1)$  versus  $\ln(r/r_{0.5})$  is linear with its slope near origin (0,0) is d. The  $r_{0.5}$  and d parameters were thus estimated. The retention factor is obtained as the ratio of volume of entrapped mercury after first extrusion to total intrusion volume of mercury at maximum intrusion pressure.

Thermal conductivity was tested by line source hot wire method using HC-60 of M/s EKO Instrument Trading Co. Japan with measuring range up to 11.3 W/m °C and accuracy  $\pm 5\%$ . In the test, two blocks of the material to be tested are put one over another in close contact, with hot-wire and the thermo-couple sensor assembly sandwiched between the two blocks. The size of each block is  $0.11 \times 0.11 \times 0.035$  m<sup>3</sup>. (Bhattacharjee and Krishnamoorthy, 2004) The thermal conductivity was measured at oven dry, i.e. 0%, 2.5%, 5%, and 7.5% moisture contents. The water was added drop by drop from oven dried condition after cooling to ambient temperature, until required moisture content was attained. The specific heat was determined using method of mixture using a setup fabricated as par guidelines of ASTM C 351 – 92b (1999).

Compressive strength was determined on cube specimen of 50cm<sup>2</sup> base area accordance with the testing procedure prescribed for cement mortar cube in Indian Standard, IS 4031 part 6: 1988.

#### **RESULTS OF EXPERIMENTAL INVESTIGATION**

The porosity values obtained through kerosene absorption test and bulk density values obtained for all sixteen experimental cases are given in Table 1. Both porosity and bulk density exhibits marginal improvement with mixing water and cement contents. The  $r_{0.5}$  and d, for all sixteen cases are given in Table 2. It can be seen that median pores, i.e., mean distribution radius decreases with increase in cement content. With mixing water content the trend is not very clear. The dispersion coefficient "d" increases with cement content exhibiting more uniform pore sizes. Value of d is quite low for this material and that represent widely dispersed pores.

Thermal conductivity and specific heat values for dry conditions are given in Table 3. Thermal conductivity increases with both cement content and mixing water content. Conductivity of cement is likely to be higher than that of soil and, water is more conductive than air, thus the observed conductivity of soil cement system increases with both increase in cement content and mixing water content. However, such clear trend is not observed in case of specific heat although it is known that specific heat of water is much higher than that of solids and air.

Cement	Porosity (%)				Bulk Density (kg/m <sup>3</sup> )			
content	Mixing Water Content (%)				Mixing Water Content (%)			
(%)	8.5	10.5	14.5	18	8.5	10.5	14.5	18
4	40	45.0	39.0	36.0	1612	1600	1642	1720
6	42.9	41.2	38.1	38.0	1528	1573	1651	1654
8	41.5	40.1	37.8	36.0	1555	1592	1654	1699
10	41.0	40.9	36.8	39.0	1561	1564	1670	1668

Table 1. Permeable porosity and Bulk Density

Cement	Median pore size(m)×10 <sup>9</sup>				Dispersion coefficient			
content	Mixir	Mixing Water Content (%)						
(%)	8.5	10.5	14.5	18	8.5	10.5	14.5	18
4	1096.6	1212	1635	897.8	0.466	0.547	0.419	0.432
6	897.8	1212	2208	1998	0.495	0.429	0.400	0.461
8	492	492.8	445.9	365.0	0.571	0.537	0.561	0.586
10	365	665.1	298.9	601.8	0.692	0.671	0.687	0.570

Cement	Thermal Conductivity (W/mK)Mixing Water Content (%)				Specific Heat (J/kgK) Mixing Water Content (%)			
content								
(%)	8.5	10.5	14.5	18	8.5	10.5	14.5	18
4	0.50	0.52	0.66	0.64	987	890	994	1021
6	0.57	0.59	0.67	0.63	831	868	754	889
8	0.66	0.66	0.72	0.73	879	770	902	976
10	0.67	0.66	0.74	0.77	891	821	857	973

The compressive strength results are given in Table 4. The compressive strength increases with cement content, but for mixing water content, the initial increase with increase in mixing water content is followed by a decrease beyond a point.

## PORE CHARACTERISTICS AND PROPERTIES

#### **Thermal Conductivity**

Thermal conductivity of can be related to permeable porosity and conductivity of solid through fraction of enclosed pores as given in equation (3), (4) and (5) (Bhattacharjee and Krishnamoorthy, 2004).

$$\mathbf{k}_{ed} = \mathbf{k}_{s} \left[ \mathbf{k}_{1d}^{(1-f)} \times \mathbf{k}_{2d}^{f} \right]$$
(3)

The  $k_{1d}$  and  $k_{2d}$  in equation (3) are effective thermal conductivities of unit cell containing enclosing pores and enclosed pores respectively and are as given below. The  $k_{ed}$  is the effective thermal conductivity of the overall dry material, where two types of unit cells are arranged randomly. The  $k_s$  is the conductivity of the pore free solid skeleton. The detailed description of the model and concepts are available in the previous publication (Bhattacharjee and Krishnamoorthy, 2004).

$$\frac{1}{k_{1d}} = A1.k_s + B1;$$
 (4)

$$k_{2d} = B2 - A2.k_s$$
(5)

In the above equations,  $A1=30.99P^2-0.46P+2.29$ ;  $B1=1.17P^2-0.51P+1.15$ ;  $A2=(0.63P^2+3.3P+0.30) \times 10^{-3}$ ;  $B2=0.33P^2-1.32P+1.01$ . The fraction of enclosed pores f is related to retention fraction R measured using MIP and is given in equation (6).

$$f = 3.13R - 3.18R^2$$
(6)

The average R for four solid materials with cement contents 4%, 6%, 8% and 10% are 0.44, 0.47, 0.34 and 0.35 respectively. The corresponding fraction of enclosed pore f estimated from equation (6) are 0.76, 0.77 0.70 and 0.71 respectively. Using these f

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values and mean of experimentally determined P and  $k_{ed}$  for a given cement content, the value of  $k_s$  is estimated using a trial and error procedure. The estimated values of solid conductivity values for cement contents 4%, 6%, 8% and 10% are 1.49, 1.55, 2.32 and 2.32 respectively. These values of  $k_s$  can be used to estimate dry solid conductivity of soil cement block for known porosity and cement content. Thermal conductivity of partially saturated porous material can be related to degree of saturation through equation (7), given below. (Bhattacharjee, 2012)

$$\phi_m = -\alpha \theta^2 + \beta \theta \tag{7}$$

Where  $\phi_m$  is relative moist conductivity and is given as:  $(k_{em} - k_{ed})/(k_{es} - k_{ed})$ .  $k_{em}$  is the moist conductivity at degree of saturation  $\theta$  expressed as  $(w_m - w_d)/(w_s - w_d)$ ; w stands for weight and subscripts 'm', 's' and 'd' represent moist, saturated and dry states respectively. The saturated moisture content is estimated from porosity and bulk density values given earlier and thus degree of partial saturation is calculated from given and saturation moisture contents. Average  $\alpha$  and  $\beta$  are 1 and 2 respectively for most of the materials. (Bhattacharjee, 2012) The kem values have been measured at few degrees of saturation for the cases mentioned earlier. From equation (7) and for  $\alpha$  and  $\beta$  as above,  $(k_{em} - k_{ed})$  is equal to  $(k_{es} - k_{ed}) \times (-\theta^2 + \theta)$ . Thus slope of regression line between  $(k_{em} - k_{ed})$ and  $(-\theta^2 + \theta)$  is  $(k_{es} - k_{ed})$ . Hence from the slope of these lines saturated conductivity have been determined and given in Table 4. The thermal conductivity at saturation moisture content depends both on porosity as well conductivity of solid and thus a systematic pattern is not clearly visible in Table 4. However, with these values one can estimate thermal conductivity at various degrees of saturation. The specific heat of the blocks generally follows the bulk density; as specific heat of air is very small and specific heat can be assume to follow linear law of mixture for porous materials. Specific heat and diffusivity of moist soil cement blocks can also be estimated from simple law of mixture knowing moisture content (Bhattacharjee, 2012).

#### **Strength and Pore Characteristics**

A look in to Tables 2 and 4 reveals that correlation of compressive strength with density or porosity is poor. In case of cement paste, cement sand mortar and concrete the strength can be related to porosity P, median pore size  $r_{0.5}$  and cement content through equation (8); (Kumar and Bhattacharjee, 2003; Kondraivendhan and Bhattacharjee, 2010, 2013, 2014)

$$\sigma = K_2 C \frac{(1-P)}{\sqrt{r_{0.5}}}$$
(8)

In equation (8),  $\sigma$  is the compressive strength, C is the cement content expressed as fraction, K<sub>2</sub> is a constant and is function of elastic modulus and surface energy of crack free material. A plot of  $[C(1-P)]/(r_{0.5})^{\frac{1}{2}}$  after omitting a few outliers, is shown in Fig.1. The correlation involving all sixteen data points for the above line passing through

origin was somewhat low as only about 60% of variation is explained. On removal of data points, one by one, on the basis of the point having maximum deviation from estimated line, the relationship improved and 88% of the variation can be explained by the line shown confirming the linear trend. The median pores size estimated by MIP may be somewhat erroneous as larger sized pores encountered in case soil cement blocks may be excluded by the method. One may notice that permeable porosity measured is much higher than that measured through MIP. Further with larger set of data, the correlation is likely to be improved more compared to the set of data used.

Cement	Saturation C	Compressive strength (MPa)						
content	Mixing W	Mixing Water Content (%)						
(%)	8.5	10.5	14.5	18	8.5	10.5	14.5	18
4	-	1.27	2.12	2.34	2.7	3.8	3.3	2.7
6	1.34	1.65	1.70	2.22	4.2	6.5	5.8	3.5
8	2.21	2.34	2.04	2.44	6.2	7.0	8.0	6.0
10	2.12	2.39	1.90	1.81	7.3	7.9	8.8	6.2

Table 4. Thermal conductivity at moisture saturation and compressive strength

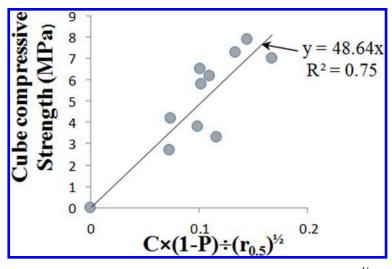


FIG. 1. Cube compressive strength versus  $C(1-P)/(r_{0.5})^{\frac{1}{2}}$ 

## CONCLUSIONS

Thus it is concluded that thermal properties of soil cement blocks are governed by the porosity and fraction of enclosed pores, like other bricks and blocks.

Strength of the soil cement blocks on the other hand is controlled by porosity and median pore size. Higher porosity and larger pore sizes lower the strength, as demonstrated through the experimental results and equations presented in the paper.